

# Analysis of lattice changes and weak interactions in a series of solvatomorphic isostructural spin-crossover compounds

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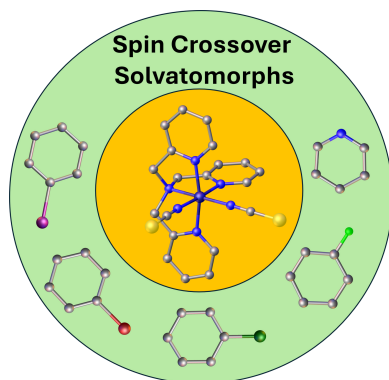
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**Supporting information:** this article has supporting information at journals.iucr.org/c

Five new solvate forms of the complex bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II),  $[\text{Fe}(\text{NCS})_2(\text{C}_{18}\text{H}_{18}\text{N}_4)]$  or  $[\text{Fe}(\text{NCS})_2(\text{tpa})]$ , a known spin-crossover compound, are reported. Among the incorporated solvents are all four phenyl monohalides (fluoro-, chloro-, bromo- and iodobenzene) and pyridine. Through variable-temperature single-crystal X-ray diffraction, we characterized the transition by means of lattice parameters, Fe–N bond lengths and distortion of the coordination sphere. The structures remain isostructural across the compound series, but exhibit distinct transition temperatures indicating a solvate-dependent behaviour. Hirshfeld surface, noncovalent interaction analyses and energy frameworks provide a more detailed view of how the structural response affects the intermolecular interaction network. In particular, the temperature dependence of the dominant pairwise interaction along the crystallographic  $b$  direction closely mirrors the evolution of the  $b$ -axis lattice parameter, suggesting that this interaction plays a key role in directing the structural response to spin crossover. Our comparison demonstrates that different transition behaviour is not caused by one fundamental change in the packing architecture but rather by subtle solvate-dependent changes.

## 1. Introduction

Changes in an external stimulus, such as temperature or pressure, can induce spin-state switching in metal complexes, particularly at iron(II) centres. The resulting spin crossover (SCO) has been well studied in countless solid samples, including exhaustive crystallographic analyses at the molecular level (Brück *et al.*, 2026; Guionneau, 2014; Halcrow, 2011; Collet & Guionneau, 2018; Guionneau *et al.*, 2004). The, to some extent, abrupt distortion experienced by the complexes during the transition is recognizable in an uneven thermal evolution of unit-cell metrics and interatomic distances. By variation in ligand designs or counter-ions, the SCO phenomenon has been studied in terms of molecular interactions and crystal packing (Halcrow *et al.*, 2019; Fang *et al.*, 2024; Molnár *et al.*, 2019; Pfaffeneder *et al.*, 2011). Numerous results in this context showed that even subtle variations, like the exchange of solvent in the crystal structure, can drastically change the transition behaviour (Chernyshov *et al.*, 2003; Hostettler *et al.*, 2004; Bartel *et al.*, 2007; Liu *et al.*, 2025). Remarkably, the thiocyanate iron(II)–tpa complex [tpa is tris(pyridin-2-ylmethyl)amine] (Højland *et al.*, 1983) is known to have several polymorphs and solvatomorphs, which are SCO active when subjected to temperature changes. Since the elucidation of its first crystal structure (Li *et al.*, 2010), four polymorphs (Wei *et al.*, 2011a) and seven solvatomorphs (Wei



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*et al.*, 2011b) of this compound have been reported up to now (Fig. 1). They exhibit varying transition behaviour depending on the solvate present. Among the different solvatomorphs, only a few are isostructural when the solvate molecules are very similar (e.g. MeOH and EtOH).

Herein, we report the structures of five new solvate forms of bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II), **1**, which all crystallize in the same space group ( $P2_1/n$ ) with similar lattice constants (see the supporting information). They can thereby be considered as isostructural (Lima-de-Faria *et al.*, 1990). SCO compounds, which mimic each other in space-group symmetry and packing, naturally arise when one tries to design SCO structures that are closely alike to analyse their (yet different) transition behaviour. Often found examples are isostructures induced by anion exchange (Hibbs *et al.*, 2003; Wiehl, 1993), partial occupation of the iron position by another metal (Mueller *et al.*, 1983), or solvate exchanges (Białońska & Bronisz, 2010; Birchall *et al.*, 2023). Yet, with regard to what is already reported and together with our findings, compound **1** appears to be a true wellspring of solvatomorphs and polymorphs, and, to the best of our knowledge, we cannot name another SCO compound possessing more solvate forms.

The focus of this work is aimed at investigating the changes that the crystal packing exhibits in response to varying temperatures. Especially as the weak intermolecular interactions determine the transition behaviour of SCO compounds (Halcrow, 2011), in-depth analysis of short contacts has proven to reveal the most directing packing features (Li *et al.*, 2010; Sheu *et al.*, 2008; Pfaffeneder *et al.*, 2011). While it is the current scientific consensus that crystal packing guides the spin transition, short interaction criteria that go beyond distance measures (Tan *et al.*, 2019) are rarely used for in-depth analysis of SCO compounds (Miyawaki *et al.*, 2020; Novikov *et al.*, 2014; Díaz-Torres *et al.*, 2022; Tailleux *et al.*, 2019; Reeves *et al.*, 2021). The cause may be that already small differences in any structural aspect can change the SCO behaviour, making an estimate of influences hard to generalize (Díaz-Torres *et al.*, 2022; Real *et al.*, 2003; Takahashi *et al.*, 2010). So, with our series of iron(II)–tpa complexes, which are

very similar in their crystal packing, we want to gain structural insights through variable-temperature single-crystal X-ray diffraction and further rationalize intermolecular interactions. For the latter, we use computational methods like Hirshfeld surfaces (Spackman & McKinnon, 2002; McKinnon *et al.*, 2007), energy frameworks (Mackenzie *et al.*, 2017) and NCI plots (Contreras-García *et al.*, 2011). The experimentally determined geometric inputs for the named methods are enhanced through Hirshfeld Atom Refinement (HAR) (Capelli *et al.*, 2014), which was shown to improve structural quality and was made readily available (Kleemiss *et al.*, 2021a).

## 2. Experimental

### 2.1. Synthesis and crystallization

#### 2.1.1. Synthesis of the phenyl halide solvates of compound **1**

A general procedure for the synthesis of suitable single crystals for diffraction analysis, where all solvents were degassed prior to use, was employed. A solution of tpa (70  $\mu\text{mol}$ , 1 equiv.) dissolved in the appropriate phenyl halide (1 ml) was placed at the bottom of a Schlenk tube. Above the ligand solution, a mixture of acetone and phenyl halide (2 ml, 1:1 v:v) was carefully layered. The top layer contains  $[\text{Fe}(\text{NCS})_2(\text{py})_4]$  (70  $\mu\text{mol}$ , 1 equiv.) in acetone. When left undisturbed, large single crystals of **1**·FPh and **1**·BrPh were obtained after 1–3 d. Crystals of **1**·ClPh and **1**·IPh were obtained after the solutions were slowly concentrated by a steady flow of nitrogen.

#### 2.1.2. Synthesis of the pyridine solvate of compound **1**

Bulk material of **1** was prepared as reported previously (Yu *et al.*, 1994). Recrystallization from pyridine yields crystals of **1**·py when the solution was left undisturbed in a fridge.

### 2.2. Refinement

Crystal data, data collection and structure refinement details for all determinations are summarized in the

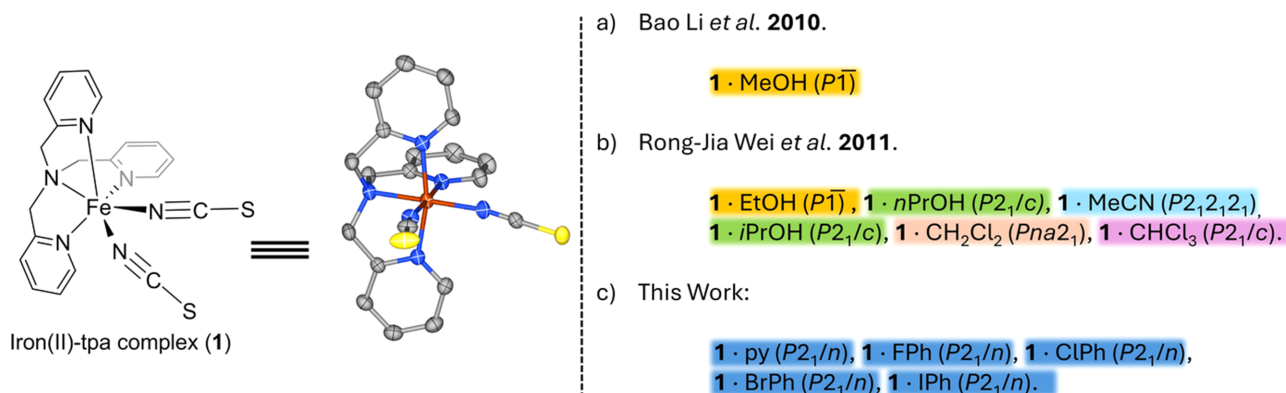


**Figure 1**

(Left) The molecular structure and representative ellipsoid plot (50% probability level) of  $[\text{Fe}(\text{NCS})_2(\text{tpa})]$ . (Right) Overview of the reported solvatomorphs of compound **1**, with the isostructural compounds colour-coded.

supporting information. For each compound, a suitable crystal was chosen and a temperature series of diffraction experiments was measured on an XtaLAB Synergy R diffractometer equipped with a HyPix-Arc 100 detector and Mo  $K\alpha$  radiation. The temperature was controlled by an Oxford Cryostream 700 and the data series was collected in cooling mode only. Data integration, reduction, scaling and Gaussian absorption correction were performed in *CrysAlis PRO* (Rigaku OD, 2025). The structures were solved using *SHELXT* (Sheldrick, 2015) and further refined with *olex2-refine* within the *OLEX2* software suite (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015). Initially, the H-atom positions were kept fixed at idealized positions. Besides **1-py**, all structures exhibit disorder in the high-temperature phase. The disorder only affects the solvate molecules, resulting in different orientations of the halide atom. While chloro- and bromobenzene split into two parts, with occupancies refined freely, fluorobenzene was modelled as three parts, with fixed occupancies to ensure convergence during refinement. Iodobenzene showed a positional disorder to a great extent, which was not resolvable by explicit modelling, so a solvent mask was applied (SQUEEZE; Spek, 2015). Each final model was subjected to an iterative Hirshfeld Atom Refinement using *NoSpherA2* (Kleemiss *et al.*, 2021a). Prior to the partitioning of the electron density, the calculation of the wavefunction was performed using *ORCA* (Version 6.0; Neese, 2012; Neese,

2023; Neese *et al.*, 2020), applying the  $r^2$ SCAN/def2-TZVP (Furness *et al.*, 2020; Weigend & Ahlrichs, 2005) level of theory, including a water CPCM (Conductor-like Polarizable Continuum Model) model (Barone & Cossi, 1998; Brüx *et al.*, 2025). Additional crystallographic information is listed in Section 2 of the supporting information.

### 2.3. Computational methods

The Hirshfeld surfaces were generated using *Crystal-Explorer17* (Spackman *et al.*, 2021). For the disordered high-temperature phases, only the major part was included in the analysis. The normalized contact distance ( $d_{\text{norm}}$ ) surface maps are visualized at a scale between  $-0.1$  and  $1.25$  a.u. Corresponding fingerprint plots are shown in the supporting information (Section 5). The pairwise energies included complete molecular units within a radius of  $3.8$  Å and were calculated using the CE-B3LYP model (Mackenzie *et al.*, 2017).

Noncovalent interaction indicators (Contreras-García *et al.*, 2011) were generated by growing an appropriate number of neighbouring molecules within *OLEX2* and saving two separate .xyz files, one containing the central unit and the other containing selected surrounding molecules. Based on this input, a modified version of *NoSpherA2* (Kleemiss *et al.*, 2021b) was used to calculate the electron density and the reduced density gradient (RDG) based on Thakkar's spherical

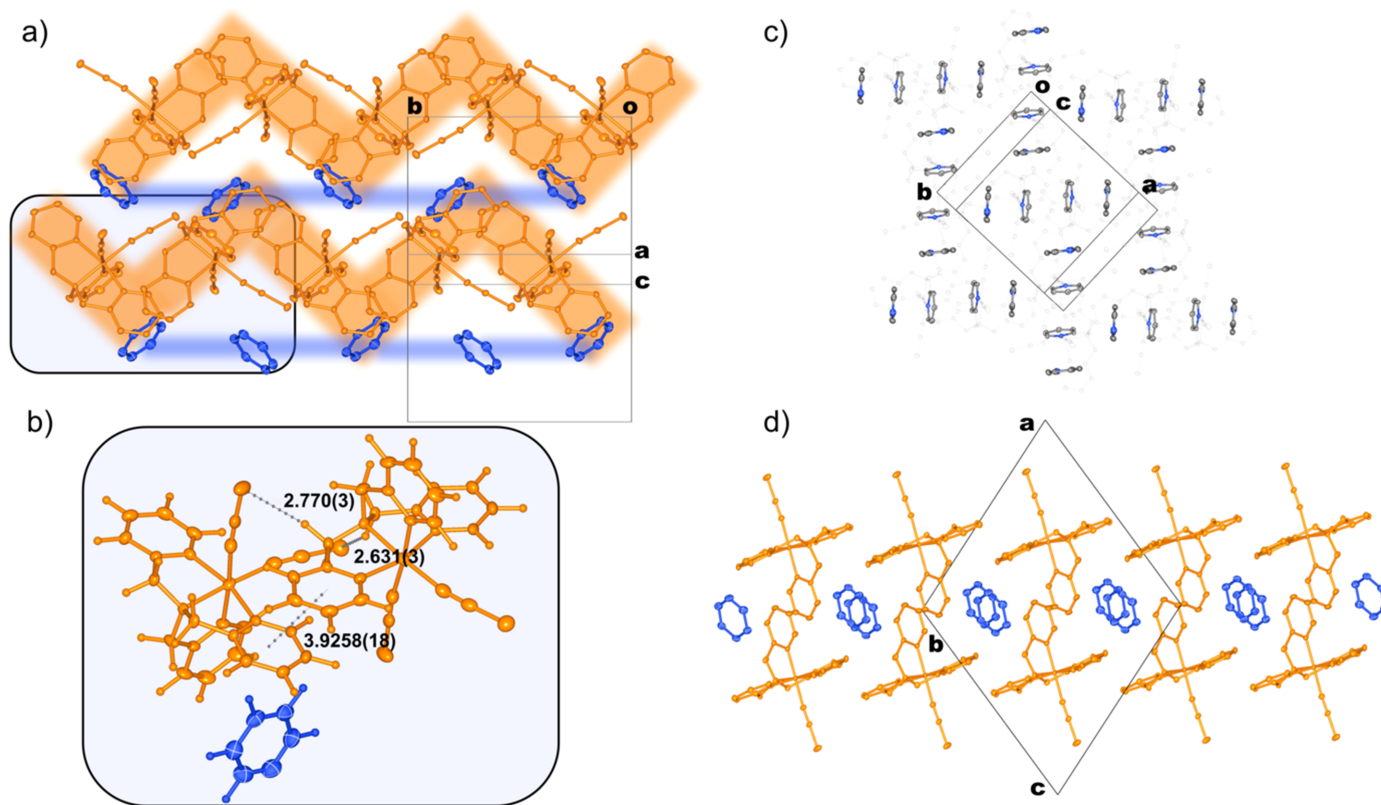


**Figure 2**

Packing diagrams of **1-py** at 95 K, with displacement ellipsoids drawn at the 50% probability level. (a) View along the *ac* diagonal, with the zigzag chain of the complex unit highlighted in orange and the direction in which the pyridine units propagate highlighted in blue. (b) Zoomed view of the arrangement of two neighbouring complexes, with close contacts annotated. (c) View approximately along *c*, highlighting the  $\pi$ -stacking between the solvate and the complexes. (d) View along the *b* axis. Only **1-py** is shown, but the description applies to all isostructural compounds.

densities (Kleemiss *et al.*, 2024). The outputs were visualized in *VMD* (Humphrey *et al.*, 1996). Corresponding scatterplots of the RDG *versus*  $\text{sign}(\lambda)\rho$  are given in Section 4 of the supporting information.

### 3. Results

Crystals of the reported compounds were obtained through the approach described above and can be stored in their mother solution for months. Yet, when they are removed from the solution for crystal picking, a loss of crystallinity occurs within minutes. A sample of **1**-py was analysed as a representative example by powder X-ray diffraction, confirming the weak diffracting nature of the bulk sample (see Fig. S5 in the supporting information). In all cases, the freshly picked yellow-coloured crystallites were suitable for in-house single-crystal analysis. It is advisable to mount the crystal at temperatures above 200 K, since otherwise the abrupt lattice contraction during the spin transition leaves the crystal shattered and hinders proper data collection (see Section 1 in the supporting information). When the samples were cooled in a controlled manner (*e.g.* at a rate of  $25\text{ K h}^{-1}$ ), suitable data could be collected even below the transition temperature.

#### 3.1. Crystal packing

The abrupt changes in the crystal lattice were in no case accompanied by a change in crystal symmetry or space group, and both the high- and low-temperature phases crystallize in the monoclinic space group  $P2_1/n$  with  $Z' = 1$ , including a

solvate molecule in the asymmetric unit. The iron(II) cation is coordinated in an octahedral fashion, without involving the solvate in the coordination sphere. Within the unit cell, the solvate molecules are always arranged at the same position along the twofold axes separated by  $b/2$  [Figs. 2(a) and 2(d)]. The iron complex units follow this direction by a zigzag chain along the screw axes, where adjacent pairs were connected by  $\pi$ - $\pi$  contacts in addition to nonclassical hydrogen bonding (Desiraju, 1991) involving C—H...S contacts [Fig. 2(b)]. The latter involves the methylene group of tpa, which was also described as a packing-directing feature in the solvent-free form of **1**, which also retains this zigzag motif along the  $b$  axis (Li *et al.*, 2010). Two staggered inversion-related complex units are also involved in a sandwich-like  $\pi$ -stacking that includes the solvate molecules in the form solvate...**1**...**1**...solvate [Fig. 2(c)] along the cell diagonals.

#### 3.2. Disorder of the solvate

During the temperature series, the pyridine solvent molecule retains its orientation (see Fig. S24) without the N atom being involved in any stronger intermolecular interaction, which could explain this preferred orientation. Besides **1**-py, all phenylic solvate molecules exhibit a disorder with respect to the direction in which the halogen atom points. In most cases, the superposition was modelled through a rotational disorder along the twofold screw axis. In all structures, the disorder resolved upon entering the low-spin (LS) state, as signified by the absence of significant residual density in minor parts that had previously been occupied by a small fraction

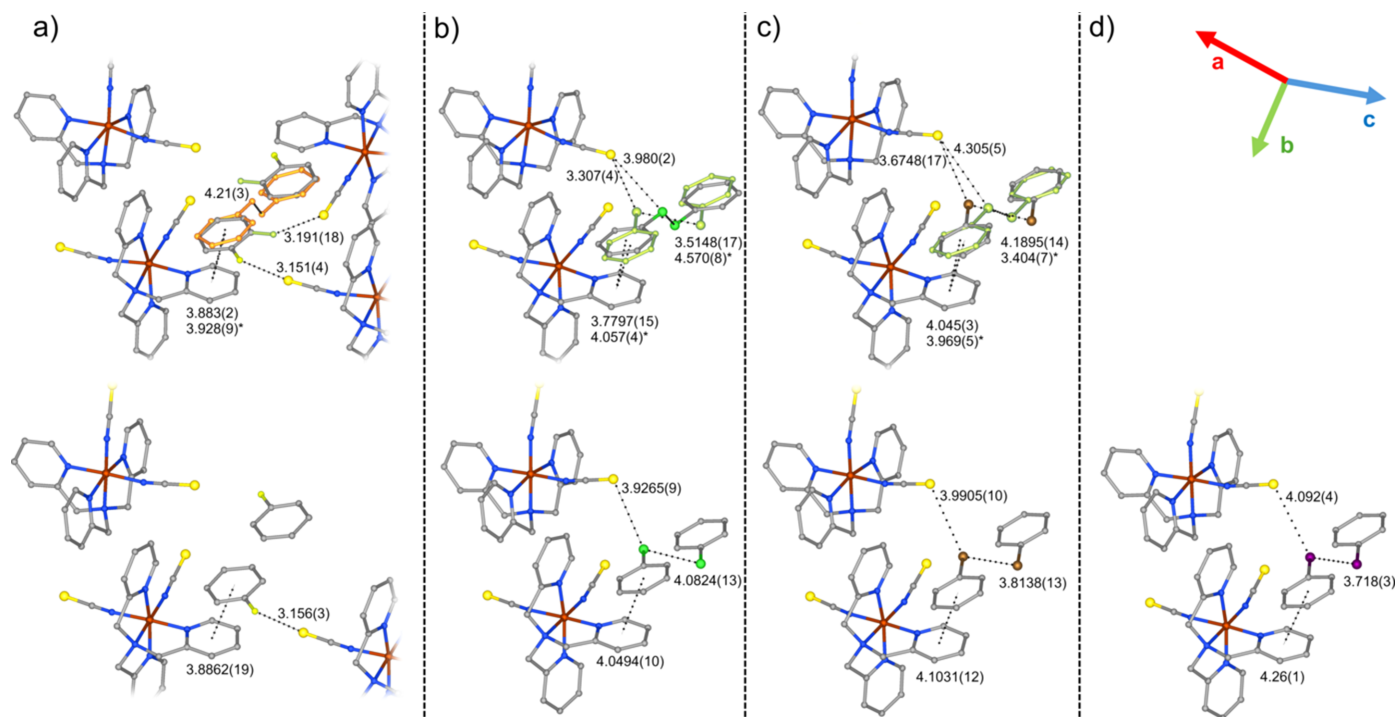


**Figure 3**

The molecular arrangements of the halide-containing solvates, before the transition (upper row) and after the transition (lower row). Structures are drawn in ball-and-stick style and H atoms have been omitted. The first minor part is drawn in green and the second minor part in orange (only for **1**-FPh). For parts (a) to (d): **1**-FPh at 155 and 150 K, **1**-ClPh at 170 and 160 K, **1**-BrPh at 190 and 180 K, and **1**-IPh at 150 K only.



(Fig. 3). The orientation of the phenyl halides seems to be influenced by adjacent sulfur or halide substituents in both the disordered high-temperature (HT) and low-temperature (LT) phases, indicating a directive role of interhalogen contacts (Desiraju & Parthasarathy, 1989). For the iodine variant, a severe displacement of the solvate over several positions hindered the modelling of the disorder and the HT datasets were subjected to *PLATON SQUEEZE* (Spek, 2015; Spek, 2020) to give a reasonable structure. The Cl- and Br-containing components show a similar disorder in which the minor fraction of the solvate is slightly rotated ( $\sim 29^\circ$  for Cl and  $\sim 46^\circ$  for Br) with respect to the major fraction. At 270 K, the highest evaluated temperature, the (minor) part has an occupancy of 41.8 (3)% for Br and 50.5 (4)% for Cl, which decreases to 34.2 (3)% for Br and to 27.8 (2)% for Cl at the final temperature before the transition. Measures of inter-

molecular distances for the solvates are displayed in Fig. 3 and show that the disordered parts prefer different short contacts. These mainly include the thiocyanate S atom of an adjacent complex, the halide of a neighbouring solvate and the above-mentioned solvate...**1**  $\pi$ -stacking within the asymmetric unit. In **1-ClPh**, the major part prefers Cl...Cl contacts over Cl...S contacts, while the minor part shows the opposite behaviour. The mentioned interatomic distances fall into the regime of halogen or interhalogen contacts (Grineva, 2023) and with decreasing temperature, the conformation having the shorter Cl...Cl distance [3.5148 (17) Å; see Fig. 3(b)] gets more populated, reducing the presence of the shorter Cl...S contact [3.307 (4) Å]. Compound **1-BrPh** shows the opposite behaviour, favouring the Br...S contact as the temperature decreases [Fig. 3(c)]. Less difference in the  $\pi$ -stacking can be observed between the disordered parts in both **1-ClPh** and **1-BrPh**. The fluorobenzene solvate is expected to have the weakest affinity to form an F...F interhalogen bond (Jelsch *et al.*, 2015), which is reflected in the distance of 4.21 (3) Å in the HT phase [Fig. 3(a)]. The disordered part having this F...F contact is one of three parts being included in the disorder model of the HT phase, and it is also the least occupied. The other parts show close contacts towards the thiocyanate S atom of different adjacent complex units, ranging around 3.2 Å. Only one F...S contact remains after the transition into the LT phase, a conformational difference unique to the fluorobenzene solvate. The Cl-, Br- and I-containing compounds also result in a disorder-free model, averaging the initially split solvate position and closely resemble each other. The X...S distances follow the trend Cl...S < Br...S < I...S, while the X...X distance follows an I...I < Br...Br < Cl...Cl order. The  $\pi$ - $\pi$  plane-centroid-to-centroid distance also increases from F to I. In particular, the contrast between a (partially) severely disordered HT phase and a fully ordered LT phase indicates the significant lattice changes that the compounds undergo, thereby forcing the solvates to adopt a single conformation.

### 3.3. Temperature dependence of the unit-cell parameters

The temperature-dependent unit-cell parameters are available for all reported compounds, since their single-crystal structures were evaluated at different temperatures over a broad temperature interval (see Fig. 4). In all compounds, the unit-cell volume decreases upon cooling and the absolute volumes differ according to the incorporated solvent molecules. The structural response associated with the spin conversion is indicated by discontinuities or changes in slope within a compound-specific temperature interval. Approximate transition regions are: py 150–175 K, F 150–155 K, I 155–160 K, Cl 165–170 K and Br 180–190 K. For the F-, Cl-, Br- and I-containing compounds, the transition into the low-spin phase is characterized by an abrupt contraction of the *a* axis, accompanied by an elongation of the *b* axis. The *c* axis decreases moderately, with only small discontinuities near the corresponding transition temperature. Compound **1-py** exhibits a different response: the changes are distributed over a

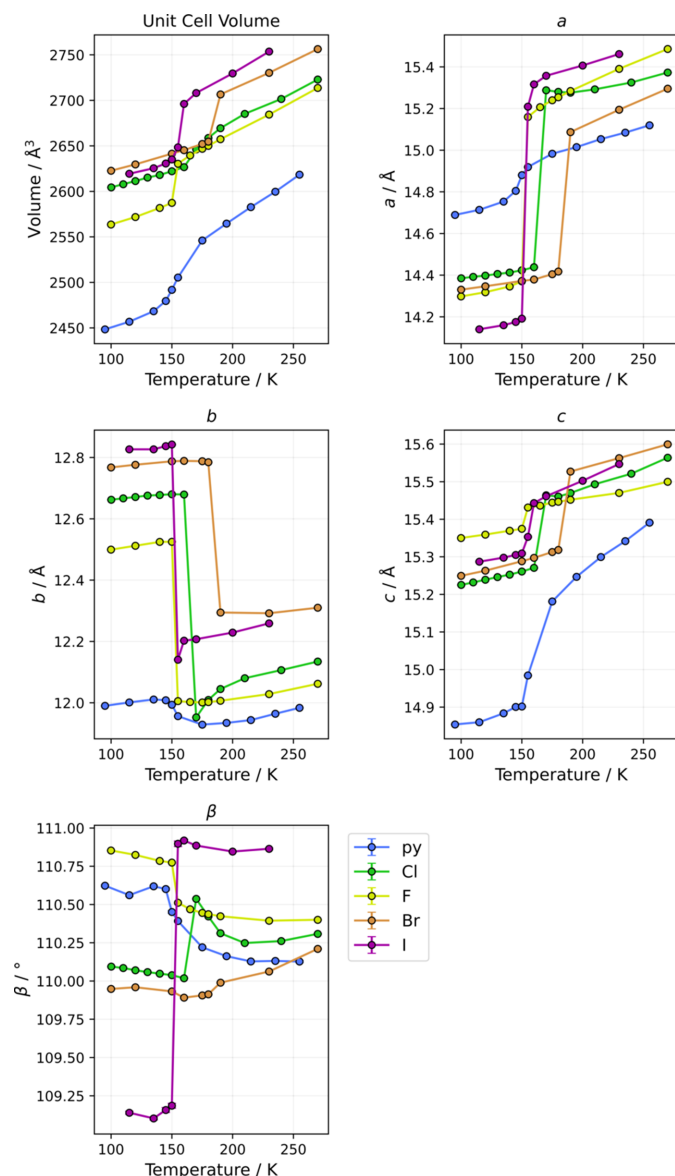


**Figure 4**

Lattice parameters as a function of the temperature. ESD-based uncertainty bars are shown but are obstructed by the marker size in many cases.

broader temperature interval. Pronounced variations are found in the *a* and *c* axes, whereas the *b* axis changes only slightly. The monoclinic angle  $\beta$  shows no uniform trend across the series. Compound **1**-IPh exhibits a pronounced increase in  $\beta$  near 155 K, whereas the changes for the compounds containing the F, Cl, Br and pyridine solvates are smaller and differ in sign. Thus, although the compounds retain a common structural type, incorporating different solvates modifies the temperature-dependent lattice response. The behaviour is consistent with previous observations, *i.e.* that solvent inclusion can strongly influence the SCO even when the principal host packing remains essentially unchanged (Cook *et al.*, 2016).

### 3.4. Temperature dependence of the iron(II) coordination centre

While the unit-cell parameters describe how the crystal lattice responds collectively to the SCO, they do not directly reveal the local structural changes within the iron(II) coordination sphere, such as changes in bond length and octahedral distortion. Hence, a direct structural indicator is the mean Fe–N bond length ( $\langle\text{Fe–N}\rangle$ ), which increases upon population of the antibonding orbitals in the HS state. In addition to this expansion, the deviation of the coordination environment from an ideal octahedron can be quantified using an angular distortion parameter (Collet & Guionneau, 2018)  $\Sigma$  with

$$\Sigma = \sum_{i=1}^{12} |90^\circ - \phi_i|$$

where  $\phi$  represents the 12 N–Fe–N angles. The value of  $\Sigma = 0^\circ$  corresponds to an ideal octahedron, whereas larger values indicate progressively stronger angular distortion of the iron coordination sphere.

The temperature dependence of  $\langle\text{Fe–N}\rangle$  and  $\Sigma$  is shown in Fig. 5. At high temperatures, the mean Fe–N distances range from approximately 2.16 to 2.18 Å across all compounds, consistent with a high-spin state of iron(II). Upon cooling, all compounds undergo a decrease to 1.96–1.98 Å, characteristic of the low-spin state. The magnitude of the bond-length change is similar throughout the series, whereas the temperature range and abruptness of the conversion depend on the incorporated solvent.

Compounds **1**-ClPh and **1**-BrPh display the most abrupt changes, and the Br compound shows the transition at the highest temperature near 190 K, while **1**-ClPh has the second highest transition at around 165 K. Both **1**-FPh and **1**-IPh display a less abrupt transition, shifted towards lower temperatures at approximately 150 K. In contrast, **1**-py shows a broader and more gradual evolution, extending approximately from 145 to 190 K. The octahedral distortion follows the same overall trend. At high temperatures,  $\Sigma$  exceeds  $80^\circ$ , while after the transition to the LT phase, the parameter is approximately  $40^\circ$  for all compounds. The changes in  $\Sigma$  occur within the same temperature intervals as the corresponding changes in the mean Fe–N distances, with the F-, Cl- and Br-containing compounds showing a step-like change, while **1**-IPh displays a more gradual evolution during cooling. Compound **1**-py again differs by exhibiting a broader continuous decrease in  $\Sigma$ , consistent with the more gradual evolution of bond length. Taken together, although the low- and high-temperature geometries are broadly comparable across the series, the nature of the transition is strongly solvate dependent.

## 4. Discussion

The different transition temperatures and steepness observed in the variable-temperature structural analysis indicate that the five compounds exhibit varying degrees of cooperativity

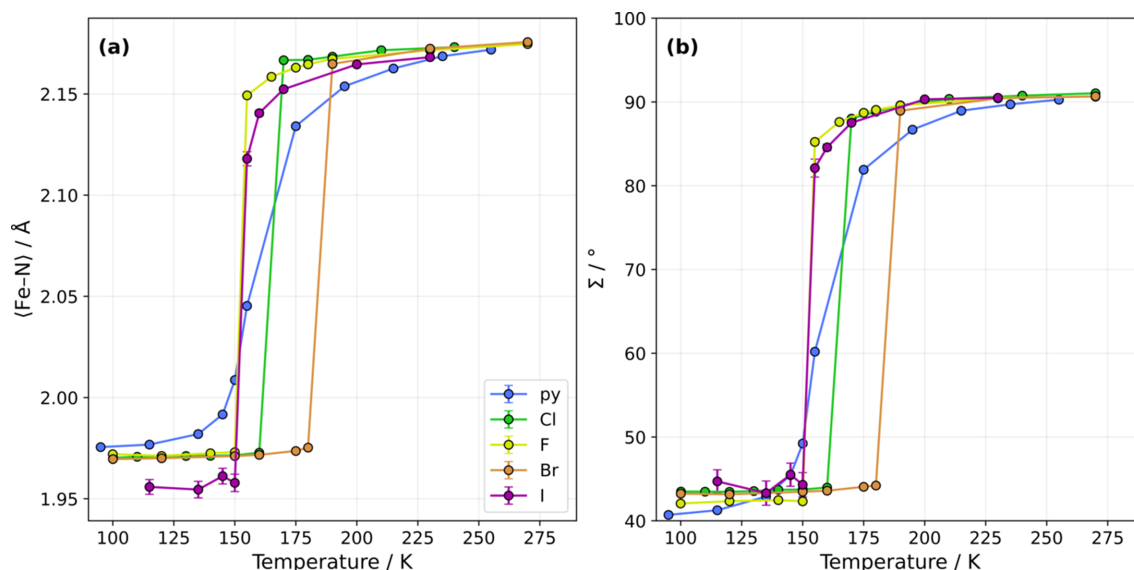


Figure 5

(a) Temperature evolution of the average Fe–N distances. (b) Temperature dependence of the octahedral distortion. Errors on the values were propagated.

despite their close structural relationship. Cooperativity describes the extent to which the spin-state change at one metal centre influences the transition of neighbouring centres (Kreutzburg *et al.*, 2017; Slichter & Drickamer, 1972). Stronger or more efficiently connected molecular architectures can therefore promote a more collective SCO, resulting in a sharper transition, whereas weaker coupling generally leads to a more gradual crossover. The differences observed across the series consequently motivate a closer examination of intermolecular environments.

Representation of the molecular environment within the crystal is enabled by Hirshfeld surfaces, which identify specific close intermolecular contacts. Complementarily, NCI analysis visualizes interactions as isosurfaces of the reduced density gradient mapped on the electron density. The distinction between attractive, weak noncovalent and repulsive is indicated by the respective colouration of the NCI isosurface in blue, green and red regions (Tan *et al.*, 2019). Fig. 6 shows the FPh-containing structure as a representative example at two temperatures: before the transition at 155 K [Figs. 6(a) and 6(c)] and after the transition at 150 K [Figs. 6(b) and 6(d)]. The Hirshfeld surface around the central complex is dominated by a small number of recurring intermolecular contacts in both states. These include most notably the F...S contact, C—H...S interactions and  $\pi$ — $\pi$  contacts between neighbouring aromatic fragments. The latter are highlighted by the NCI isosurfaces [Figs. 6(a) and 6(b)]. A comparison of the HS

and LS structures shows that the overall contact pattern is retained, consistent with the isostructural character of the transition, but the prominence of some contacts changes upon spin conversion. In the LS state, the contraction of the coordination sphere is accompanied by a generally denser packing arrangement, as reflected in the more pronounced Hirshfeld surface features. The NCI isosurfaces at the relevant interaction sites mostly agree with that but also reveal more pronounced weak C—H...S interactions towards the neighbouring complex depicted below (symmetry code:  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ). Despite being subtle, this indicates that these interactions accommodate or respond to the molecular contraction associated with the transition. The aromatic stacking and F...S interaction are comparably unaffected and may therefore contribute less to the nature of the transition. This is consistent with previous observations that short intermolecular contacts, including (nonclassical) hydrogen bonds involving the NCS ligands, are more effective in transmitting cooperative structural changes than longer-range interactions, such as  $\pi$ — $\pi$  contacts (Pfaffeneder *et al.*, 2011). Taken together, the listed tightening and changes in pre-existing intermolecular contacts may define pathways for the propagation of structural changes through the lattice. Even though the above analysis describes **1-FPh**, it also generally accounts for the other compounds, for which the corresponding plots are provided in Section 3 of the supporting information. As the differences in close contacts between the HS and LS states are

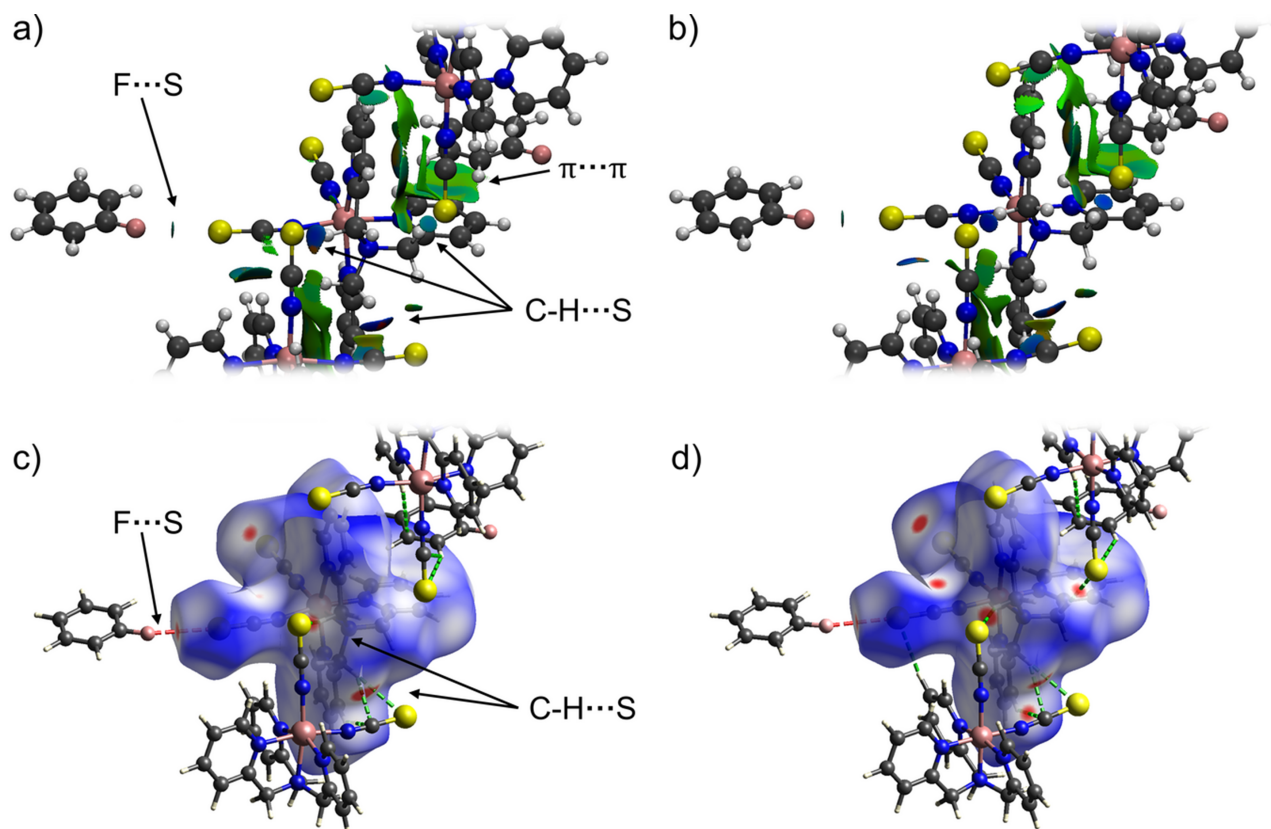


**Figure 6**  
NCI plots of **1-FPh**, highlighting the relevant short contacts at (a) 155 and (b) 150 K, the RDG is cut at 0.4, while  $\text{sign}(\lambda)\rho$  is colour-coded from  $-0.015$  (blue) via 0 (green) to  $0.015$  a.u. (red). Views of the Hirshfeld surface mapped with  $d_{\text{norm}}$  at (c) 155 and (d) 150 K.



only marginally reflected in the corresponding fingerprint plots, they are not discussed further, but are included in the supporting information for the sake of completeness (see Figs. S15–S19).

Hirshfeld surface and NCI analyses identify short contact motifs around the SCO centre, but they do not quantify their relative energetic contributions. Beyond this purely qualitative description of the short contacts, *CrystalExplorer* (Spackman *et al.*, 2021) pairwise intermolecular interaction energies were calculated for the molecular pairs surrounding the central complex. To then visualize how the pairwise interactions are distributed throughout the crystal lattice, an energy framework is constructed. The calculation relies on the assumption that an interaction energy can be expressed as the sum of its contributions, which are the electrostatic, polarization, dispersion and repulsion energy (Turner *et al.*, 2015). The total energy is then visualized as rods between the molecular centres, and the rod radius indicates the strength of the interaction. The usefulness of the method for assessing cooperativity has already been demonstrated (Reeves *et al.*, 2021). Figs. 7(a)–(e) show the energy frameworks of every compound before and after the transition. The chosen orientation reveals the anisotropy of the interaction network. The strongest energies form continuous columns along the *b* direction, thereby defining a supramolecular backbone for all structures. Thinner cylinders connecting neighbouring columns indicate weaker lateral interactions, which nevertheless contribute to a three-dimensionality. Also noteworthy

is the energy framework of **1-IPh** [Fig. 7(e)], since the solvate was omitted for the calculation in this case. Thus, it shows that the *b*-dominated network persists even in the absence of the energetic contributions of the solvate. Noteworthy is also that the F-, Cl- and Br-containing compounds show differently pronounced tightness of the lateral connecting rods depending on the spin state. In particular, the ClPh-containing compound [Fig. 7(b)] shows fewer interconnections in the contracted unit cell, whereas the lateral connections increase for **1-FPh** and **1-BrPh**. The strong interaction chain along the crystallographic *b* direction suggests that structural changes associated with the spin transition are preferentially propagated along this direction. Closer inspection of the total energy framework shows that the dominant interaction pathway along *b* is mediated by the same neighbouring complex–complex contact highlighted in the Hirshfeld surface and NCI analyses in Fig. 6.

The temperature-dependent pairwise interaction energy of the dominant complex–complex contact, compared with the evolution of the crystallographic *b* axis, is shown in Fig. 8. For all five compounds, the interaction energy follows the same overall trend as the *b* axis, confirming that the contact is directly coupled to the structural response of the system during the transition. The abrupt increase in *b* upon cooling into the LS state is marked by a destabilization of the pairwise interaction energy, which is noteworthy, since the lattice contracts overall. This destabilization may be offset by the formation of a denser interaction framework, as observed for

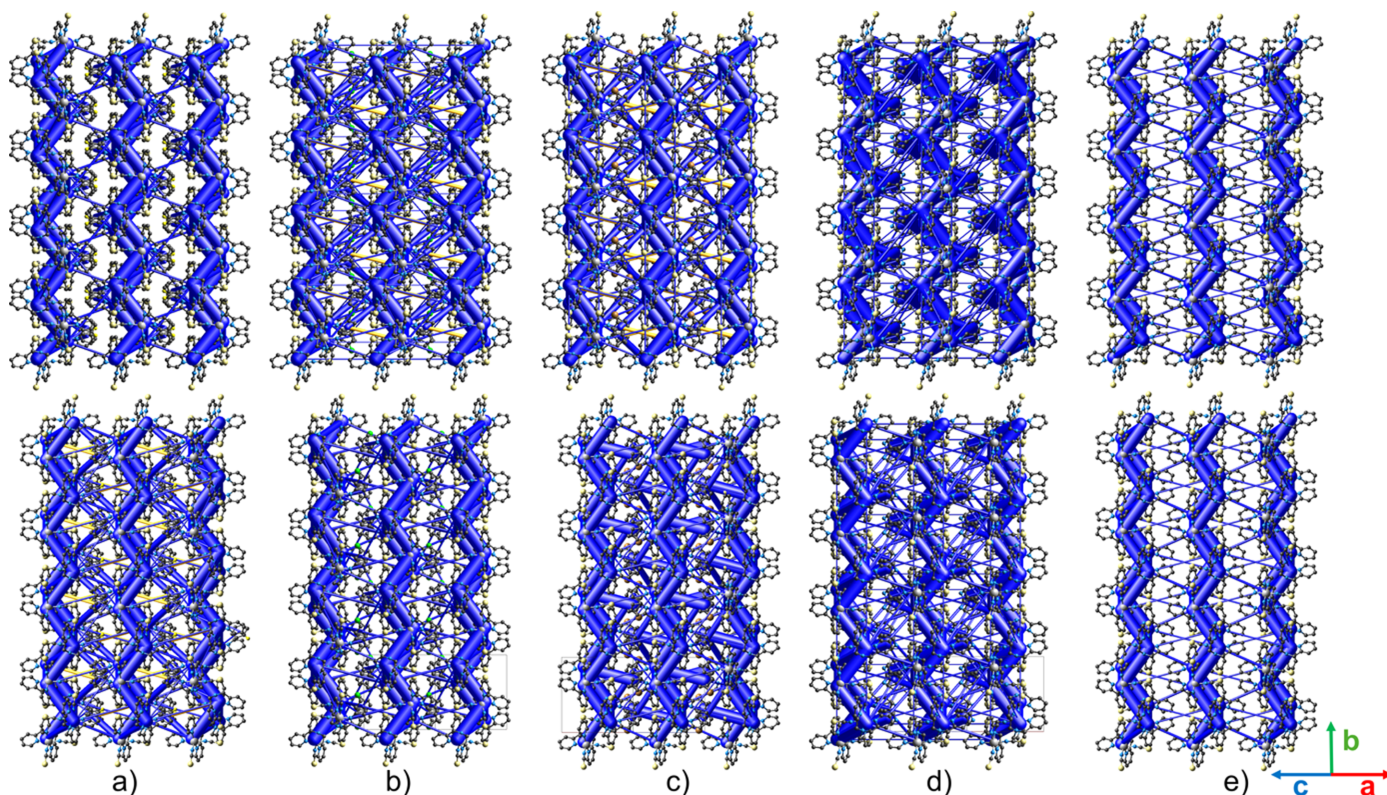


Figure 7

Frameworks of the total energy, with a tube size of 80 and a low-energy cut-off of  $10 \text{ kJ mol}^{-1}$ . Parts (a) to (e) show the F, Cl, Br, py and I variants, respectively; the upper row is before and the lower row is after the transition. H atoms have been omitted for clarity.



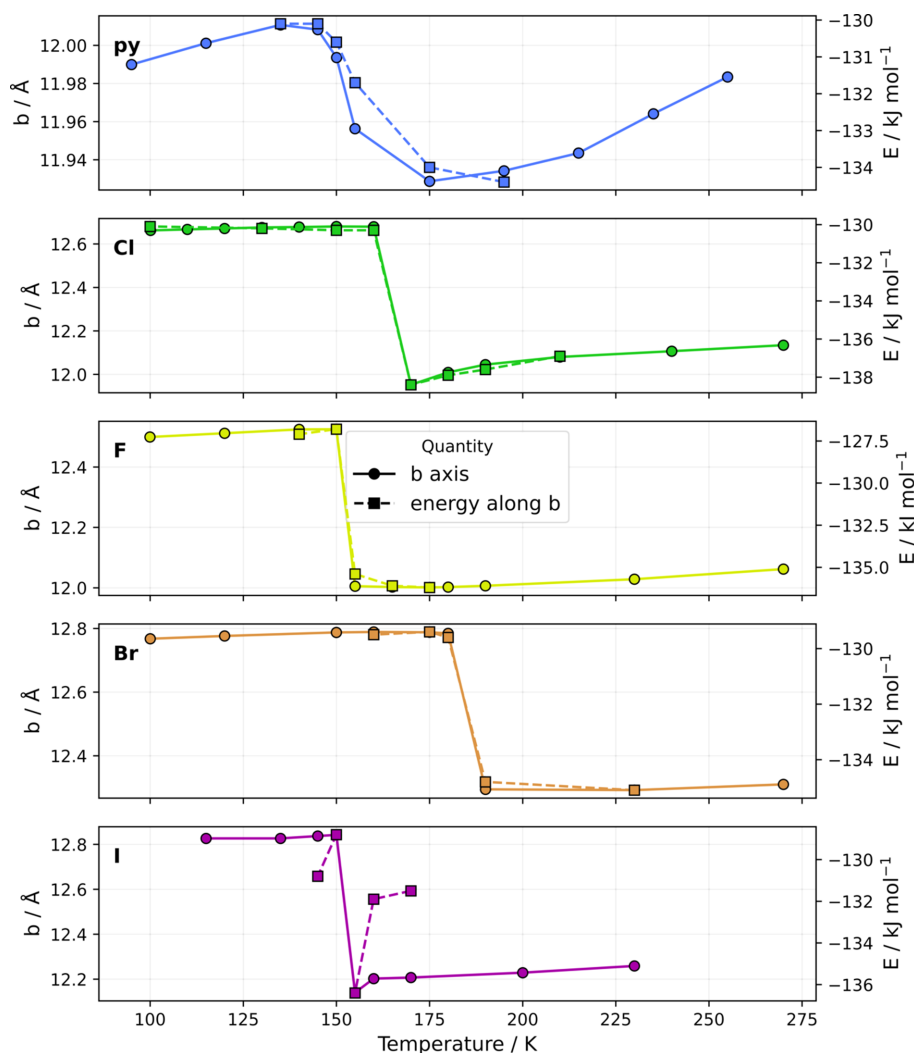


**Figure 8**

The temperature dependence of the *b* unit-cell parameter (circles), together with the pairwise interaction energy of **1** (squares) with its next neighbour generated by the symmetry operation  $(-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2})$ . Errors on *b* are shown but hidden behind the marker.

the LS phases (see Fig. 7). Again, the energy calculation for **1**-IPh excluded the solvate unit, which can explain the more significant deviations compared to the other compounds. Intermolecular interactions between neighbouring complexes which propagate approximately along the crystallographic *a* axis (symmetry code:  $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$ ) are found to be weaker. The contraction of the *a* axis during the transition is then accompanied by an increase in interaction strength between these neighbours (see Fig. S25), except for **1**-BrPh. However, the correlation with the *a* axis is considerably weaker compared to the *b* axis. No corresponding pairwise interaction along the *c* axis has been identified. Analysis of the interaction energies involving the incorporated solvent shows that these interactions are smaller than  $40 \text{ kJ mol}^{-1}$  in all cases and are therefore substantially weaker than the intermolecular interactions between neighbouring complexes (see Figs. S20–S23). In the context of the initially observed differences in transition steepness, the lattice analysis suggests that varying cooperativity of the five compounds originates from subtle changes in how the conserved packing motif transmits

the response of the SCO centres. Even though the strong *b*-axis interaction chain appears to represent a common pathway for structural communication, the varying solvate influences the nature of the transition remarkably, without being directly part of the strongest interaction.

## 5. Conclusion

In summary, variable-temperature single-crystal X-ray diffraction reveals that the five solvate-containing SCO compounds remain essentially isostructural during the transition. The changes in Fe–N bond length, coordination-sphere distortion and lattice parameters demonstrate the solvate-dependent structural response, whereas Hirshfeld surface, NCI and energy-framework analyses provide a more detailed view of how this response is embedded in the interaction network. The compounds reveal a common feature, *i.e.* a dominant pairwise interaction along the crystallographic *b* direction. Calculation of this pairwise energy reveals that the temperature dependence of the interaction energy closely

mirrors that of the *b*-axis lattice parameter, whereas no comparable correlation is observed for the remaining unit-cell parameters. Yet the role of solvent disorder and solvate–solvate interactions is unclear. Also, relying solely on crystallographic data makes it difficult to justify statements about hysteresis and incomplete transitions, and they would need to be fully analysed to understand the nature of the SCO.

## 6. Related literature

The following references are cited in the supporting information: Kratzert *et al.* (2015); Kratzert & Krossing (2018).

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## Conflict of interest

There are no conflicts of interest to declare.

## Data availability

The supplementary material contains additional crystallographic information and data that support the findings of this study.

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## supporting information

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## Analysis of lattice changes and weak interactions in a series of solvatomorphic isostructural spin-crossover compounds

**Daniel Brück, Johannes Bartusel and Florian Kleemiss**

### Computing details

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) bromobenzene monosolvate (br\_100K)**

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.3302$  (3) Å

$b = 12.7675$  (2) Å

$c = 15.2494$  (4) Å

$\beta = 109.948$  (3)°

$V = 2622.65$  (11) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.569$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 39227 reflections

$\theta = 2.0$ – $31.1$ °

$\mu = 2.29$  mm<sup>-1</sup>

$T = 100$  K

Block, red

$0.36 \times 0.26 \times 0.09$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.387$ ,  $T_{\max} = 1.000$

65301 measured reflections

6503 independent reflections

5756 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 28.3$ °,  $\theta_{\min} = 2.1$ °

$h = -19 \rightarrow 19$

$k = -17 \rightarrow 17$

$l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.022$

$wR(F^2) = 0.052$

$S = 1.05$

6503 reflections

335 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 0.5059P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.63$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

|     | <i>x</i>    | <i>y</i>    | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Br3 | 0.36266 (2) | 0.03078 (3) | 0.44510 (2) | 0.03428 (5)                      |

|     |               |               |               |              |
|-----|---------------|---------------|---------------|--------------|
| Fe1 | 0.339329 (11) | 0.338135 (13) | 0.680217 (11) | 0.01413 (4)  |
| S2  | 0.52317 (2)   | 0.04363 (2)   | 0.83154 (2)   | 0.02262 (7)  |
| S1  | 0.25488 (3)   | 0.30051 (3)   | 0.95355 (2)   | 0.02632 (7)  |
| N3  | 0.39018 (7)   | 0.34535 (7)   | 0.57599 (6)   | 0.01590 (19) |
| N1  | 0.28851 (7)   | 0.33582 (8)   | 0.78500 (7)   | 0.0184 (2)   |
| N5  | 0.23033 (7)   | 0.24402 (7)   | 0.60765 (6)   | 0.01606 (19) |
| N4  | 0.24684 (7)   | 0.45189 (7)   | 0.61395 (6)   | 0.01523 (19) |
| N6  | 0.42564 (7)   | 0.45742 (8)   | 0.73858 (6)   | 0.01633 (19) |
| N2  | 0.42840 (7)   | 0.22609 (8)   | 0.74249 (7)   | 0.0195 (2)   |
| C7  | 0.34929 (8)   | 0.42084 (9)   | 0.51287 (7)   | 0.0158 (2)   |
| C19 | 0.37620 (8)   | 0.54936 (9)   | 0.72938 (8)   | 0.0167 (2)   |
| C13 | 0.14391 (8)   | 0.29533 (9)   | 0.56627 (8)   | 0.0164 (2)   |
| C14 | 0.14498 (8)   | 0.40939 (9)   | 0.59137 (8)   | 0.0180 (2)   |
| H18 | 0.12018 (8)   | 0.41862 (9)   | 0.65247 (8)   | 0.0215 (3)*  |
| H17 | 0.09291 (8)   | 0.45352 (9)   | 0.53193 (8)   | 0.0215 (3)*  |
| C18 | 0.42384 (9)   | 0.64286 (10)  | 0.76414 (8)   | 0.0202 (2)   |
| H10 | 0.38174 (9)   | 0.71683 (10)  | 0.75431 (8)   | 0.0243 (3)*  |
| C3  | 0.46272 (9)   | 0.28551 (10)  | 0.56453 (8)   | 0.0201 (2)   |
| H1  | 0.49612 (9)   | 0.22340 (10)  | 0.61594 (8)   | 0.0241 (3)*  |
| C2  | 0.46806 (8)   | 0.15064 (9)   | 0.78005 (8)   | 0.0163 (2)   |
| C12 | 0.05879 (9)   | 0.24480 (9)   | 0.50962 (8)   | 0.0197 (2)   |
| H16 | −0.01014 (9)  | 0.28923 (9)   | 0.47571 (8)   | 0.0236 (3)*  |
| C8  | 0.26730 (9)   | 0.48393 (9)   | 0.52768 (8)   | 0.0185 (2)   |
| H5  | 0.19854 (9)   | 0.47424 (9)   | 0.46622 (8)   | 0.0222 (3)*  |
| H6  | 0.28792 (9)   | 0.56816 (9)   | 0.53297 (8)   | 0.0222 (3)*  |
| C20 | 0.26579 (8)   | 0.54090 (9)   | 0.68099 (8)   | 0.0177 (2)   |
| H11 | 0.23624 (8)   | 0.61480 (9)   | 0.64327 (8)   | 0.0212 (3)*  |
| H12 | 0.22843 (8)   | 0.52665 (9)   | 0.73286 (8)   | 0.0212 (3)*  |
| C1  | 0.27450 (8)   | 0.32183 (9)   | 0.85525 (8)   | 0.0180 (2)   |
| C9  | 0.23337 (9)   | 0.14015 (9)   | 0.59366 (8)   | 0.0193 (2)   |
| H13 | 0.30389 (9)   | 0.09804 (9)   | 0.62666 (8)   | 0.0231 (3)*  |
| C15 | 0.52444 (9)   | 0.45709 (10)  | 0.78183 (8)   | 0.0202 (2)   |
| H7  | 0.56523 (9)   | 0.38272 (10)  | 0.78743 (8)   | 0.0243 (3)*  |
| C10 | 0.15070 (9)   | 0.08398 (10)  | 0.53903 (8)   | 0.0221 (2)   |
| H14 | 0.15546 (9)   | −0.00129 (10) | 0.52971 (8)   | 0.0265 (3)*  |
| C6  | 0.38004 (9)   | 0.43930 (10)  | 0.43694 (8)   | 0.0211 (2)   |
| H4  | 0.34534 (9)   | 0.50189 (10)  | 0.38650 (8)   | 0.0253 (3)*  |
| C11 | 0.06199 (9)   | 0.13712 (10)  | 0.49638 (8)   | 0.0221 (2)   |
| H15 | −0.00473 (9)  | 0.09466 (10)  | 0.45278 (8)   | 0.0266 (3)*  |
| C16 | 0.57702 (9)   | 0.54718 (10)  | 0.81976 (8)   | 0.0233 (3)   |
| H8  | 0.65806 (9)   | 0.54410 (10)  | 0.85573 (8)   | 0.0279 (3)*  |
| C4  | 0.49684 (9)   | 0.29925 (10)  | 0.49046 (8)   | 0.0235 (2)   |
| H2  | 0.55617 (9)   | 0.24876 (10)  | 0.48304 (8)   | 0.0282 (3)*  |
| C22 | 0.24691 (9)   | 0.20684 (10)  | 0.35561 (9)   | 0.0246 (3)   |
| H19 | 0.23283 (9)   | 0.21794 (10)  | 0.42175 (9)   | 0.0295 (3)*  |
| C21 | 0.30466 (9)   | 0.12430 (10)  | 0.34462 (9)   | 0.0228 (2)   |
| C17 | 0.52594 (9)   | 0.64151 (10)  | 0.81173 (8)   | 0.0236 (3)   |
| H9  | 0.56563 (9)   | 0.71393 (10)  | 0.84252 (8)   | 0.0283 (3)*  |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| C5  | 0.45503 (9)  | 0.37776 (11) | 0.42563 (8)  | 0.0246 (3)  |
| H3  | 0.48102 (9)  | 0.39088 (11) | 0.36626 (8)  | 0.0295 (3)* |
| C23 | 0.20692 (9)  | 0.27567 (11) | 0.28105 (10) | 0.0298 (3)  |
| H20 | 0.16136 (9)  | 0.34245 (11) | 0.28846 (10) | 0.0358 (3)* |
| C26 | 0.32274 (11) | 0.10778 (11) | 0.26224 (10) | 0.0322 (3)  |
| H23 | 0.36890 (11) | 0.04132 (11) | 0.25529 (10) | 0.0386 (4)* |
| C24 | 0.22405 (11) | 0.26072 (12) | 0.19759 (10) | 0.0345 (3)  |
| H21 | 0.19217 (11) | 0.31531 (12) | 0.13890 (10) | 0.0414 (4)* |
| C25 | 0.28177 (12) | 0.17633 (12) | 0.18846 (10) | 0.0373 (3)  |
| H22 | 0.29498 (12) | 0.16394 (12) | 0.12207 (10) | 0.0447 (4)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Br3 | 0.03574 (9)  | 0.02662 (8)  | 0.03309 (8)  | 0.00268 (6)   | 0.00215 (6)  | 0.00146 (5)   |
| Fe1 | 0.01462 (8)  | 0.01589 (8)  | 0.01087 (8)  | 0.00204 (6)   | 0.00305 (6)  | 0.00085 (6)   |
| S2  | 0.02025 (14) | 0.02098 (15) | 0.02581 (15) | 0.00426 (11)  | 0.00680 (12) | 0.00762 (12)  |
| S1  | 0.03996 (18) | 0.02489 (16) | 0.01795 (14) | −0.00762 (13) | 0.01485 (13) | −0.00319 (12) |
| N3  | 0.0159 (4)   | 0.0193 (5)   | 0.0122 (4)   | 0.0015 (4)    | 0.0043 (4)   | 0.0006 (4)    |
| N1  | 0.0216 (5)   | 0.0203 (5)   | 0.0135 (5)   | 0.0001 (4)    | 0.0063 (4)   | −0.0001 (4)   |
| N5  | 0.0181 (5)   | 0.0162 (5)   | 0.0132 (4)   | 0.0008 (4)    | 0.0044 (4)   | −0.0000 (4)   |
| N4  | 0.0147 (4)   | 0.0157 (5)   | 0.0131 (4)   | 0.0020 (4)    | 0.0019 (4)   | −0.0002 (3)   |
| N6  | 0.0152 (5)   | 0.0191 (5)   | 0.0123 (4)   | 0.0003 (4)    | 0.0017 (4)   | 0.0005 (4)    |
| N2  | 0.0203 (5)   | 0.0204 (5)   | 0.0158 (5)   | 0.0049 (4)    | 0.0036 (4)   | 0.0035 (4)    |
| C7  | 0.0161 (5)   | 0.0181 (5)   | 0.0119 (5)   | −0.0003 (4)   | 0.0033 (4)   | 0.0005 (4)    |
| C19 | 0.0173 (5)   | 0.0181 (6)   | 0.0128 (5)   | −0.0001 (4)   | 0.0029 (4)   | −0.0007 (4)   |
| C13 | 0.0152 (5)   | 0.0172 (5)   | 0.0150 (5)   | −0.0005 (4)   | 0.0031 (4)   | −0.0009 (4)   |
| C14 | 0.0149 (5)   | 0.0186 (6)   | 0.0185 (5)   | 0.0016 (4)    | 0.0032 (4)   | −0.0019 (4)   |
| C18 | 0.0207 (6)   | 0.0205 (6)   | 0.0174 (5)   | −0.0021 (5)   | 0.0039 (5)   | −0.0032 (4)   |
| C3  | 0.0181 (5)   | 0.0247 (6)   | 0.0174 (5)   | 0.0053 (5)    | 0.0060 (4)   | 0.0019 (5)    |
| C2  | 0.0155 (5)   | 0.0180 (6)   | 0.0141 (5)   | 0.0023 (4)    | 0.0033 (4)   | 0.0025 (4)    |
| C12 | 0.0186 (6)   | 0.0205 (6)   | 0.0174 (5)   | −0.0023 (5)   | 0.0029 (5)   | −0.0008 (4)   |
| C8  | 0.0193 (5)   | 0.0196 (6)   | 0.0146 (5)   | 0.0039 (4)    | 0.0034 (4)   | 0.0031 (4)    |
| C20 | 0.0172 (5)   | 0.0170 (5)   | 0.0172 (5)   | 0.0013 (4)    | 0.0036 (4)   | −0.0025 (4)   |
| C1  | 0.0219 (5)   | 0.0174 (5)   | 0.0150 (5)   | −0.0016 (4)   | 0.0066 (4)   | −0.0013 (4)   |
| C9  | 0.0241 (6)   | 0.0168 (5)   | 0.0165 (5)   | 0.0013 (5)    | 0.0064 (5)   | −0.0000 (4)   |
| C15 | 0.0157 (5)   | 0.0254 (6)   | 0.0162 (5)   | 0.0007 (5)    | 0.0011 (4)   | 0.0018 (4)    |
| C10 | 0.0292 (6)   | 0.0168 (6)   | 0.0197 (6)   | −0.0028 (5)   | 0.0077 (5)   | −0.0023 (4)   |
| C6  | 0.0230 (6)   | 0.0255 (6)   | 0.0149 (5)   | −0.0005 (5)   | 0.0067 (5)   | 0.0032 (5)    |
| C11 | 0.0246 (6)   | 0.0213 (6)   | 0.0184 (6)   | −0.0049 (5)   | 0.0046 (5)   | −0.0028 (5)   |
| C16 | 0.0175 (6)   | 0.0293 (7)   | 0.0190 (6)   | −0.0027 (5)   | 0.0010 (5)   | 0.0003 (5)    |
| C4  | 0.0203 (6)   | 0.0322 (7)   | 0.0195 (6)   | 0.0032 (5)    | 0.0089 (5)   | −0.0008 (5)   |
| C22 | 0.0232 (6)   | 0.0257 (6)   | 0.0266 (6)   | −0.0003 (5)   | 0.0109 (5)   | −0.0039 (5)   |
| C21 | 0.0210 (6)   | 0.0215 (6)   | 0.0254 (6)   | −0.0009 (5)   | 0.0072 (5)   | −0.0029 (5)   |
| C17 | 0.0226 (6)   | 0.0256 (6)   | 0.0194 (6)   | −0.0058 (5)   | 0.0030 (5)   | −0.0028 (5)   |
| C5  | 0.0250 (6)   | 0.0337 (7)   | 0.0175 (6)   | −0.0001 (5)   | 0.0104 (5)   | 0.0013 (5)    |
| C23 | 0.0199 (6)   | 0.0265 (7)   | 0.0400 (8)   | 0.0002 (5)    | 0.0063 (6)   | 0.0011 (6)    |
| C26 | 0.0396 (8)   | 0.0274 (7)   | 0.0363 (8)   | −0.0035 (6)   | 0.0217 (7)   | −0.0077 (6)   |

|     |            |            |            |             |             |             |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C24 | 0.0320 (7) | 0.0354 (8) | 0.0279 (7) | −0.0108 (6) | −0.0003 (6) | 0.0050 (6)  |
| C25 | 0.0526 (9) | 0.0371 (8) | 0.0267 (7) | −0.0140 (7) | 0.0195 (7)  | −0.0061 (6) |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Br3—C21   | 1.8962 (13) | C12—H16     | 1.1030      |
| Fe1—N3    | 1.9636 (9)  | C12—C11     | 1.3925 (17) |
| Fe1—N1    | 1.9694 (10) | C8—H5       | 1.1110      |
| Fe1—N5    | 1.9822 (10) | C8—H6       | 1.1110      |
| Fe1—N4    | 1.9933 (9)  | C20—H11     | 1.1110      |
| Fe1—N6    | 1.9723 (10) | C20—H12     | 1.1110      |
| Fe1—N2    | 1.9365 (10) | C9—H13      | 1.1030      |
| S2—C2     | 1.6383 (12) | C9—C10      | 1.3923 (17) |
| S1—C1     | 1.6394 (12) | C15—H7      | 1.1030      |
| N3—C7     | 1.3468 (14) | C15—C16     | 1.3885 (17) |
| N3—C3     | 1.3487 (15) | C10—H14     | 1.1030      |
| N1—C1     | 1.1689 (15) | C10—C11     | 1.3911 (18) |
| N5—C13    | 1.3520 (14) | C6—H4       | 1.1030      |
| N5—C9     | 1.3462 (15) | C6—C5       | 1.3886 (18) |
| N4—C14    | 1.4835 (14) | C11—H15     | 1.1030      |
| N4—C8     | 1.4990 (14) | C16—H8      | 1.1030      |
| N4—C20    | 1.4903 (14) | C16—C17     | 1.3928 (18) |
| N6—C19    | 1.3534 (15) | C4—H2       | 1.1030      |
| N6—C15    | 1.3425 (15) | C4—C5       | 1.3918 (18) |
| N2—C2     | 1.1649 (15) | C22—H19     | 1.1030      |
| C7—C8     | 1.5038 (15) | C22—C21     | 1.3850 (17) |
| C7—C6     | 1.3924 (15) | C22—C23     | 1.3960 (19) |
| C19—C18   | 1.3876 (16) | C21—C26     | 1.3827 (18) |
| C19—C20   | 1.5042 (15) | C17—H9      | 1.1030      |
| C13—C14   | 1.5045 (16) | C5—H3       | 1.1030      |
| C13—C12   | 1.3897 (16) | C23—H20     | 1.1030      |
| C14—H18   | 1.1110      | C23—C24     | 1.389 (2)   |
| C14—H17   | 1.1110      | C26—H23     | 1.1030      |
| C18—H10   | 1.1030      | C26—C25     | 1.388 (2)   |
| C18—C17   | 1.3940 (17) | C24—H21     | 1.1030      |
| C3—H1     | 1.1030      | C24—C25     | 1.394 (2)   |
| C3—C4     | 1.3865 (16) | C25—H22     | 1.1030      |
| N1—Fe1—N3 | 178.17 (4)  | H5—C8—N4    | 109.10 (6)  |
| N5—Fe1—N3 | 90.91 (4)   | H5—C8—C7    | 109.10 (6)  |
| N5—Fe1—N1 | 90.28 (4)   | H6—C8—N4    | 109.10 (6)  |
| N4—Fe1—N3 | 85.50 (4)   | H6—C8—C7    | 109.10 (6)  |
| N4—Fe1—N1 | 93.23 (4)   | H6—C8—H5    | 107.8       |
| N4—Fe1—N5 | 84.13 (4)   | C19—C20—N4  | 107.83 (9)  |
| N6—Fe1—N3 | 88.87 (4)   | H11—C20—N4  | 110.14 (6)  |
| N6—Fe1—N1 | 89.66 (4)   | H11—C20—C19 | 110.14 (6)  |
| N6—Fe1—N5 | 166.76 (4)  | H12—C20—N4  | 110.14 (6)  |
| N6—Fe1—N4 | 82.66 (4)   | H12—C20—C19 | 110.14 (6)  |



|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N2—Fe1—N3   | 93.85 (4)   | H12—C20—H11 | 108.5       |
| N2—Fe1—N1   | 87.44 (4)   | N1—C1—S1    | 179.23 (11) |
| N2—Fe1—N5   | 95.05 (4)   | H13—C9—N5   | 118.82 (7)  |
| N2—Fe1—N4   | 178.94 (4)  | C10—C9—N5   | 122.36 (11) |
| N2—Fe1—N6   | 98.17 (4)   | C10—C9—H13  | 118.82 (7)  |
| C7—N3—Fe1   | 114.89 (7)  | H7—C15—N6   | 118.88 (7)  |
| C3—N3—Fe1   | 126.41 (8)  | C16—C15—N6  | 122.24 (11) |
| C3—N3—C7    | 118.65 (10) | C16—C15—H7  | 118.88 (7)  |
| C1—N1—Fe1   | 166.52 (10) | H14—C10—C9  | 120.53 (7)  |
| C13—N5—Fe1  | 112.87 (8)  | C11—C10—C9  | 118.93 (11) |
| C9—N5—Fe1   | 128.59 (8)  | C11—C10—H14 | 120.53 (7)  |
| C9—N5—C13   | 118.51 (10) | H4—C6—C7    | 120.48 (7)  |
| C14—N4—Fe1  | 106.41 (7)  | C5—C6—C7    | 119.04 (11) |
| C8—N4—Fe1   | 110.40 (7)  | C5—C6—H4    | 120.48 (7)  |
| C8—N4—C14   | 111.57 (9)  | C10—C11—C12 | 118.94 (11) |
| C20—N4—Fe1  | 105.89 (7)  | H15—C11—C12 | 120.53 (7)  |
| C20—N4—C14  | 111.88 (9)  | H15—C11—C10 | 120.53 (7)  |
| C20—N4—C8   | 110.46 (9)  | H8—C16—C15  | 120.48 (7)  |
| C19—N6—Fe1  | 113.42 (7)  | C17—C16—C15 | 119.05 (11) |
| C15—N6—Fe1  | 127.82 (8)  | C17—C16—H8  | 120.48 (7)  |
| C15—N6—C19  | 118.72 (10) | H2—C4—C3    | 120.54 (7)  |
| C2—N2—Fe1   | 168.93 (10) | C5—C4—C3    | 118.93 (11) |
| C8—C7—N3    | 116.60 (10) | C5—C4—H2    | 120.54 (7)  |
| C6—C7—N3    | 122.11 (11) | C21—C22—H19 | 120.77 (8)  |
| C6—C7—C8    | 121.29 (10) | C23—C22—H19 | 120.77 (8)  |
| C18—C19—N6  | 122.33 (11) | C23—C22—C21 | 118.46 (12) |
| C20—C19—N6  | 114.48 (10) | C22—C21—Br3 | 119.78 (10) |
| C20—C19—C18 | 123.18 (10) | C26—C21—Br3 | 118.15 (10) |
| C14—C13—N5  | 114.96 (10) | C26—C21—C22 | 122.07 (13) |
| C12—C13—N5  | 122.35 (11) | C16—C17—C18 | 118.89 (12) |
| C12—C13—C14 | 122.56 (10) | H9—C17—C18  | 120.55 (7)  |
| C13—C14—N4  | 109.69 (9)  | H9—C17—C16  | 120.55 (7)  |
| H18—C14—N4  | 109.73 (6)  | C4—C5—C6    | 118.89 (11) |
| H18—C14—C13 | 109.73 (6)  | H3—C5—C6    | 120.56 (7)  |
| H17—C14—N4  | 109.73 (6)  | H3—C5—C4    | 120.56 (7)  |
| H17—C14—C13 | 109.73 (6)  | H20—C23—C22 | 119.73 (8)  |
| H17—C14—H18 | 108.2       | C24—C23—C22 | 120.53 (13) |
| H10—C18—C19 | 120.64 (7)  | C24—C23—H20 | 119.73 (9)  |
| C17—C18—C19 | 118.71 (11) | H23—C26—C21 | 120.60 (8)  |
| C17—C18—H10 | 120.64 (7)  | C25—C26—C21 | 118.80 (13) |
| H1—C3—N3    | 118.81 (6)  | C25—C26—H23 | 120.60 (9)  |
| C4—C3—N3    | 122.39 (11) | H21—C24—C23 | 120.19 (9)  |
| C4—C3—H1    | 118.81 (7)  | C25—C24—C23 | 119.61 (13) |
| N2—C2—S2    | 179.22 (11) | C25—C24—H21 | 120.19 (9)  |
| H16—C12—C13 | 120.56 (7)  | C24—C25—C26 | 120.52 (13) |
| C11—C12—C13 | 118.89 (11) | H22—C25—C26 | 119.74 (9)  |
| C11—C12—H16 | 120.56 (7)  | H22—C25—C24 | 119.74 (9)  |
| C7—C8—N4    | 112.48 (9)  |             |             |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Br3—C21—C22—C23 | 178.67 (10)  | C7—C8—N4—C20    | 118.19 (10)  |
| Br3—C21—C26—C25 | −179.23 (11) | C7—C6—C5—C4     | 0.24 (14)    |
| Fe1—N3—C7—C8    | −3.51 (9)    | C19—N6—C15—C16  | 1.95 (12)    |
| Fe1—N3—C7—C6    | 177.24 (8)   | C19—C18—C17—C16 | 2.29 (13)    |
| Fe1—N3—C3—C4    | −177.15 (10) | C19—C20—N4—C14  | 156.97 (9)   |
| Fe1—N5—C13—C14  | −6.06 (8)    | C19—C20—N4—C8   | −78.09 (10)  |
| Fe1—N5—C13—C12  | 177.92 (7)   | C13—N5—C9—C10   | −0.63 (12)   |
| Fe1—N5—C9—C10   | −178.67 (9)  | C13—C14—N4—C8   | 83.77 (10)   |
| Fe1—N4—C14—C13  | −36.70 (7)   | C13—C14—N4—C20  | −151.90 (9)  |
| Fe1—N4—C8—C7    | 1.41 (7)     | C13—C12—C11—C10 | −1.06 (13)   |
| Fe1—N4—C20—C19  | 41.44 (7)    | C14—C13—N5—C9   | 175.60 (9)   |
| Fe1—N6—C19—C18  | −178.82 (8)  | C14—C13—C12—C11 | −174.46 (11) |
| Fe1—N6—C19—C20  | 2.78 (8)     | C18—C19—N6—C15  | −0.88 (13)   |
| Fe1—N6—C15—C16  | 179.55 (10)  | C18—C17—C16—C15 | −1.31 (14)   |
| N3—C7—C8—N4     | 1.32 (11)    | C3—N3—C7—C8     | 178.78 (10)  |
| N3—C7—C6—C5     | 0.22 (13)    | C3—N3—C7—C6     | −0.47 (12)   |
| N3—C3—C4—C5     | 0.18 (14)    | C3—C4—C5—C6     | −0.43 (14)   |
| N5—C13—C14—N4   | 29.16 (10)   | C12—C13—N5—C9   | −0.42 (13)   |
| N5—C13—C12—C11  | 1.27 (13)    | C12—C11—C10—C9  | 0.08 (13)    |
| N5—C9—C10—C11   | 0.79 (13)    | C8—C7—C6—C5     | −179.00 (11) |
| N4—C14—C13—C12  | −154.82 (9)  | C20—C19—N6—C15  | −179.28 (9)  |
| N4—C8—C7—C6     | −179.43 (9)  | C20—C19—C18—C17 | 177.02 (11)  |
| N4—C20—C19—N6   | −29.95 (10)  | C22—C21—C26—C25 | 0.12 (15)    |
| N4—C20—C19—C18  | 151.67 (9)   | C22—C23—C24—C25 | −0.08 (15)   |
| N6—C19—C18—C17  | −1.24 (13)   | C21—C22—C23—C24 | 0.65 (14)    |
| N6—C15—C16—C17  | −0.85 (14)   | C21—C26—C25—C24 | 0.47 (16)    |
| C7—N3—C3—C4     | 0.27 (12)    | C23—C22—C21—C26 | −0.68 (15)   |
| C7—C8—N4—C14    | −116.70 (10) | C23—C24—C25—C26 | −0.49 (15)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate (br\_120K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.3465$  (3) Å

$b = 12.7762$  (2) Å

$c = 15.2630$  (4) Å

$\beta = 109.959$  (3)°

$V = 2629.58$  (11) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.565$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 37292 reflections

$\theta = 2.0$ – $31.2^\circ$

$\mu = 2.28$  mm<sup>−1</sup>

$T = 120$  K

Block, red

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.378$ ,  $T_{\max} = 1.000$

65396 measured reflections  
 6520 independent reflections  
 5641 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 28.3^\circ$ ,  $\theta_{\text{min}} = 2.1^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -17 \rightarrow 17$   
 $l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.058$   
 $S = 1.04$   
 6520 reflections  
 335 parameters  
 0 restraints

43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 0.4987P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.81 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Br3 | 0.63721 (3)   | 0.03063 (3)   | 0.55557 (3)   | 0.04095 (6)                      |
| Fe1 | 0.660771 (12) | 0.337967 (13) | 0.319716 (11) | 0.01630 (5)                      |
| S2  | 0.47676 (2)   | 0.04403 (3)   | 0.16832 (2)   | 0.02609 (8)                      |
| S1  | 0.74503 (3)   | 0.30055 (3)   | 0.04662 (2)   | 0.03095 (8)                      |
| N3  | 0.60997 (7)   | 0.34529 (8)   | 0.42390 (7)   | 0.0183 (2)                       |
| N5  | 0.76967 (7)   | 0.24384 (8)   | 0.39216 (7)   | 0.0184 (2)                       |
| N1  | 0.71151 (8)   | 0.33566 (8)   | 0.21499 (7)   | 0.0210 (2)                       |
| N4  | 0.75318 (7)   | 0.45162 (8)   | 0.38593 (7)   | 0.0171 (2)                       |
| N6  | 0.57443 (8)   | 0.45710 (8)   | 0.26144 (7)   | 0.0190 (2)                       |
| N2  | 0.57169 (8)   | 0.22597 (9)   | 0.25750 (7)   | 0.0224 (2)                       |
| C7  | 0.65088 (9)   | 0.42061 (10)  | 0.48697 (8)   | 0.0185 (2)                       |
| C19 | 0.62409 (9)   | 0.54909 (10)  | 0.27076 (8)   | 0.0190 (2)                       |
| C13 | 0.85595 (9)   | 0.29542 (10)  | 0.43342 (8)   | 0.0186 (2)                       |
| C14 | 0.85492 (9)   | 0.40909 (10)  | 0.40827 (9)   | 0.0207 (2)                       |
| H18 | 0.87947 (9)   | 0.41817 (10)  | 0.34707 (9)   | 0.0248 (3)*                      |
| H17 | 0.90711 (9)   | 0.45323 (10)  | 0.46753 (9)   | 0.0248 (3)*                      |
| C2  | 0.53207 (9)   | 0.15067 (10)  | 0.21991 (8)   | 0.0187 (2)                       |
| C18 | 0.57617 (10)  | 0.64257 (10)  | 0.23625 (9)   | 0.0233 (3)                       |
| H10 | 0.61807 (10)  | 0.71661 (10)  | 0.24638 (9)   | 0.0280 (3)*                      |
| C12 | 0.94112 (9)   | 0.24491 (10)  | 0.48981 (9)   | 0.0226 (3)                       |
| H16 | 1.01012 (9)   | 0.28925 (10)  | 0.52328 (9)   | 0.0271 (3)*                      |
| C3  | 0.53740 (9)   | 0.28550 (11)  | 0.43538 (9)   | 0.0230 (3)                       |
| H1  | 0.50401 (9)   | 0.22335 (11)  | 0.38410 (9)   | 0.0276 (3)*                      |
| C1  | 0.72566 (9)   | 0.32171 (9)   | 0.14484 (9)   | 0.0206 (2)                       |
| C8  | 0.73284 (9)   | 0.48346 (10)  | 0.47219 (8)   | 0.0214 (2)                       |
| H5  | 0.80154 (9)   | 0.47356 (10)  | 0.53355 (8)   | 0.0257 (3)*                      |
| H6  | 0.71242 (9)   | 0.56769 (10)  | 0.46707 (8)   | 0.0257 (3)*                      |
| C9  | 0.76659 (10)  | 0.14025 (10)  | 0.40618 (9)   | 0.0222 (3)                       |
| H13 | 0.69610 (10)  | 0.09822 (10)  | 0.37324 (9)   | 0.0266 (3)*                      |
| C20 | 0.73412 (9)   | 0.54063 (10)  | 0.31901 (9)   | 0.0201 (2)                       |
| H11 | 0.76361 (9)   | 0.61446 (10)  | 0.35677 (9)   | 0.0242 (3)*                      |
| H12 | 0.77143 (9)   | 0.52653 (10)  | 0.26717 (9)   | 0.0242 (3)*                      |

|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| C10 | 0.84910 (10) | 0.08408 (11)  | 0.46075 (9)  | 0.0257 (3)  |
| H14 | 0.84432 (10) | −0.00112 (11) | 0.47012 (9)  | 0.0308 (3)* |
| C6  | 0.62012 (10) | 0.43908 (11)  | 0.56271 (9)  | 0.0244 (3)  |
| H4  | 0.65484 (10) | 0.50159 (11)  | 0.61313 (9)  | 0.0293 (3)* |
| C11 | 0.93784 (10) | 0.13732 (11)  | 0.50332 (9)  | 0.0255 (3)  |
| H15 | 1.00446 (10) | 0.09495 (11)  | 0.54705 (9)  | 0.0306 (3)* |
| C15 | 0.47582 (9)  | 0.45677 (11)  | 0.21837 (9)  | 0.0232 (3)  |
| H7  | 0.43505 (9)  | 0.38247 (11)  | 0.21282 (9)  | 0.0278 (3)* |
| C16 | 0.42340 (10) | 0.54674 (11)  | 0.18057 (9)  | 0.0265 (3)  |
| H8  | 0.34244 (10) | 0.54368 (11)  | 0.14459 (9)  | 0.0318 (3)* |
| C4  | 0.50337 (10) | 0.29950 (11)  | 0.50939 (9)  | 0.0274 (3)  |
| H2  | 0.44403 (10) | 0.24920 (11)  | 0.51682 (9)  | 0.0328 (3)* |
| C22 | 0.75287 (10) | 0.20630 (11)  | 0.64473 (10) | 0.0287 (3)  |
| H19 | 0.76699 (10) | 0.21717 (11)  | 0.57866 (10) | 0.0344 (3)* |
| C17 | 0.47450 (10) | 0.64116 (11)  | 0.18870 (9)  | 0.0273 (3)  |
| H9  | 0.43486 (10) | 0.71352 (11)  | 0.15791 (9)  | 0.0328 (3)* |
| C21 | 0.69518 (10) | 0.12427 (11)  | 0.65560 (10) | 0.0264 (3)  |
| C5  | 0.54508 (10) | 0.37771 (12)  | 0.57397 (9)  | 0.0287 (3)  |
| H3  | 0.51909 (10) | 0.39093 (12)  | 0.63325 (9)  | 0.0345 (4)* |
| C23 | 0.79298 (10) | 0.27521 (12)  | 0.71903 (12) | 0.0357 (3)  |
| H20 | 0.83867 (10) | 0.34176 (12)  | 0.71156 (12) | 0.0428 (4)* |
| C26 | 0.67720 (12) | 0.10797 (12)  | 0.73794 (11) | 0.0370 (3)  |
| H23 | 0.63107 (12) | 0.04160 (12)  | 0.74493 (11) | 0.0444 (4)* |
| C24 | 0.77569 (12) | 0.26046 (13)  | 0.80234 (11) | 0.0400 (4)  |
| H21 | 0.80748 (12) | 0.31517 (13)  | 0.86088 (11) | 0.0480 (5)* |
| C25 | 0.71815 (14) | 0.17648 (14)  | 0.81167 (12) | 0.0433 (4)  |
| H22 | 0.70498 (14) | 0.16424 (14)  | 0.87803 (12) | 0.0520 (5)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Br3 | 0.04290 (10) | 0.03154 (9)  | 0.03947 (10) | −0.00316 (7)  | 0.00246 (7)  | −0.00184 (6)  |
| Fe1 | 0.01703 (9)  | 0.01830 (9)  | 0.01253 (8)  | −0.00248 (6)  | 0.00371 (6)  | −0.00108 (6)  |
| S2  | 0.02319 (16) | 0.02429 (16) | 0.02999 (17) | −0.00495 (12) | 0.00804 (13) | −0.00894 (13) |
| S1  | 0.0475 (2)   | 0.02910 (18) | 0.02109 (16) | 0.00942 (16)  | 0.01791 (15) | 0.00401 (13)  |
| N3  | 0.0184 (5)   | 0.0221 (5)   | 0.0139 (5)   | −0.0028 (4)   | 0.0048 (4)   | −0.0012 (4)   |
| N5  | 0.0203 (5)   | 0.0184 (5)   | 0.0157 (5)   | −0.0005 (4)   | 0.0052 (4)   | 0.0001 (4)    |
| N1  | 0.0244 (5)   | 0.0233 (5)   | 0.0159 (5)   | 0.0001 (4)    | 0.0076 (4)   | −0.0001 (4)   |
| N4  | 0.0170 (5)   | 0.0174 (5)   | 0.0150 (5)   | −0.0028 (4)   | 0.0027 (4)   | 0.0001 (4)    |
| N6  | 0.0182 (5)   | 0.0219 (5)   | 0.0145 (5)   | −0.0013 (4)   | 0.0024 (4)   | −0.0012 (4)   |
| N2  | 0.0232 (5)   | 0.0229 (6)   | 0.0185 (5)   | −0.0057 (4)   | 0.0040 (4)   | −0.0042 (4)   |
| C7  | 0.0186 (5)   | 0.0220 (6)   | 0.0133 (5)   | 0.0008 (5)    | 0.0032 (4)   | −0.0004 (4)   |
| C19 | 0.0186 (6)   | 0.0208 (6)   | 0.0154 (5)   | 0.0002 (5)    | 0.0029 (5)   | 0.0018 (4)    |
| C13 | 0.0186 (6)   | 0.0191 (6)   | 0.0168 (5)   | 0.0003 (5)    | 0.0043 (5)   | 0.0011 (4)    |
| C14 | 0.0176 (5)   | 0.0199 (6)   | 0.0222 (6)   | −0.0016 (5)   | 0.0037 (5)   | 0.0020 (5)    |
| C2  | 0.0178 (5)   | 0.0209 (6)   | 0.0158 (5)   | −0.0027 (5)   | 0.0038 (4)   | −0.0028 (5)   |
| C18 | 0.0237 (6)   | 0.0236 (6)   | 0.0202 (6)   | 0.0025 (5)    | 0.0045 (5)   | 0.0033 (5)    |
| C12 | 0.0208 (6)   | 0.0241 (7)   | 0.0199 (6)   | 0.0030 (5)    | 0.0032 (5)   | 0.0012 (5)    |



|     |             |            |            |             |             |             |
|-----|-------------|------------|------------|-------------|-------------|-------------|
| C3  | 0.0208 (6)  | 0.0288 (7) | 0.0192 (6) | −0.0046 (5) | 0.0066 (5)  | −0.0009 (5) |
| C1  | 0.0255 (6)  | 0.0198 (6) | 0.0170 (6) | 0.0016 (5)  | 0.0078 (5)  | 0.0012 (5)  |
| C8  | 0.0233 (6)  | 0.0228 (6) | 0.0162 (6) | −0.0044 (5) | 0.0043 (5)  | −0.0033 (5) |
| C9  | 0.0287 (7)  | 0.0185 (6) | 0.0190 (6) | −0.0018 (5) | 0.0077 (5)  | 0.0003 (5)  |
| C20 | 0.0194 (6)  | 0.0197 (6) | 0.0194 (6) | −0.0015 (5) | 0.0042 (5)  | 0.0026 (5)  |
| C10 | 0.0337 (7)  | 0.0198 (6) | 0.0230 (6) | 0.0029 (5)  | 0.0089 (6)  | 0.0031 (5)  |
| C6  | 0.0267 (6)  | 0.0296 (7) | 0.0170 (6) | 0.0013 (5)  | 0.0075 (5)  | −0.0031 (5) |
| C11 | 0.0289 (7)  | 0.0243 (7) | 0.0214 (6) | 0.0059 (5)  | 0.0062 (5)  | 0.0034 (5)  |
| C15 | 0.0181 (6)  | 0.0287 (7) | 0.0190 (6) | −0.0009 (5) | 0.0016 (5)  | −0.0030 (5) |
| C16 | 0.0203 (6)  | 0.0345 (8) | 0.0201 (6) | 0.0035 (5)  | 0.0009 (5)  | 0.0001 (5)  |
| C4  | 0.0242 (6)  | 0.0366 (7) | 0.0230 (6) | −0.0035 (6) | 0.0103 (5)  | 0.0017 (6)  |
| C22 | 0.0267 (7)  | 0.0301 (7) | 0.0312 (7) | 0.0010 (6)  | 0.0124 (6)  | 0.0056 (6)  |
| C17 | 0.0246 (7)  | 0.0313 (7) | 0.0223 (6) | 0.0072 (6)  | 0.0031 (5)  | 0.0046 (5)  |
| C21 | 0.0243 (6)  | 0.0244 (6) | 0.0304 (7) | 0.0014 (5)  | 0.0092 (6)  | 0.0040 (5)  |
| C5  | 0.0289 (7)  | 0.0399 (8) | 0.0202 (6) | 0.0003 (6)  | 0.0121 (6)  | −0.0017 (6) |
| C23 | 0.0235 (7)  | 0.0312 (8) | 0.0487 (9) | −0.0005 (6) | 0.0076 (7)  | −0.0002 (7) |
| C26 | 0.0456 (9)  | 0.0309 (8) | 0.0425 (9) | 0.0041 (7)  | 0.0253 (8)  | 0.0091 (7)  |
| C24 | 0.0387 (8)  | 0.0395 (9) | 0.0323 (8) | 0.0127 (7)  | −0.0003 (7) | −0.0053 (7) |
| C25 | 0.0625 (11) | 0.0408 (9) | 0.0321 (8) | 0.0161 (8)  | 0.0232 (8)  | 0.0070 (7)  |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| Br3—C21 | 1.8939 (15) | C3—H1   | 1.1030      |
| Fe1—N3  | 1.9646 (10) | C3—C4   | 1.3867 (17) |
| Fe1—N5  | 1.9825 (10) | C8—H5   | 1.1110      |
| Fe1—N1  | 1.9698 (11) | C8—H6   | 1.1110      |
| Fe1—N4  | 1.9931 (10) | C9—H13  | 1.1030      |
| Fe1—N6  | 1.9724 (11) | C9—C10  | 1.3918 (18) |
| Fe1—N2  | 1.9375 (11) | C20—H11 | 1.1110      |
| S2—C2   | 1.6368 (13) | C20—H12 | 1.1110      |
| S1—C1   | 1.6377 (13) | C10—H14 | 1.1030      |
| N3—C7   | 1.3459 (15) | C10—C11 | 1.3932 (19) |
| N3—C3   | 1.3500 (16) | C6—H4   | 1.1030      |
| N5—C13  | 1.3531 (15) | C6—C5   | 1.3891 (19) |
| N5—C9   | 1.3438 (16) | C11—H15 | 1.1030      |
| N1—C1   | 1.1695 (16) | C15—H7  | 1.1030      |
| N4—C14  | 1.4840 (15) | C15—C16 | 1.3869 (19) |
| N4—C8   | 1.4987 (15) | C16—H8  | 1.1030      |
| N4—C20  | 1.4902 (15) | C16—C17 | 1.395 (2)   |
| N6—C19  | 1.3564 (16) | C4—H2   | 1.1030      |
| N6—C15  | 1.3411 (16) | C4—C5   | 1.388 (2)   |
| N2—C2   | 1.1641 (16) | C22—H19 | 1.1030      |
| C7—C8   | 1.5031 (17) | C22—C21 | 1.3801 (19) |
| C7—C6   | 1.3911 (16) | C22—C23 | 1.395 (2)   |
| C19—C18 | 1.3889 (17) | C17—H9  | 1.1030      |
| C19—C20 | 1.5007 (17) | C21—C26 | 1.382 (2)   |
| C13—C14 | 1.5009 (17) | C5—H3   | 1.1030      |
| C13—C12 | 1.3901 (17) | C23—H20 | 1.1030      |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C14—H18     | 1.1110      | C23—C24     | 1.389 (2)   |
| C14—H17     | 1.1110      | C26—H23     | 1.1030      |
| C18—H10     | 1.1030      | C26—C25     | 1.388 (2)   |
| C18—C17     | 1.3900 (18) | C24—H21     | 1.1030      |
| C12—H16     | 1.1030      | C24—C25     | 1.390 (3)   |
| C12—C11     | 1.3931 (18) | C25—H22     | 1.1030      |
| N5—Fe1—N3   | 90.97 (4)   | C7—C8—N4    | 112.56 (10) |
| N1—Fe1—N3   | 178.13 (4)  | H5—C8—N4    | 109.08 (6)  |
| N1—Fe1—N5   | 90.25 (4)   | H5—C8—C7    | 109.08 (6)  |
| N4—Fe1—N3   | 85.48 (4)   | H6—C8—N4    | 109.08 (6)  |
| N4—Fe1—N5   | 84.14 (4)   | H6—C8—C7    | 109.08 (7)  |
| N4—Fe1—N1   | 93.23 (4)   | H6—C8—H5    | 107.8       |
| N6—Fe1—N3   | 88.79 (4)   | H13—C9—N5   | 118.80 (7)  |
| N6—Fe1—N5   | 166.83 (4)  | C10—C9—N5   | 122.39 (12) |
| N6—Fe1—N1   | 89.70 (4)   | C10—C9—H13  | 118.80 (8)  |
| N6—Fe1—N4   | 82.71 (4)   | C19—C20—N4  | 107.91 (10) |
| N2—Fe1—N3   | 93.84 (4)   | H11—C20—N4  | 110.12 (6)  |
| N2—Fe1—N5   | 95.03 (4)   | H11—C20—C19 | 110.12 (7)  |
| N2—Fe1—N1   | 87.47 (4)   | H12—C20—N4  | 110.12 (6)  |
| N2—Fe1—N4   | 178.92 (4)  | H12—C20—C19 | 110.12 (7)  |
| N2—Fe1—N6   | 98.12 (4)   | H12—C20—H11 | 108.4       |
| C7—N3—Fe1   | 114.91 (8)  | H14—C10—C9  | 120.58 (8)  |
| C3—N3—Fe1   | 126.42 (9)  | C11—C10—C9  | 118.84 (13) |
| C3—N3—C7    | 118.62 (11) | C11—C10—H14 | 120.58 (8)  |
| C13—N5—Fe1  | 112.69 (8)  | H4—C6—C7    | 120.46 (8)  |
| C9—N5—Fe1   | 128.59 (9)  | C5—C6—C7    | 119.09 (12) |
| C9—N5—C13   | 118.69 (11) | C5—C6—H4    | 120.46 (8)  |
| C1—N1—Fe1   | 166.63 (10) | C10—C11—C12 | 118.97 (12) |
| C14—N4—Fe1  | 106.36 (7)  | H15—C11—C12 | 120.51 (8)  |
| C8—N4—Fe1   | 110.36 (7)  | H15—C11—C10 | 120.51 (8)  |
| C8—N4—C14   | 111.64 (9)  | H7—C15—N6   | 118.90 (7)  |
| C20—N4—Fe1  | 105.89 (7)  | C16—C15—N6  | 122.20 (12) |
| C20—N4—C14  | 111.89 (9)  | C16—C15—H7  | 118.90 (8)  |
| C20—N4—C8   | 110.47 (9)  | H8—C16—C15  | 120.47 (8)  |
| C19—N6—Fe1  | 113.25 (8)  | C17—C16—C15 | 119.06 (12) |
| C15—N6—Fe1  | 127.85 (9)  | C17—C16—H8  | 120.47 (8)  |
| C15—N6—C19  | 118.85 (11) | H2—C4—C3    | 120.49 (8)  |
| C2—N2—Fe1   | 168.90 (10) | C5—C4—C3    | 119.02 (12) |
| C8—C7—N3    | 116.56 (10) | C5—C4—H2    | 120.49 (8)  |
| C6—C7—N3    | 122.08 (12) | C21—C22—H19 | 120.68 (9)  |
| C6—C7—C8    | 121.36 (11) | C23—C22—H19 | 120.68 (9)  |
| C18—C19—N6  | 122.07 (11) | C23—C22—C21 | 118.64 (14) |
| C20—C19—N6  | 114.58 (10) | C16—C17—C18 | 118.93 (13) |
| C20—C19—C18 | 123.33 (11) | H9—C17—C18  | 120.54 (8)  |
| C14—C13—N5  | 115.13 (10) | H9—C17—C16  | 120.54 (8)  |
| C12—C13—N5  | 122.22 (12) | C22—C21—Br3 | 119.94 (11) |
| C12—C13—C14 | 122.50 (11) | C26—C21—Br3 | 118.09 (11) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C13—C14—N4      | 109.63 (10)  | C26—C21—C22     | 121.96 (14)  |
| H18—C14—N4      | 109.74 (6)   | C4—C5—C6        | 118.89 (12)  |
| H18—C14—C13     | 109.74 (7)   | H3—C5—C6        | 120.56 (8)   |
| H17—C14—N4      | 109.74 (6)   | H3—C5—C4        | 120.56 (8)   |
| H17—C14—C13     | 109.74 (7)   | H20—C23—C22     | 119.82 (9)   |
| H17—C14—H18     | 108.2        | C24—C23—C22     | 120.36 (15)  |
| N2—C2—S2        | 179.28 (11)  | C24—C23—H20     | 119.82 (10)  |
| H10—C18—C19     | 120.59 (7)   | H23—C26—C21     | 120.54 (9)   |
| C17—C18—C19     | 118.83 (12)  | C25—C26—C21     | 118.91 (15)  |
| C17—C18—H10     | 120.59 (8)   | C25—C26—H23     | 120.54 (10)  |
| H16—C12—C13     | 120.57 (8)   | H21—C24—C23     | 120.12 (10)  |
| C11—C12—C13     | 118.87 (12)  | C25—C24—C23     | 119.75 (15)  |
| C11—C12—H16     | 120.57 (8)   | C25—C24—H21     | 120.12 (10)  |
| H1—C3—N3        | 118.85 (7)   | C24—C25—C26     | 120.36 (15)  |
| C4—C3—N3        | 122.30 (12)  | H22—C25—C26     | 119.82 (10)  |
| C4—C3—H1        | 118.85 (8)   | H22—C25—C24     | 119.82 (10)  |
| N1—C1—S1        | 179.24 (12)  |                 |              |
| Br3—C21—C22—C23 | 178.74 (11)  | C7—C8—N4—C20    | 118.02 (10)  |
| Br3—C21—C26—C25 | −179.29 (12) | C7—C6—C5—C4     | 0.26 (15)    |
| Fe1—N3—C7—C8    | −3.51 (9)    | C19—N6—C15—C16  | 2.01 (13)    |
| Fe1—N3—C7—C6    | 177.20 (8)   | C19—C18—C17—C16 | 2.59 (14)    |
| Fe1—N3—C3—C4    | −177.09 (11) | C19—C20—N4—C14  | 156.84 (10)  |
| Fe1—N5—C13—C14  | −6.19 (9)    | C19—C20—N4—C8   | −78.12 (10)  |
| Fe1—N5—C13—C12  | 178.09 (8)   | C13—N5—C9—C10   | −0.67 (13)   |
| Fe1—N5—C9—C10   | −178.60 (10) | C13—C14—N4—C8   | 83.57 (11)   |
| Fe1—N4—C14—C13  | −36.86 (7)   | C13—C14—N4—C20  | −152.04 (10) |
| Fe1—N4—C8—C7    | 1.26 (8)     | C13—C12—C11—C10 | −0.69 (14)   |
| Fe1—N4—C20—C19  | 41.38 (7)    | C14—C13—N5—C9   | 175.56 (10)  |
| Fe1—N6—C19—C18  | −178.59 (8)  | C14—C13—C12—C11 | −174.57 (12) |
| Fe1—N6—C19—C20  | 2.74 (9)     | C18—C19—N6—C15  | −0.70 (14)   |
| Fe1—N6—C15—C16  | 179.55 (10)  | C18—C17—C16—C15 | −1.37 (15)   |
| N3—C7—C8—N4     | 1.42 (12)    | C12—C13—N5—C9   | −0.16 (14)   |
| N3—C7—C6—C5     | 0.24 (14)    | C12—C11—C10—C9  | −0.08 (15)   |
| N3—C3—C4—C5     | 0.10 (15)    | C3—N3—C7—C8     | 178.72 (11)  |
| N5—C13—C14—N4   | 29.37 (11)   | C3—N3—C7—C6     | −0.56 (13)   |
| N5—C13—C12—C11  | 0.84 (14)    | C3—C4—C5—C6     | −0.42 (16)   |
| N5—C9—C10—C11   | 0.79 (14)    | C8—C7—C6—C5     | −179.01 (12) |
| N4—C14—C13—C12  | −154.92 (10) | C20—C19—N6—C15  | −179.37 (10) |
| N4—C8—C7—C6     | −179.29 (10) | C20—C19—C18—C17 | 176.95 (12)  |
| N4—C20—C19—N6   | −29.88 (11)  | C22—C21—C26—C25 | 0.26 (17)    |
| N4—C20—C19—C18  | 151.47 (10)  | C22—C23—C24—C25 | 0.06 (17)    |
| N6—C19—C18—C17  | −1.60 (14)   | C21—C22—C23—C24 | 0.63 (16)    |
| N6—C15—C16—C17  | −0.97 (15)   | C21—C26—C25—C24 | 0.45 (17)    |
| C7—N3—C3—C4     | 0.39 (13)    | C23—C22—C21—C26 | −0.79 (16)   |
| C7—C8—N4—C14    | −116.80 (10) | C23—C24—C25—C26 | −0.60 (17)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate  
(br\_150K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.3721$  (4) Å

$b = 12.7874$  (3) Å

$c = 15.2882$  (4) Å

$\beta = 109.932$  (3)°

$V = 2641.39$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.558$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 34339 reflections

$\theta = 2.0\text{--}31.0^\circ$

$\mu = 2.27$  mm<sup>-1</sup>

$T = 150$  K

Block, red

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.368$ ,  $T_{\max} = 1.000$

64978 measured reflections

6551 independent reflections

5561 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -19 \rightarrow 19$

$k = -17 \rightarrow 17$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.069$

$S = 1.05$

6551 reflections

335 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 0.7668P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.00$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.47$  e Å<sup>-3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Br3 | 0.63699 (4)   | 0.03043 (4)   | 0.55655 (3)   | 0.05151 (7)                      |
| Fe1 | 0.660881 (15) | 0.337683 (16) | 0.319564 (13) | 0.01980 (6)                      |
| S2  | 0.47669 (3)   | 0.04455 (3)   | 0.16812 (3)   | 0.03155 (9)                      |
| S1  | 0.74487 (4)   | 0.30060 (4)   | 0.04686 (3)   | 0.03831 (11)                     |
| N3  | 0.61015 (9)   | 0.34511 (10)  | 0.42368 (8)   | 0.0219 (2)                       |
| N5  | 0.76988 (9)   | 0.24381 (9)   | 0.39203 (8)   | 0.0221 (2)                       |
| N4  | 0.75309 (9)   | 0.45127 (9)   | 0.38561 (8)   | 0.0208 (2)                       |
| N1  | 0.71159 (9)   | 0.33541 (10)  | 0.21490 (9)   | 0.0250 (3)                       |
| N6  | 0.57481 (9)   | 0.45678 (10)  | 0.26148 (8)   | 0.0229 (3)                       |
| N2  | 0.57190 (9)   | 0.22568 (10)  | 0.25758 (9)   | 0.0267 (3)                       |
| C7  | 0.65098 (10)  | 0.42023 (12)  | 0.48669 (9)   | 0.0224 (3)                       |
| C19 | 0.62402 (11)  | 0.54883 (12)  | 0.27088 (10)  | 0.0229 (3)                       |
| C13 | 0.85594 (11)  | 0.29514 (12)  | 0.43293 (10)  | 0.0228 (3)                       |
| C14 | 0.85482 (10)  | 0.40888 (12)  | 0.40780 (11)  | 0.0248 (3)                       |

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|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| H18 | 0.87924 (10) | 0.41802 (12)  | 0.34665 (11) | 0.0298 (4)* |
| H17 | 0.90690 (10) | 0.45301 (12)  | 0.46692 (11) | 0.0298 (4)* |
| C8  | 0.73288 (11) | 0.48289 (12)  | 0.47193 (10) | 0.0260 (3)  |
| H5  | 0.80145 (11) | 0.47274 (12)  | 0.53309 (10) | 0.0312 (4)* |
| H6  | 0.71268 (11) | 0.56709 (12)  | 0.46705 (10) | 0.0312 (4)* |
| C2  | 0.53203 (10) | 0.15081 (12)  | 0.21990 (10) | 0.0227 (3)  |
| C18 | 0.57632 (12) | 0.64207 (13)  | 0.23672 (10) | 0.0279 (3)  |
| H10 | 0.61817 (12) | 0.71603 (13)  | 0.24686 (10) | 0.0335 (4)* |
| C12 | 0.94092 (12) | 0.24492 (12)  | 0.48933 (10) | 0.0276 (3)  |
| H16 | 1.00971 (12) | 0.28929 (12)  | 0.52286 (10) | 0.0331 (4)* |
| C1  | 0.72545 (11) | 0.32169 (11)  | 0.14498 (10) | 0.0246 (3)  |
| C3  | 0.53775 (11) | 0.28546 (13)  | 0.43519 (11) | 0.0283 (3)  |
| H1  | 0.50443 (11) | 0.22335 (13)  | 0.38402 (11) | 0.0339 (4)* |
| C20 | 0.73393 (11) | 0.54038 (11)  | 0.31895 (10) | 0.0238 (3)  |
| H11 | 0.76336 (11) | 0.61408 (11)  | 0.35675 (10) | 0.0285 (3)* |
| H12 | 0.77112 (11) | 0.52650 (11)  | 0.26710 (10) | 0.0285 (3)* |
| C9  | 0.76666 (12) | 0.14019 (12)  | 0.40605 (10) | 0.0262 (3)  |
| H13 | 0.69629 (12) | 0.09819 (12)  | 0.37332 (10) | 0.0315 (4)* |
| C6  | 0.62037 (12) | 0.43869 (13)  | 0.56221 (10) | 0.0296 (3)  |
| H4  | 0.65515 (12) | 0.50110 (13)  | 0.61254 (10) | 0.0356 (4)* |
| C10 | 0.84912 (13) | 0.08427 (13)  | 0.46044 (11) | 0.0311 (3)  |
| H14 | 0.84442 (13) | −0.00084 (13) | 0.46990 (11) | 0.0374 (4)* |
| C15 | 0.47635 (11) | 0.45650 (13)  | 0.21847 (10) | 0.0282 (3)  |
| H7  | 0.43578 (11) | 0.38215 (13)  | 0.21272 (10) | 0.0338 (4)* |
| C16 | 0.42377 (12) | 0.54630 (14)  | 0.18101 (11) | 0.0326 (4)  |
| H8  | 0.34297 (12) | 0.54320 (14)  | 0.14513 (11) | 0.0391 (4)* |
| C11 | 0.93758 (13) | 0.13734 (13)  | 0.50271 (11) | 0.0312 (3)  |
| H15 | 1.00411 (13) | 0.09494 (13)  | 0.54617 (11) | 0.0375 (4)* |
| C17 | 0.47490 (12) | 0.64074 (14)  | 0.18949 (11) | 0.0324 (3)  |
| H9  | 0.43534 (12) | 0.71318 (14)  | 0.15915 (11) | 0.0388 (4)* |
| C22 | 0.75254 (12) | 0.20572 (14)  | 0.64487 (13) | 0.0350 (4)  |
| H19 | 0.76643 (12) | 0.21641 (14)  | 0.57879 (13) | 0.0420 (4)* |
| C4  | 0.50377 (12) | 0.29945 (14)  | 0.50900 (11) | 0.0334 (4)  |
| H2  | 0.44448 (12) | 0.24927 (14)  | 0.51640 (11) | 0.0401 (4)* |
| C5  | 0.54552 (13) | 0.37765 (15)  | 0.57355 (11) | 0.0349 (4)  |
| H3  | 0.51960 (13) | 0.39096 (15)  | 0.63275 (11) | 0.0419 (4)* |
| C21 | 0.69494 (12) | 0.12407 (13)  | 0.65620 (12) | 0.0325 (3)  |
| C23 | 0.79294 (13) | 0.27468 (15)  | 0.71934 (15) | 0.0436 (4)  |
| H20 | 0.83885 (13) | 0.34095 (15)  | 0.71201 (15) | 0.0523 (5)* |
| C26 | 0.67704 (15) | 0.10818 (16)  | 0.73838 (15) | 0.0457 (5)  |
| H23 | 0.63097 (15) | 0.04200 (16)  | 0.74559 (15) | 0.0548 (6)* |
| C24 | 0.77546 (15) | 0.26001 (17)  | 0.80238 (14) | 0.0502 (5)  |
| H21 | 0.80729 (15) | 0.31448 (17)  | 0.86094 (14) | 0.0602 (6)* |
| C25 | 0.71775 (18) | 0.17648 (17)  | 0.81133 (15) | 0.0532 (5)  |
| H22 | 0.70427 (18) | 0.16454 (17)  | 0.87747 (15) | 0.0638 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Br3 | 0.05464 (14) | 0.03893 (12) | 0.04945 (13) | −0.00369 (9)  | 0.00281 (10) | −0.00251 (9)  |
| Fe1 | 0.02091 (10) | 0.02203 (11) | 0.01540 (10) | −0.00303 (8)  | 0.00480 (8)  | −0.00121 (7)  |
| S2  | 0.02838 (19) | 0.0291 (2)   | 0.0366 (2)   | −0.00644 (15) | 0.01038 (17) | −0.01114 (16) |
| S1  | 0.0597 (3)   | 0.0357 (2)   | 0.0259 (2)   | 0.0119 (2)    | 0.0229 (2)   | 0.00512 (17)  |
| N3  | 0.0228 (6)   | 0.0265 (6)   | 0.0161 (5)   | −0.0027 (5)   | 0.0060 (5)   | −0.0013 (5)   |
| N5  | 0.0251 (6)   | 0.0218 (6)   | 0.0186 (6)   | −0.0014 (5)   | 0.0064 (5)   | 0.0004 (5)    |
| N4  | 0.0197 (6)   | 0.0212 (6)   | 0.0190 (6)   | −0.0023 (5)   | 0.0032 (5)   | 0.0002 (4)    |
| N1  | 0.0293 (6)   | 0.0277 (6)   | 0.0186 (6)   | 0.0002 (5)    | 0.0091 (5)   | −0.0002 (5)   |
| N6  | 0.0220 (6)   | 0.0272 (6)   | 0.0165 (5)   | −0.0007 (5)   | 0.0028 (5)   | −0.0015 (5)   |
| N2  | 0.0282 (6)   | 0.0270 (7)   | 0.0226 (6)   | −0.0075 (5)   | 0.0059 (5)   | −0.0051 (5)   |
| C7  | 0.0232 (7)   | 0.0258 (7)   | 0.0160 (6)   | 0.0016 (6)    | 0.0041 (5)   | −0.0008 (5)   |
| C19 | 0.0241 (7)   | 0.0238 (7)   | 0.0186 (6)   | 0.0004 (6)    | 0.0044 (6)   | 0.0010 (5)    |
| C13 | 0.0229 (7)   | 0.0233 (7)   | 0.0209 (7)   | 0.0005 (6)    | 0.0058 (6)   | 0.0011 (5)    |
| C14 | 0.0213 (7)   | 0.0235 (7)   | 0.0269 (7)   | −0.0025 (6)   | 0.0046 (6)   | 0.0023 (6)    |
| C8  | 0.0280 (7)   | 0.0268 (8)   | 0.0208 (7)   | −0.0052 (6)   | 0.0053 (6)   | −0.0044 (6)   |
| C2  | 0.0217 (7)   | 0.0251 (7)   | 0.0203 (7)   | −0.0027 (6)   | 0.0057 (5)   | −0.0039 (6)   |
| C18 | 0.0295 (8)   | 0.0280 (8)   | 0.0244 (7)   | 0.0035 (6)    | 0.0067 (6)   | 0.0041 (6)    |
| C12 | 0.0263 (7)   | 0.0285 (8)   | 0.0247 (7)   | 0.0037 (6)    | 0.0043 (6)   | 0.0004 (6)    |
| C1  | 0.0303 (7)   | 0.0240 (7)   | 0.0204 (7)   | 0.0028 (6)    | 0.0098 (6)   | 0.0024 (6)    |
| C3  | 0.0262 (7)   | 0.0343 (8)   | 0.0246 (7)   | −0.0064 (6)   | 0.0090 (6)   | −0.0020 (6)   |
| C20 | 0.0231 (7)   | 0.0223 (7)   | 0.0237 (7)   | −0.0024 (6)   | 0.0051 (6)   | 0.0027 (5)    |
| C9  | 0.0341 (8)   | 0.0214 (7)   | 0.0235 (7)   | −0.0031 (6)   | 0.0100 (6)   | 0.0001 (6)    |
| C6  | 0.0323 (8)   | 0.0356 (9)   | 0.0211 (7)   | 0.0017 (7)    | 0.0091 (6)   | −0.0039 (6)   |
| C10 | 0.0412 (9)   | 0.0243 (8)   | 0.0276 (8)   | 0.0039 (7)    | 0.0114 (7)   | 0.0027 (6)    |
| C15 | 0.0221 (7)   | 0.0348 (9)   | 0.0231 (7)   | −0.0005 (6)   | 0.0018 (6)   | −0.0032 (6)   |
| C16 | 0.0243 (8)   | 0.0420 (10)  | 0.0255 (8)   | 0.0042 (7)    | 0.0007 (6)   | −0.0012 (7)   |
| C11 | 0.0361 (9)   | 0.0289 (8)   | 0.0268 (8)   | 0.0079 (7)    | 0.0083 (7)   | 0.0043 (6)    |
| C17 | 0.0304 (8)   | 0.0358 (9)   | 0.0274 (8)   | 0.0090 (7)    | 0.0053 (7)   | 0.0047 (7)    |
| C22 | 0.0319 (8)   | 0.0373 (9)   | 0.0387 (9)   | 0.0017 (7)    | 0.0156 (7)   | 0.0086 (8)    |
| C4  | 0.0299 (8)   | 0.0450 (9)   | 0.0284 (8)   | −0.0048 (7)   | 0.0138 (7)   | 0.0013 (7)    |
| C5  | 0.0350 (9)   | 0.0497 (10)  | 0.0240 (8)   | 0.0018 (8)    | 0.0151 (7)   | −0.0013 (7)   |
| C21 | 0.0293 (8)   | 0.0307 (8)   | 0.0375 (9)   | 0.0017 (7)    | 0.0113 (7)   | 0.0046 (7)    |
| C23 | 0.0276 (8)   | 0.0370 (10)  | 0.0613 (13)  | −0.0008 (7)   | 0.0088 (8)   | 0.0002 (9)    |
| C26 | 0.0547 (11)  | 0.0397 (10)  | 0.0529 (12)  | 0.0051 (9)    | 0.0317 (10)  | 0.0125 (9)    |
| C24 | 0.0477 (11)  | 0.0487 (12)  | 0.0418 (11)  | 0.0169 (9)    | −0.0007 (9)  | −0.0069 (9)   |
| C25 | 0.0750 (15)  | 0.0516 (13)  | 0.0393 (11)  | 0.0203 (11)   | 0.0278 (11)  | 0.0095 (9)    |

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

|         |             |         |           |
|---------|-------------|---------|-----------|
| Br3—C21 | 1.8929 (18) | C12—H16 | 1.1030    |
| Fe1—N3  | 1.9661 (12) | C12—C11 | 1.394 (2) |
| Fe1—N5  | 1.9847 (12) | C3—H1   | 1.1030    |
| Fe1—N4  | 1.9933 (12) | C3—C4   | 1.385 (2) |
| Fe1—N1  | 1.9716 (12) | C20—H11 | 1.1110    |
| Fe1—N6  | 1.9721 (13) | C20—H12 | 1.1110    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| Fe1—N2     | 1.9386 (13) | C9—H13      | 1.1030      |
| S2—C2      | 1.6359 (15) | C9—C10      | 1.391 (2)   |
| S1—C1      | 1.6390 (15) | C6—H4       | 1.1030      |
| N3—C7      | 1.3452 (18) | C6—C5       | 1.387 (2)   |
| N3—C3      | 1.3491 (19) | C10—H14     | 1.1030      |
| N5—C13     | 1.3505 (18) | C10—C11     | 1.391 (2)   |
| N5—C9      | 1.3456 (19) | C15—H7      | 1.1030      |
| N4—C14     | 1.4861 (18) | C15—C16     | 1.387 (2)   |
| N4—C8      | 1.5002 (18) | C16—H8      | 1.1030      |
| N4—C20     | 1.4905 (18) | C16—C17     | 1.397 (2)   |
| N1—C1      | 1.1655 (19) | C11—H15     | 1.1030      |
| N6—C19     | 1.3554 (19) | C17—H9      | 1.1030      |
| N6—C15     | 1.3417 (19) | C22—H19     | 1.1030      |
| N2—C2      | 1.1626 (19) | C22—C21     | 1.380 (2)   |
| C7—C8      | 1.503 (2)   | C22—C23     | 1.400 (3)   |
| C7—C6      | 1.389 (2)   | C4—H2       | 1.1030      |
| C19—C18    | 1.386 (2)   | C4—C5       | 1.389 (2)   |
| C19—C20    | 1.502 (2)   | C5—H3       | 1.1030      |
| C13—C14    | 1.503 (2)   | C21—C26     | 1.381 (3)   |
| C13—C12    | 1.389 (2)   | C23—H20     | 1.1030      |
| C14—H18    | 1.1110      | C23—C24     | 1.388 (3)   |
| C14—H17    | 1.1110      | C26—H23     | 1.1030      |
| C8—H5      | 1.1110      | C26—C25     | 1.380 (3)   |
| C8—H6      | 1.1110      | C24—H21     | 1.1030      |
| C18—H10    | 1.1030      | C24—C25     | 1.387 (3)   |
| C18—C17    | 1.389 (2)   | C25—H22     | 1.1030      |
|            |             |             |             |
| N5—Fe1—N3  | 90.99 (5)   | C11—C12—C13 | 118.70 (15) |
| N4—Fe1—N3  | 85.45 (5)   | C11—C12—H16 | 120.65 (10) |
| N4—Fe1—N5  | 84.03 (5)   | N1—C1—S1    | 179.18 (14) |
| N1—Fe1—N3  | 178.08 (5)  | H1—C3—N3    | 118.83 (8)  |
| N1—Fe1—N5  | 90.24 (5)   | C4—C3—N3    | 122.33 (15) |
| N1—Fe1—N4  | 93.21 (5)   | C4—C3—H1    | 118.83 (10) |
| N6—Fe1—N3  | 88.77 (5)   | C19—C20—N4  | 107.93 (11) |
| N6—Fe1—N5  | 166.65 (5)  | H11—C20—N4  | 110.12 (7)  |
| N6—Fe1—N4  | 82.65 (5)   | H11—C20—C19 | 110.12 (8)  |
| N6—Fe1—N1  | 89.69 (5)   | H12—C20—N4  | 110.12 (7)  |
| N2—Fe1—N3  | 93.81 (5)   | H12—C20—C19 | 110.12 (8)  |
| N2—Fe1—N5  | 95.15 (5)   | H12—C20—H11 | 108.4       |
| N2—Fe1—N4  | 178.88 (5)  | H13—C9—N5   | 118.90 (9)  |
| N2—Fe1—N1  | 87.54 (5)   | C10—C9—N5   | 122.19 (15) |
| N2—Fe1—N6  | 98.19 (5)   | C10—C9—H13  | 118.90 (10) |
| C7—N3—Fe1  | 114.99 (10) | H4—C6—C7    | 120.42 (9)  |
| C3—N3—Fe1  | 126.41 (10) | C5—C6—C7    | 119.17 (15) |
| C3—N3—C7   | 118.55 (13) | C5—C6—H4    | 120.42 (9)  |
| C13—N5—Fe1 | 112.86 (10) | H14—C10—C9  | 120.51 (10) |
| C9—N5—Fe1  | 128.39 (11) | C11—C10—C9  | 118.97 (15) |
| C9—N5—C13  | 118.72 (13) | C11—C10—H14 | 120.51 (10) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C14—N4—Fe1      | 106.40 (9)   | H7—C15—N6       | 118.81 (9)   |
| C8—N4—Fe1       | 110.32 (9)   | C16—C15—N6      | 122.37 (15)  |
| C8—N4—C14       | 111.59 (11)  | C16—C15—H7      | 118.81 (10)  |
| C20—N4—Fe1      | 105.95 (8)   | H8—C16—C15      | 120.58 (10)  |
| C20—N4—C14      | 111.90 (11)  | C17—C16—C15     | 118.83 (15)  |
| C20—N4—C8       | 110.45 (11)  | C17—C16—H8      | 120.58 (9)   |
| C1—N1—Fe1       | 166.56 (13)  | C10—C11—C12     | 119.02 (15)  |
| C19—N6—Fe1      | 113.47 (10)  | H15—C11—C12     | 120.49 (10)  |
| C15—N6—Fe1      | 127.84 (11)  | H15—C11—C10     | 120.49 (10)  |
| C15—N6—C19      | 118.64 (13)  | C16—C17—C18     | 118.99 (15)  |
| C2—N2—Fe1       | 169.14 (12)  | H9—C17—C18      | 120.50 (10)  |
| C8—C7—N3        | 116.49 (12)  | H9—C17—C16      | 120.50 (9)   |
| C6—C7—N3        | 122.15 (14)  | C21—C22—H19     | 120.83 (11)  |
| C6—C7—C8        | 121.36 (13)  | C23—C22—H19     | 120.83 (11)  |
| C18—C19—N6      | 122.28 (14)  | C23—C22—C21     | 118.35 (17)  |
| C20—C19—N6      | 114.41 (12)  | H2—C4—C3        | 120.50 (10)  |
| C20—C19—C18     | 123.29 (13)  | C5—C4—C3        | 119.00 (15)  |
| C14—C13—N5      | 115.06 (13)  | C5—C4—H2        | 120.50 (9)   |
| C12—C13—N5      | 122.37 (14)  | C4—C5—C6        | 118.80 (14)  |
| C12—C13—C14     | 122.45 (13)  | H3—C5—C6        | 120.60 (9)   |
| C13—C14—N4      | 109.59 (11)  | H3—C5—C4        | 120.60 (9)   |
| H18—C14—N4      | 109.75 (7)   | C22—C21—Br3     | 119.73 (14)  |
| H18—C14—C13     | 109.75 (8)   | C26—C21—Br3     | 118.16 (14)  |
| H17—C14—N4      | 109.75 (8)   | C26—C21—C22     | 122.11 (18)  |
| H17—C14—C13     | 109.75 (8)   | H20—C23—C22     | 119.86 (11)  |
| H17—C14—H18     | 108.2        | C24—C23—C22     | 120.29 (18)  |
| C7—C8—N4        | 112.63 (12)  | C24—C23—H20     | 119.86 (12)  |
| H5—C8—N4        | 109.07 (7)   | H23—C26—C21     | 120.55 (12)  |
| H5—C8—C7        | 109.07 (8)   | C25—C26—C21     | 118.89 (19)  |
| H6—C8—N4        | 109.07 (8)   | C25—C26—H23     | 120.55 (12)  |
| H6—C8—C7        | 109.07 (8)   | H21—C24—C23     | 120.17 (12)  |
| H6—C8—H5        | 107.8        | C25—C24—C23     | 119.66 (19)  |
| N2—C2—S2        | 179.24 (14)  | C25—C24—H21     | 120.17 (13)  |
| H10—C18—C19     | 120.59 (9)   | C24—C25—C26     | 120.70 (19)  |
| C17—C18—C19     | 118.83 (15)  | H22—C25—C26     | 119.65 (12)  |
| C17—C18—H10     | 120.59 (10)  | H22—C25—C24     | 119.65 (13)  |
| H16—C12—C13     | 120.65 (9)   |                 |              |
| Br3—C21—C22—C23 | 178.90 (13)  | C7—C8—N4—C20    | 117.87 (12)  |
| Br3—C21—C26—C25 | −179.23 (15) | C7—C6—C5—C4     | 0.29 (18)    |
| Fe1—N3—C7—C8    | −3.51 (11)   | C19—N6—C15—C16  | 1.95 (15)    |
| Fe1—N3—C7—C6    | 177.21 (10)  | C19—C18—C17—C16 | 2.37 (17)    |
| Fe1—N3—C3—C4    | −177.08 (13) | C19—C20—N4—C14  | 156.89 (11)  |
| Fe1—N5—C13—C14  | −6.00 (10)   | C19—C20—N4—C8   | −78.14 (12)  |
| Fe1—N5—C13—C12  | 177.94 (9)   | C13—N5—C9—C10   | −0.62 (15)   |
| Fe1—N5—C9—C10   | −178.67 (12) | C13—C14—N4—C8   | 83.51 (13)   |
| Fe1—N4—C14—C13  | −36.88 (9)   | C13—C14—N4—C20  | −152.16 (12) |
| Fe1—N4—C8—C7    | 1.07 (9)     | C13—C12—C11—C10 | −0.98 (17)   |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| Fe1—N4—C20—C19 | 41.34 (9)    | C14—C13—N5—C9   | 175.66 (12)  |
| Fe1—N6—C19—C18 | −178.49 (10) | C14—C13—C12—C11 | −174.57 (15) |
| Fe1—N6—C19—C20 | 2.96 (10)    | C8—C7—N3—C3     | 178.66 (13)  |
| Fe1—N6—C15—C16 | 179.31 (13)  | C8—C7—C6—C5     | −179.01 (15) |
| N3—C7—C8—N4    | 1.55 (14)    | C18—C19—N6—C15  | −0.76 (17)   |
| N3—C7—C6—C5    | 0.23 (17)    | C18—C17—C16—C15 | −1.26 (18)   |
| N3—C3—C4—C5    | 0.05 (19)    | C12—C13—N5—C9   | −0.40 (17)   |
| N5—C13—C14—N4  | 29.22 (13)   | C12—C11—C10—C9  | 0.03 (18)    |
| N5—C13—C12—C11 | 1.20 (16)    | C3—N3—C7—C6     | −0.61 (16)   |
| N5—C9—C10—C11  | 0.80 (17)    | C3—C4—C5—C6     | −0.43 (19)   |
| N4—C14—C13—C12 | −154.72 (11) | C20—C19—N6—C15  | −179.32 (12) |
| N4—C8—C7—C6    | −179.17 (12) | C20—C19—C18—C17 | 177.03 (15)  |
| N4—C20—C19—N6  | −29.97 (13)  | C22—C21—C26—C25 | 0.3 (2)      |
| N4—C20—C19—C18 | 151.49 (11)  | C22—C23—C24—C25 | 0.1 (2)      |
| N6—C19—C18—C17 | −1.40 (17)   | C21—C22—C23—C24 | 0.42 (19)    |
| N6—C15—C16—C17 | −0.94 (18)   | C21—C26—C25—C24 | 0.2 (2)      |
| C7—N3—C3—C4    | 0.47 (16)    | C23—C22—C21—C26 | −0.6 (2)     |
| C7—C8—N4—C14   | −116.99 (13) | C23—C24—C25—C26 | −0.4 (2)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N, $N'$ , $N''$ , $N'''$ ]iron(II) bromobenzene monosolvate (br\_160K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.3790$  (4) Å

$b = 12.7888$  (3) Å

$c = 15.2972$  (4) Å

$\beta = 109.891$  (3)°

$V = 2645.19$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.555$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 33365 reflections

$\theta = 2.0$ – $31.3^\circ$

$\mu = 2.27$  mm<sup>−1</sup>

$T = 160$  K

, red

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.407$ ,  $T_{\max} = 1.000$

65622 measured reflections

6560 independent reflections

5516 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -19 \rightarrow 19$

$k = -17 \rightarrow 17$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.070$

$S = 1.05$

6560 reflections

335 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 0.9106P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$$

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.339098 (15) | 0.337553 (16) | 0.680516 (14) | 0.02082 (6)                      |
| Br3 | 0.36308 (4)   | 0.03036 (4)   | 0.44312 (4)   | 0.05500 (8)                      |
| S2  | 0.52335 (3)   | 0.04458 (3)   | 0.83197 (3)   | 0.03330 (10)                     |
| S1  | 0.25521 (4)   | 0.30064 (4)   | 0.95308 (3)   | 0.04047 (12)                     |
| N5  | 0.23019 (9)   | 0.24372 (10)  | 0.60815 (8)   | 0.0230 (3)                       |
| N3  | 0.38985 (9)   | 0.34494 (10)  | 0.57642 (8)   | 0.0230 (3)                       |
| N4  | 0.24690 (9)   | 0.45119 (9)   | 0.61449 (8)   | 0.0216 (2)                       |
| N1  | 0.28838 (10)  | 0.33531 (10)  | 0.78517 (9)   | 0.0263 (3)                       |
| N6  | 0.42511 (9)   | 0.45674 (10)  | 0.73851 (8)   | 0.0242 (3)                       |
| N2  | 0.42801 (10)  | 0.22558 (11)  | 0.74252 (9)   | 0.0281 (3)                       |
| C7  | 0.34890 (11)  | 0.41983 (12)  | 0.51342 (10)  | 0.0233 (3)                       |
| C19 | 0.37604 (11)  | 0.54874 (12)  | 0.72911 (10)  | 0.0240 (3)                       |
| C8  | 0.26704 (12)  | 0.48268 (13)  | 0.52825 (10)  | 0.0271 (3)                       |
| H5  | 0.19849 (12)  | 0.47256 (13)  | 0.46719 (10)  | 0.0326 (4)*                      |
| H6  | 0.28729 (12)  | 0.56685 (13)  | 0.53306 (10)  | 0.0326 (4)*                      |
| C2  | 0.46769 (11)  | 0.15073 (12)  | 0.78021 (10)  | 0.0237 (3)                       |
| C13 | 0.14421 (11)  | 0.29523 (12)  | 0.56729 (10)  | 0.0236 (3)                       |
| C14 | 0.14533 (11)  | 0.40884 (12)  | 0.59242 (11)  | 0.0257 (3)                       |
| H18 | 0.12095 (11)  | 0.41796 (12)  | 0.65356 (11)  | 0.0309 (4)*                      |
| H17 | 0.09328 (11)  | 0.45298 (12)  | 0.53339 (11)  | 0.0309 (4)*                      |
| C1  | 0.27449 (12)  | 0.32163 (12)  | 0.85513 (11)  | 0.0259 (3)                       |
| C12 | 0.05925 (12)  | 0.24483 (13)  | 0.51090 (11)  | 0.0289 (3)                       |
| H16 | −0.00949 (12) | 0.28922 (13)  | 0.47747 (11)  | 0.0347 (4)*                      |
| C20 | 0.26612 (11)  | 0.54014 (12)  | 0.68102 (11)  | 0.0252 (3)                       |
| H11 | 0.23670 (11)  | 0.61382 (12)  | 0.64325 (11)  | 0.0302 (4)*                      |
| H12 | 0.22895 (11)  | 0.52627 (12)  | 0.73285 (11)  | 0.0302 (4)*                      |
| C9  | 0.23313 (13)  | 0.14019 (12)  | 0.59386 (10)  | 0.0279 (3)                       |
| H13 | 0.30343 (13)  | 0.09812 (12)  | 0.62640 (10)  | 0.0335 (4)*                      |
| C18 | 0.42370 (12)  | 0.64195 (13)  | 0.76304 (11)  | 0.0299 (3)                       |
| H10 | 0.38188 (12)  | 0.71591 (13)  | 0.75275 (11)  | 0.0359 (4)*                      |
| C3  | 0.46208 (12)  | 0.28540 (13)  | 0.56489 (11)  | 0.0297 (3)                       |
| H1  | 0.49532 (12)  | 0.22330 (13)  | 0.61602 (11)  | 0.0357 (4)*                      |
| C15 | 0.52352 (12)  | 0.45647 (14)  | 0.78148 (11)  | 0.0299 (3)                       |
| H7  | 0.56408 (12)  | 0.38214 (14)  | 0.78724 (11)  | 0.0358 (4)*                      |
| C10 | 0.15088 (13)  | 0.08428 (13)  | 0.53960 (11)  | 0.0324 (4)                       |
| H14 | 0.15560 (13)  | −0.00082 (13) | 0.53018 (11)  | 0.0389 (4)*                      |
| C11 | 0.06244 (13)  | 0.13740 (13)  | 0.49735 (11)  | 0.0325 (4)                       |
| H15 | −0.00405 (13) | 0.09509 (13)  | 0.45384 (11)  | 0.0390 (4)*                      |
| C6  | 0.37943 (12)  | 0.43885 (14)  | 0.43789 (11)  | 0.0311 (3)                       |
| H4  | 0.34483 (12)  | 0.50152 (14)  | 0.38786 (11)  | 0.0373 (4)*                      |
| C16 | 0.57604 (13)  | 0.54632 (15)  | 0.81889 (11)  | 0.0348 (4)                       |



|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| H8  | 0.65674 (13) | 0.54323 (15) | 0.85489 (11) | 0.0418 (4)* |
| C17 | 0.52491 (13) | 0.64075 (14) | 0.81017 (12) | 0.0346 (4)  |
| H9  | 0.56439 (13) | 0.71326 (14) | 0.84031 (12) | 0.0415 (4)* |
| C4  | 0.49619 (13) | 0.29931 (15) | 0.49111 (12) | 0.0346 (4)  |
| H2  | 0.55553 (13) | 0.24924 (15) | 0.48371 (12) | 0.0416 (4)* |
| C22 | 0.24757 (13) | 0.20551 (15) | 0.35530 (13) | 0.0379 (4)  |
| H19 | 0.23374 (13) | 0.21619 (15) | 0.42137 (13) | 0.0454 (5)* |
| C21 | 0.30506 (13) | 0.12389 (14) | 0.34363 (13) | 0.0340 (4)  |
| C5  | 0.45413 (13) | 0.37769 (15) | 0.42651 (12) | 0.0367 (4)  |
| H3  | 0.47985 (13) | 0.39091 (15) | 0.36720 (12) | 0.0441 (5)* |
| C23 | 0.20731 (13) | 0.27439 (16) | 0.28074 (16) | 0.0464 (5)  |
| H20 | 0.16137 (13) | 0.34066 (16) | 0.28798 (16) | 0.0557 (6)* |
| C26 | 0.32286 (16) | 0.10839 (16) | 0.26161 (15) | 0.0472 (5)  |
| H23 | 0.36901 (16) | 0.04232 (16) | 0.25437 (15) | 0.0567 (6)* |
| C24 | 0.22487 (16) | 0.25977 (18) | 0.19793 (15) | 0.0522 (5)  |
| H21 | 0.19318 (16) | 0.31437 (18) | 0.13953 (15) | 0.0627 (7)* |
| C25 | 0.28227 (19) | 0.17645 (18) | 0.18873 (16) | 0.0563 (6)  |
| H22 | 0.29563 (19) | 0.16445 (18) | 0.12258 (16) | 0.0675 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.02195 (10) | 0.02334 (11) | 0.01606 (10) | 0.00320 (8)  | 0.00502 (8)  | 0.00117 (8)   |
| Br3 | 0.05854 (15) | 0.04131 (12) | 0.05273 (14) | 0.00379 (9)  | 0.00278 (11) | 0.00277 (9)   |
| S2  | 0.0298 (2)   | 0.0310 (2)   | 0.0383 (2)   | 0.00658 (16) | 0.01071 (18) | 0.01163 (17)  |
| S1  | 0.0633 (3)   | 0.0376 (2)   | 0.0272 (2)   | −0.0128 (2)  | 0.0241 (2)   | −0.00548 (17) |
| N5  | 0.0264 (6)   | 0.0229 (6)   | 0.0194 (6)   | 0.0011 (5)   | 0.0071 (5)   | 0.0000 (5)    |
| N3  | 0.0234 (6)   | 0.0283 (6)   | 0.0169 (6)   | 0.0024 (5)   | 0.0062 (5)   | 0.0014 (5)    |
| N4  | 0.0215 (6)   | 0.0215 (6)   | 0.0194 (6)   | 0.0028 (5)   | 0.0038 (5)   | −0.0001 (5)   |
| N1  | 0.0307 (7)   | 0.0294 (7)   | 0.0197 (6)   | 0.0004 (5)   | 0.0097 (5)   | −0.0002 (5)   |
| N6  | 0.0228 (6)   | 0.0284 (7)   | 0.0181 (6)   | 0.0011 (5)   | 0.0028 (5)   | 0.0017 (5)    |
| N2  | 0.0301 (7)   | 0.0290 (7)   | 0.0228 (6)   | 0.0078 (6)   | 0.0057 (5)   | 0.0059 (5)    |
| C7  | 0.0244 (7)   | 0.0265 (7)   | 0.0173 (7)   | −0.0007 (6)  | 0.0049 (6)   | 0.0011 (6)    |
| C19 | 0.0251 (7)   | 0.0256 (7)   | 0.0188 (7)   | 0.0001 (6)   | 0.0041 (6)   | −0.0013 (5)   |
| C8  | 0.0292 (8)   | 0.0292 (8)   | 0.0209 (7)   | 0.0060 (6)   | 0.0059 (6)   | 0.0052 (6)    |
| C2  | 0.0231 (7)   | 0.0270 (8)   | 0.0197 (7)   | 0.0031 (6)   | 0.0055 (6)   | 0.0042 (6)    |
| C13 | 0.0235 (7)   | 0.0246 (7)   | 0.0216 (7)   | −0.0003 (6)  | 0.0060 (6)   | −0.0010 (6)   |
| C14 | 0.0225 (7)   | 0.0246 (7)   | 0.0273 (8)   | 0.0032 (6)   | 0.0048 (6)   | −0.0016 (6)   |
| C1  | 0.0331 (8)   | 0.0239 (7)   | 0.0215 (7)   | −0.0031 (6)  | 0.0104 (6)   | −0.0023 (6)   |
| C12 | 0.0273 (8)   | 0.0303 (8)   | 0.0257 (7)   | −0.0047 (6)  | 0.0044 (6)   | −0.0011 (6)   |
| C20 | 0.0247 (7)   | 0.0233 (7)   | 0.0253 (7)   | 0.0022 (6)   | 0.0057 (6)   | −0.0031 (6)   |
| C9  | 0.0366 (9)   | 0.0235 (7)   | 0.0237 (7)   | 0.0026 (6)   | 0.0104 (7)   | −0.0001 (6)   |
| C18 | 0.0317 (8)   | 0.0297 (8)   | 0.0264 (8)   | −0.0040 (7)  | 0.0075 (7)   | −0.0052 (6)   |
| C3  | 0.0278 (8)   | 0.0362 (9)   | 0.0257 (8)   | 0.0069 (7)   | 0.0098 (6)   | 0.0022 (7)    |
| C15 | 0.0230 (7)   | 0.0382 (9)   | 0.0237 (7)   | 0.0012 (7)   | 0.0019 (6)   | 0.0036 (6)    |
| C10 | 0.0436 (10)  | 0.0249 (8)   | 0.0283 (8)   | −0.0041 (7)  | 0.0115 (7)   | −0.0031 (6)   |
| C11 | 0.0370 (9)   | 0.0308 (9)   | 0.0272 (8)   | −0.0083 (7)  | 0.0077 (7)   | −0.0036 (6)   |
| C6  | 0.0344 (8)   | 0.0376 (9)   | 0.0218 (7)   | −0.0013 (7)  | 0.0101 (7)   | 0.0050 (6)    |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C16 | 0.0258 (8)  | 0.0461 (10) | 0.0261 (8)  | −0.0052 (7)  | 0.0004 (6)  | 0.0008 (7)   |
| C17 | 0.0326 (9)  | 0.0378 (9)  | 0.0288 (8)  | −0.0089 (7)  | 0.0045 (7)  | −0.0056 (7)  |
| C4  | 0.0293 (8)  | 0.0484 (10) | 0.0295 (8)  | 0.0042 (7)   | 0.0143 (7)  | −0.0026 (8)  |
| C22 | 0.0346 (9)  | 0.0399 (10) | 0.0426 (10) | −0.0020 (8)  | 0.0177 (8)  | −0.0091 (8)  |
| C21 | 0.0313 (8)  | 0.0317 (8)  | 0.0391 (9)  | −0.0015 (7)  | 0.0123 (7)  | −0.0053 (7)  |
| C5  | 0.0365 (9)  | 0.0519 (11) | 0.0263 (8)  | −0.0017 (8)  | 0.0166 (7)  | 0.0017 (7)   |
| C23 | 0.0277 (9)  | 0.0397 (10) | 0.0668 (14) | 0.0007 (8)   | 0.0096 (9)  | −0.0015 (10) |
| C26 | 0.0569 (12) | 0.0410 (11) | 0.0542 (12) | −0.0050 (9)  | 0.0324 (10) | −0.0136 (9)  |
| C24 | 0.0501 (12) | 0.0522 (13) | 0.0415 (11) | −0.0186 (10) | −0.0011 (9) | 0.0059 (9)   |
| C25 | 0.0796 (16) | 0.0533 (13) | 0.0431 (12) | −0.0216 (12) | 0.0303 (11) | −0.0096 (10) |

*Geometric parameters (Å, °)*

|           |             |            |             |
|-----------|-------------|------------|-------------|
| Fe1—N5    | 1.9844 (13) | C20—H11    | 1.1110      |
| Fe1—N3    | 1.9666 (12) | C20—H12    | 1.1110      |
| Fe1—N4    | 1.9946 (12) | C9—H13     | 1.1030      |
| Fe1—N1    | 1.9722 (13) | C9—C10     | 1.390 (2)   |
| Fe1—N6    | 1.9732 (13) | C18—H10    | 1.1030      |
| Fe1—N2    | 1.9390 (13) | C18—C17    | 1.387 (2)   |
| Br3—C21   | 1.8918 (19) | C3—H1      | 1.1030      |
| S2—C2     | 1.6369 (15) | C3—C4      | 1.387 (2)   |
| S1—C1     | 1.6361 (16) | C15—H7     | 1.1030      |
| N5—C13    | 1.3517 (19) | C15—C16    | 1.387 (2)   |
| N5—C9     | 1.3449 (19) | C10—H14    | 1.1030      |
| N3—C7     | 1.3441 (19) | C10—C11    | 1.392 (2)   |
| N3—C3     | 1.347 (2)   | C11—H15    | 1.1030      |
| N4—C8     | 1.4988 (19) | C6—H4      | 1.1030      |
| N4—C14    | 1.4848 (18) | C6—C5      | 1.387 (2)   |
| N4—C20    | 1.4882 (18) | C16—H8     | 1.1030      |
| N1—C1     | 1.167 (2)   | C16—C17    | 1.396 (3)   |
| N6—C19    | 1.3541 (19) | C17—H9     | 1.1030      |
| N6—C15    | 1.342 (2)   | C4—H2      | 1.1030      |
| N2—C2     | 1.162 (2)   | C4—C5      | 1.393 (3)   |
| C7—C8     | 1.505 (2)   | C22—H19    | 1.1030      |
| C7—C6     | 1.390 (2)   | C22—C21    | 1.380 (2)   |
| C19—C20   | 1.503 (2)   | C22—C23    | 1.401 (3)   |
| C19—C18   | 1.385 (2)   | C21—C26    | 1.377 (3)   |
| C8—H5     | 1.1110      | C5—H3      | 1.1030      |
| C8—H6     | 1.1110      | C23—H20    | 1.1030      |
| C13—C14   | 1.502 (2)   | C23—C24    | 1.386 (3)   |
| C13—C12   | 1.391 (2)   | C26—H23    | 1.1030      |
| C14—H18   | 1.1110      | C26—C25    | 1.378 (3)   |
| C14—H17   | 1.1110      | C24—H21    | 1.1030      |
| C12—H16   | 1.1030      | C24—C25    | 1.384 (3)   |
| C12—C11   | 1.393 (2)   | C25—H22    | 1.1030      |
| N3—Fe1—N5 | 90.99 (5)   | C19—C20—N4 | 108.08 (12) |
| N4—Fe1—N5 | 84.02 (5)   | H11—C20—N4 | 110.09 (8)  |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N4—Fe1—N3   | 85.46 (5)   | H11—C20—C19 | 110.09 (8)  |
| N1—Fe1—N5   | 90.25 (5)   | H12—C20—N4  | 110.09 (8)  |
| N1—Fe1—N3   | 178.08 (5)  | H12—C20—C19 | 110.09 (8)  |
| N1—Fe1—N4   | 93.21 (5)   | H12—C20—H11 | 108.4       |
| N6—Fe1—N5   | 166.62 (5)  | H13—C9—N5   | 118.79 (9)  |
| N6—Fe1—N3   | 88.76 (5)   | C10—C9—N5   | 122.43 (15) |
| N6—Fe1—N4   | 82.63 (5)   | C10—C9—H13  | 118.79 (10) |
| N6—Fe1—N1   | 89.69 (5)   | H10—C18—C19 | 120.56 (9)  |
| N2—Fe1—N5   | 95.17 (6)   | C17—C18—C19 | 118.89 (16) |
| N2—Fe1—N3   | 93.84 (5)   | C17—C18—H10 | 120.56 (10) |
| N2—Fe1—N4   | 178.91 (5)  | H1—C3—N3    | 118.79 (9)  |
| N2—Fe1—N1   | 87.51 (5)   | C4—C3—N3    | 122.42 (15) |
| N2—Fe1—N6   | 98.19 (6)   | C4—C3—H1    | 118.79 (10) |
| C13—N5—Fe1  | 112.78 (10) | H7—C15—N6   | 118.82 (9)  |
| C9—N5—Fe1   | 128.57 (11) | C16—C15—N6  | 122.36 (16) |
| C9—N5—C13   | 118.61 (14) | C16—C15—H7  | 118.82 (10) |
| C7—N3—Fe1   | 114.97 (10) | H14—C10—C9  | 120.53 (10) |
| C3—N3—Fe1   | 126.44 (10) | C11—C10—C9  | 118.93 (16) |
| C3—N3—C7    | 118.56 (13) | C11—C10—H14 | 120.53 (10) |
| C8—N4—Fe1   | 110.32 (9)  | C10—C11—C12 | 118.92 (15) |
| C14—N4—Fe1  | 106.39 (9)  | H15—C11—C12 | 120.54 (10) |
| C14—N4—C8   | 111.56 (12) | H15—C11—C10 | 120.54 (10) |
| C20—N4—Fe1  | 105.89 (9)  | H4—C6—C7    | 120.56 (10) |
| C20—N4—C8   | 110.49 (12) | C5—C6—C7    | 118.87 (15) |
| C20—N4—C14  | 111.95 (11) | C5—C6—H4    | 120.56 (10) |
| C1—N1—Fe1   | 166.58 (13) | H8—C16—C15  | 120.60 (10) |
| C19—N6—Fe1  | 113.53 (10) | C17—C16—C15 | 118.81 (15) |
| C15—N6—Fe1  | 127.83 (11) | C17—C16—H8  | 120.60 (10) |
| C15—N6—C19  | 118.60 (14) | C16—C17—C18 | 118.98 (16) |
| C2—N2—Fe1   | 169.05 (13) | H9—C17—C18  | 120.51 (10) |
| C8—C7—N3    | 116.53 (13) | H9—C17—C16  | 120.51 (10) |
| C6—C7—N3    | 122.38 (14) | H2—C4—C3    | 120.62 (10) |
| C6—C7—C8    | 121.08 (14) | C5—C4—C3    | 118.77 (16) |
| C20—C19—N6  | 114.30 (13) | C5—C4—H2    | 120.62 (10) |
| C18—C19—N6  | 122.32 (14) | C21—C22—H19 | 120.98 (11) |
| C18—C19—C20 | 123.38 (14) | C23—C22—H19 | 120.98 (12) |
| C7—C8—N4    | 112.61 (12) | C23—C22—C21 | 118.04 (18) |
| H5—C8—N4    | 109.07 (8)  | C22—C21—Br3 | 119.59 (14) |
| H5—C8—C7    | 109.07 (8)  | C26—C21—Br3 | 118.29 (15) |
| H6—C8—N4    | 109.07 (8)  | C26—C21—C22 | 122.11 (18) |
| H6—C8—C7    | 109.07 (8)  | C4—C5—C6    | 118.99 (15) |
| H6—C8—H5    | 107.8       | H3—C5—C6    | 120.50 (10) |
| N2—C2—S2    | 179.25 (14) | H3—C5—C4    | 120.50 (10) |
| C14—C13—N5  | 115.13 (13) | H20—C23—C22 | 119.80 (12) |
| C12—C13—N5  | 122.22 (14) | C24—C23—C22 | 120.39 (19) |
| C12—C13—C14 | 122.53 (14) | C24—C23—H20 | 119.80 (13) |
| C13—C14—N4  | 109.58 (12) | H23—C26—C21 | 120.42 (12) |
| H18—C14—N4  | 109.75 (8)  | C25—C26—C21 | 119.2 (2)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| H18—C14—C13     | 109.75 (8)   | C25—C26—H23     | 120.42 (13)  |
| H17—C14—N4      | 109.75 (8)   | H21—C24—C23     | 120.09 (13)  |
| H17—C14—C13     | 109.75 (8)   | C25—C24—C23     | 119.8 (2)    |
| H17—C14—H18     | 108.2        | C25—C24—H21     | 120.09 (13)  |
| N1—C1—S1        | 179.18 (14)  | C24—C25—C26     | 120.5 (2)    |
| H16—C12—C13     | 120.56 (9)   | H22—C25—C26     | 119.76 (13)  |
| C11—C12—C13     | 118.88 (15)  | H22—C25—C24     | 119.76 (13)  |
| C11—C12—H16     | 120.56 (10)  |                 |              |
| Fe1—N5—C13—C14  | −6.05 (11)   | C7—C8—N4—C20    | 117.85 (13)  |
| Fe1—N5—C13—C12  | 177.94 (10)  | C7—C6—C5—C4     | −0.06 (19)   |
| Fe1—N5—C9—C10   | −178.65 (13) | C19—N6—C15—C16  | 1.98 (16)    |
| Fe1—N3—C7—C8    | −3.30 (11)   | C19—C20—N4—C8   | −78.12 (13)  |
| Fe1—N3—C7—C6    | 177.00 (11)  | C19—C20—N4—C14  | 156.88 (12)  |
| Fe1—N3—C3—C4    | −177.06 (13) | C19—C18—C17—C16 | 2.35 (18)    |
| Fe1—N4—C8—C7    | 1.10 (10)    | C8—N4—C14—C13   | 83.44 (12)   |
| Fe1—N4—C14—C13  | −36.92 (9)   | C8—C7—N3—C3     | 178.73 (14)  |
| Fe1—N4—C20—C19  | 41.34 (9)    | C8—C7—C6—C5     | −179.00 (16) |
| Fe1—N6—C19—C20  | 2.95 (11)    | C13—N5—C9—C10   | −0.75 (16)   |
| Fe1—N6—C19—C18  | −178.38 (10) | C13—C14—N4—C20  | −152.16 (12) |
| Fe1—N6—C15—C16  | 179.33 (13)  | C13—C12—C11—C10 | −0.90 (17)   |
| Br3—C21—C22—C23 | 178.82 (14)  | C14—C13—N5—C9   | 175.74 (13)  |
| Br3—C21—C26—C25 | −179.19 (16) | C14—C13—C12—C11 | −174.61 (15) |
| N5—C13—C14—N4   | 29.30 (13)   | C12—C13—N5—C9   | −0.28 (17)   |
| N5—C13—C12—C11  | 1.10 (17)    | C12—C11—C10—C9  | −0.06 (18)   |
| N5—C9—C10—C11   | 0.92 (18)    | C20—C19—N6—C15  | −179.33 (12) |
| N3—C7—C8—N4     | 1.40 (14)    | C20—C19—C18—C17 | 177.04 (15)  |
| N3—C7—C6—C5     | 0.68 (18)    | C18—C19—N6—C15  | −0.66 (18)   |
| N3—C3—C4—C5     | −0.1 (2)     | C18—C17—C16—C15 | −1.12 (19)   |
| N4—C8—C7—C6     | −178.90 (13) | C3—N3—C7—C6     | −0.97 (17)   |
| N4—C14—C13—C12  | −154.70 (12) | C3—C4—C5—C6     | −0.2 (2)     |
| N4—C20—C19—N6   | −30.00 (14)  | C22—C21—C26—C25 | 0.1 (2)      |
| N4—C20—C19—C18  | 151.34 (12)  | C22—C23—C24—C25 | 0.2 (2)      |
| N6—C19—C18—C17  | −1.51 (17)   | C21—C22—C23—C24 | 0.3 (2)      |
| N6—C15—C16—C17  | −1.10 (18)   | C21—C26—C25—C24 | 0.4 (2)      |
| C7—N3—C3—C4     | 0.66 (16)    | C23—C22—C21—C26 | −0.5 (2)     |
| C7—C8—N4—C14    | −116.93 (13) | C23—C24—C25—C26 | −0.6 (2)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate (br\_175K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.4045$  (4) Å

$b = 12.7878$  (3) Å

$c = 15.3126$  (4) Å

$\beta = 109.906$  (3)°

$V = 2652.09$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.551$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 31680 reflections

$\theta = 2.0$ – $30.2^\circ$

$\mu = 2.26 \text{ mm}^{-1}$   
 $T = 175 \text{ K}$

Block, red  
 $0.36 \times 0.26 \times 0.09 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.359$ ,  $T_{\max} = 1.000$   
 65372 measured reflections  
 6581 independent reflections  
 5419 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.028$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -17 \rightarrow 17$   
 $l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.075$   
 $S = 1.04$   
 6581 reflections  
 335 parameters  
 0 restraints

43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 1.0843P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.04 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.55 \text{ e } \text{\AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.660795 (16) | 0.337245 (18) | 0.319227 (15) | 0.02315 (6)                      |
| Br3 | 0.63688 (4)   | 0.03017 (4)   | 0.55732 (4)   | 0.06116 (9)                      |
| S2  | 0.47652 (3)   | 0.04447 (4)   | 0.16794 (3)   | 0.03678 (11)                     |
| S1  | 0.74467 (5)   | 0.30069 (4)   | 0.04691 (3)   | 0.04497 (13)                     |
| N5  | 0.76986 (10)  | 0.24359 (10)  | 0.39172 (9)   | 0.0250 (3)                       |
| N3  | 0.61040 (10)  | 0.34470 (11)  | 0.42358 (9)   | 0.0256 (3)                       |
| N4  | 0.75288 (9)   | 0.45116 (10)  | 0.38541 (9)   | 0.0237 (3)                       |
| N6  | 0.57475 (10)  | 0.45669 (11)  | 0.26131 (9)   | 0.0261 (3)                       |
| N1  | 0.71138 (10)  | 0.33523 (11)  | 0.21455 (9)   | 0.0288 (3)                       |
| N2  | 0.57217 (11)  | 0.22531 (12)  | 0.25753 (10)  | 0.0312 (3)                       |
| C7  | 0.65112 (12)  | 0.41965 (13)  | 0.48644 (10)  | 0.0258 (3)                       |
| C19 | 0.62396 (12)  | 0.54877 (13)  | 0.27106 (11)  | 0.0265 (3)                       |
| C2  | 0.53224 (12)  | 0.15052 (13)  | 0.21984 (11)  | 0.0267 (3)                       |
| C13 | 0.85555 (12)  | 0.29512 (13)  | 0.43225 (11)  | 0.0261 (3)                       |
| C8  | 0.73280 (13)  | 0.48221 (14)  | 0.47146 (11)  | 0.0303 (4)                       |
| H5  | 0.80128 (13)  | 0.47187 (14)  | 0.53239 (11)  | 0.0364 (4)*                      |
| H6  | 0.71278 (13)  | 0.56643 (14)  | 0.46688 (11)  | 0.0364 (4)*                      |
| C1  | 0.72534 (13)  | 0.32160 (13)  | 0.14475 (11)  | 0.0285 (3)                       |
| C12 | 0.94060 (13)  | 0.24505 (14)  | 0.48882 (12)  | 0.0313 (4)                       |
| H16 | 1.00913 (13)  | 0.28960 (14)  | 0.52222 (12)  | 0.0375 (4)*                      |
| C20 | 0.73349 (12)  | 0.54008 (13)  | 0.31884 (11)  | 0.0273 (3)                       |
| H11 | 0.76300 (12)  | 0.61378 (13)  | 0.35649 (11)  | 0.0328 (4)*                      |
| H12 | 0.77042 (12)  | 0.52612 (13)  | 0.26694 (11)  | 0.0328 (4)*                      |
| C18 | 0.57616 (13)  | 0.64188 (14)  | 0.23726 (12)  | 0.0324 (4)                       |

|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| H10 | 0.61779 (13) | 0.71594 (14)  | 0.24774 (12) | 0.0389 (4)* |
| C14 | 0.85438 (12) | 0.40882 (13)  | 0.40727 (12) | 0.0283 (3)  |
| H18 | 0.87857 (12) | 0.41807 (13)  | 0.34612 (12) | 0.0340 (4)* |
| H17 | 0.90643 (12) | 0.45289 (13)  | 0.46626 (12) | 0.0340 (4)* |
| C3  | 0.53795 (13) | 0.28536 (15)  | 0.43493 (12) | 0.0319 (4)  |
| H1  | 0.50442 (13) | 0.22356 (15)  | 0.38364 (12) | 0.0383 (4)* |
| C15 | 0.47676 (13) | 0.45639 (15)  | 0.21846 (12) | 0.0326 (4)  |
| H7  | 0.43628 (13) | 0.38201 (15)  | 0.21242 (12) | 0.0391 (4)* |
| C9  | 0.76727 (14) | 0.13998 (13)  | 0.40607 (11) | 0.0307 (4)  |
| H13 | 0.69723 (14) | 0.09768 (13)  | 0.37347 (11) | 0.0369 (4)* |
| C6  | 0.62068 (13) | 0.43847 (15)  | 0.56167 (12) | 0.0341 (4)  |
| H4  | 0.65531 (13) | 0.50109 (15)  | 0.61169 (12) | 0.0409 (5)* |
| C10 | 0.84941 (15) | 0.08430 (14)  | 0.46036 (12) | 0.0359 (4)  |
| H14 | 0.84488 (15) | −0.00080 (14) | 0.46997 (12) | 0.0431 (5)* |
| C11 | 0.93742 (14) | 0.13766 (15)  | 0.50238 (12) | 0.0360 (4)  |
| H15 | 1.00386 (14) | 0.09548 (15)  | 0.54590 (12) | 0.0432 (5)* |
| C16 | 0.42436 (14) | 0.54641 (16)  | 0.18136 (13) | 0.0385 (4)  |
| H8  | 0.34375 (14) | 0.54337 (16)  | 0.14559 (13) | 0.0462 (5)* |
| C17 | 0.47517 (14) | 0.64067 (16)  | 0.19002 (13) | 0.0379 (4)  |
| H9  | 0.43574 (14) | 0.71314 (16)  | 0.15981 (13) | 0.0455 (5)* |
| C4  | 0.50414 (14) | 0.29922 (16)  | 0.50864 (13) | 0.0383 (4)  |
| H2  | 0.44497 (14) | 0.24906 (16)  | 0.51604 (13) | 0.0460 (5)* |
| C22 | 0.75250 (14) | 0.20504 (16)  | 0.64493 (15) | 0.0409 (4)  |
| H19 | 0.76640 (14) | 0.21540 (16)  | 0.57894 (15) | 0.0491 (5)* |
| C21 | 0.69505 (14) | 0.12371 (15)  | 0.65642 (14) | 0.0365 (4)  |
| C5  | 0.54596 (14) | 0.37736 (17)  | 0.57318 (13) | 0.0403 (4)  |
| H3  | 0.52029 (14) | 0.39058 (17)  | 0.63241 (13) | 0.0483 (5)* |
| C23 | 0.79278 (15) | 0.27416 (18)  | 0.71899 (18) | 0.0511 (5)  |
| H20 | 0.83865 (15) | 0.34035 (18)  | 0.71160 (18) | 0.0613 (7)* |
| C26 | 0.67720 (18) | 0.10843 (18)  | 0.73826 (17) | 0.0518 (6)  |
| H23 | 0.63101 (18) | 0.04247 (18)  | 0.74551 (17) | 0.0622 (7)* |
| C24 | 0.77506 (18) | 0.2595 (2)    | 0.80189 (17) | 0.0584 (6)  |
| H21 | 0.80670 (18) | 0.3141 (2)    | 0.86028 (17) | 0.0701 (8)* |
| C25 | 0.7177 (2)   | 0.1765 (2)    | 0.81094 (17) | 0.0622 (7)  |
| H22 | 0.7043 (2)   | 0.1646 (2)    | 0.87698 (17) | 0.0747 (8)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02446 (11) | 0.02570 (12) | 0.01825 (11) | −0.00335 (9)  | 0.00590 (9)  | −0.00100 (8)  |
| Br3 | 0.06567 (17) | 0.04492 (14) | 0.05866 (16) | −0.00401 (11) | 0.00271 (13) | −0.00310 (11) |
| S2  | 0.0324 (2)   | 0.0346 (2)   | 0.0425 (3)   | −0.00691 (18) | 0.0117 (2)   | −0.01260 (19) |
| S1  | 0.0703 (4)   | 0.0414 (3)   | 0.0306 (2)   | 0.0141 (2)    | 0.0268 (2)   | 0.0063 (2)    |
| N5  | 0.0283 (7)   | 0.0249 (7)   | 0.0210 (6)   | −0.0021 (5)   | 0.0074 (5)   | 0.0001 (5)    |
| N3  | 0.0259 (6)   | 0.0312 (7)   | 0.0190 (6)   | −0.0024 (6)   | 0.0067 (5)   | −0.0012 (5)   |
| N4  | 0.0230 (6)   | 0.0238 (7)   | 0.0211 (6)   | −0.0038 (5)   | 0.0034 (5)   | −0.0001 (5)   |
| N6  | 0.0254 (7)   | 0.0307 (7)   | 0.0189 (6)   | −0.0005 (6)   | 0.0032 (5)   | −0.0019 (5)   |
| N1  | 0.0339 (7)   | 0.0318 (7)   | 0.0216 (7)   | 0.0001 (6)    | 0.0108 (6)   | 0.0007 (6)    |



|     |             |             |             |             |              |              |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| N2  | 0.0328 (7)  | 0.0325 (8)  | 0.0253 (7)  | −0.0086 (6) | 0.0060 (6)   | −0.0069 (6)  |
| C7  | 0.0272 (8)  | 0.0295 (8)  | 0.0189 (7)  | 0.0012 (6)  | 0.0055 (6)   | −0.0017 (6)  |
| C19 | 0.0280 (8)  | 0.0282 (8)  | 0.0208 (7)  | 0.0003 (6)  | 0.0052 (6)   | 0.0020 (6)   |
| C2  | 0.0257 (7)  | 0.0307 (9)  | 0.0225 (7)  | −0.0024 (7) | 0.0066 (6)   | −0.0028 (6)  |
| C13 | 0.0265 (8)  | 0.0266 (8)  | 0.0237 (7)  | 0.0004 (6)  | 0.0066 (6)   | 0.0019 (6)   |
| C8  | 0.0324 (9)  | 0.0328 (9)  | 0.0233 (8)  | −0.0060 (7) | 0.0063 (7)   | −0.0054 (7)  |
| C1  | 0.0358 (9)  | 0.0265 (8)  | 0.0241 (8)  | 0.0032 (7)  | 0.0114 (7)   | 0.0033 (6)   |
| C12 | 0.0296 (8)  | 0.0323 (9)  | 0.0281 (8)  | 0.0051 (7)  | 0.0050 (7)   | 0.0012 (7)   |
| C20 | 0.0267 (8)  | 0.0246 (8)  | 0.0275 (8)  | −0.0024 (6) | 0.0053 (7)   | 0.0033 (6)   |
| C18 | 0.0343 (9)  | 0.0322 (9)  | 0.0283 (8)  | 0.0037 (7)  | 0.0076 (7)   | 0.0052 (7)   |
| C14 | 0.0240 (7)  | 0.0276 (8)  | 0.0306 (8)  | −0.0023 (6) | 0.0057 (7)   | 0.0027 (7)   |
| C3  | 0.0297 (8)  | 0.0392 (10) | 0.0276 (8)  | −0.0072 (7) | 0.0107 (7)   | −0.0019 (7)  |
| C15 | 0.0250 (8)  | 0.0412 (10) | 0.0263 (8)  | −0.0016 (7) | 0.0020 (7)   | −0.0048 (7)  |
| C9  | 0.0415 (10) | 0.0251 (8)  | 0.0262 (8)  | −0.0023 (7) | 0.0124 (7)   | 0.0004 (6)   |
| C6  | 0.0375 (9)  | 0.0422 (10) | 0.0229 (8)  | 0.0014 (8)  | 0.0107 (7)   | −0.0044 (7)  |
| C10 | 0.0497 (11) | 0.0262 (9)  | 0.0316 (9)  | 0.0039 (8)  | 0.0134 (8)   | 0.0033 (7)   |
| C11 | 0.0410 (10) | 0.0338 (9)  | 0.0304 (9)  | 0.0087 (8)  | 0.0085 (8)   | 0.0049 (7)   |
| C16 | 0.0285 (9)  | 0.0509 (12) | 0.0293 (9)  | 0.0057 (8)  | 0.0010 (7)   | −0.0000 (8)  |
| C17 | 0.0354 (10) | 0.0427 (11) | 0.0312 (9)  | 0.0108 (8)  | 0.0055 (8)   | 0.0062 (8)   |
| C4  | 0.0331 (9)  | 0.0523 (11) | 0.0332 (9)  | −0.0059 (8) | 0.0161 (8)   | 0.0019 (8)   |
| C22 | 0.0378 (10) | 0.0428 (11) | 0.0460 (11) | 0.0019 (8)  | 0.0191 (9)   | 0.0101 (9)   |
| C21 | 0.0337 (9)  | 0.0345 (9)  | 0.0417 (10) | 0.0019 (7)  | 0.0132 (8)   | 0.0063 (8)   |
| C5  | 0.0410 (10) | 0.0567 (12) | 0.0283 (9)  | 0.0025 (9)  | 0.0185 (8)   | −0.0015 (8)  |
| C23 | 0.0313 (10) | 0.0431 (12) | 0.0732 (16) | −0.0009 (9) | 0.0103 (10)  | 0.0014 (11)  |
| C26 | 0.0626 (14) | 0.0455 (12) | 0.0591 (14) | 0.0050 (11) | 0.0358 (12)  | 0.0143 (11)  |
| C24 | 0.0568 (14) | 0.0569 (15) | 0.0471 (13) | 0.0206 (12) | −0.0009 (11) | −0.0081 (11) |
| C25 | 0.0880 (19) | 0.0595 (15) | 0.0466 (13) | 0.0236 (14) | 0.0326 (13)  | 0.0097 (11)  |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N5  | 1.9867 (14) | C18—H10 | 1.1030    |
| Fe1—N3  | 1.9696 (13) | C18—C17 | 1.387 (3) |
| Fe1—N4  | 1.9982 (13) | C14—H18 | 1.1110    |
| Fe1—N6  | 1.9767 (14) | C14—H17 | 1.1110    |
| Fe1—N1  | 1.9739 (14) | C3—H1   | 1.1030    |
| Fe1—N2  | 1.9365 (14) | C3—C4   | 1.384 (2) |
| Br3—C21 | 1.889 (2)   | C15—H7  | 1.1030    |
| S2—C2   | 1.6372 (17) | C15—C16 | 1.388 (3) |
| S1—C1   | 1.6363 (17) | C9—H13  | 1.1030    |
| N5—C13  | 1.350 (2)   | C9—C10  | 1.389 (3) |
| N5—C9   | 1.346 (2)   | C6—H4   | 1.1030    |
| N3—C7   | 1.343 (2)   | C6—C5   | 1.389 (3) |
| N3—C3   | 1.349 (2)   | C10—H14 | 1.1030    |
| N4—C8   | 1.496 (2)   | C10—C11 | 1.390 (3) |
| N4—C20  | 1.489 (2)   | C11—H15 | 1.1030    |
| N4—C14  | 1.487 (2)   | C16—H8  | 1.1030    |
| N6—C19  | 1.356 (2)   | C16—C17 | 1.393 (3) |
| N6—C15  | 1.339 (2)   | C17—H9  | 1.1030    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| N1—C1      | 1.166 (2)   | C4—H2       | 1.1030      |
| N2—C2      | 1.163 (2)   | C4—C5       | 1.390 (3)   |
| C7—C8      | 1.504 (2)   | C22—H19     | 1.1030      |
| C7—C6      | 1.386 (2)   | C22—C21     | 1.377 (3)   |
| C19—C20    | 1.500 (2)   | C22—C23     | 1.399 (3)   |
| C19—C18    | 1.385 (2)   | C21—C26     | 1.376 (3)   |
| C13—C12    | 1.392 (2)   | C5—H3       | 1.1030      |
| C13—C14    | 1.502 (2)   | C23—H20     | 1.1030      |
| C8—H5      | 1.1110      | C23—C24     | 1.390 (3)   |
| C8—H6      | 1.1110      | C26—H23     | 1.1030      |
| C12—H16    | 1.1030      | C26—C25     | 1.376 (4)   |
| C12—C11    | 1.392 (3)   | C24—H21     | 1.1030      |
| C20—H11    | 1.1110      | C24—C25     | 1.381 (4)   |
| C20—H12    | 1.1110      | C25—H22     | 1.1030      |
|            |             |             |             |
| N3—Fe1—N5  | 90.90 (5)   | H10—C18—C19 | 120.49 (10) |
| N4—Fe1—N5  | 83.91 (5)   | C17—C18—C19 | 119.02 (17) |
| N4—Fe1—N3  | 85.27 (5)   | C17—C18—H10 | 120.49 (11) |
| N6—Fe1—N5  | 166.47 (6)  | C13—C14—N4  | 109.56 (13) |
| N6—Fe1—N3  | 88.77 (5)   | H18—C14—N4  | 109.76 (8)  |
| N6—Fe1—N4  | 82.58 (5)   | H18—C14—C13 | 109.76 (9)  |
| N1—Fe1—N5  | 90.32 (6)   | H17—C14—N4  | 109.76 (8)  |
| N1—Fe1—N3  | 177.97 (6)  | H17—C14—C13 | 109.76 (9)  |
| N1—Fe1—N4  | 93.24 (6)   | H17—C14—H18 | 108.2       |
| N1—Fe1—N6  | 89.67 (6)   | H1—C3—N3    | 118.81 (9)  |
| N2—Fe1—N5  | 95.25 (6)   | C4—C3—N3    | 122.38 (17) |
| N2—Fe1—N3  | 93.89 (6)   | C4—C3—H1    | 118.81 (11) |
| N2—Fe1—N4  | 178.80 (6)  | H7—C15—N6   | 118.86 (10) |
| N2—Fe1—N6  | 98.27 (6)   | C16—C15—N6  | 122.28 (17) |
| N2—Fe1—N1  | 87.62 (6)   | C16—C15—H7  | 118.86 (11) |
| C13—N5—Fe1 | 112.83 (11) | H13—C9—N5   | 118.75 (10) |
| C9—N5—Fe1  | 128.62 (12) | C10—C9—N5   | 122.50 (17) |
| C9—N5—C13  | 118.53 (15) | C10—C9—H13  | 118.75 (11) |
| C7—N3—Fe1  | 115.10 (11) | H4—C6—C7    | 120.47 (11) |
| C3—N3—Fe1  | 126.33 (11) | C5—C6—C7    | 119.06 (17) |
| C3—N3—C7   | 118.52 (14) | C5—C6—H4    | 120.47 (11) |
| C8—N4—Fe1  | 110.26 (10) | H14—C10—C9  | 120.58 (11) |
| C20—N4—Fe1 | 105.79 (9)  | C11—C10—C9  | 118.84 (17) |
| C20—N4—C8  | 110.67 (13) | C11—C10—H14 | 120.58 (11) |
| C14—N4—Fe1 | 106.31 (10) | C10—C11—C12 | 119.11 (17) |
| C14—N4—C8  | 111.63 (13) | H15—C11—C12 | 120.44 (11) |
| C14—N4—C20 | 111.91 (12) | H15—C11—C10 | 120.44 (11) |
| C19—N6—Fe1 | 113.39 (11) | H8—C16—C15  | 120.53 (11) |
| C15—N6—Fe1 | 127.82 (12) | C17—C16—C15 | 118.94 (17) |
| C15—N6—C19 | 118.74 (15) | C17—C16—H8  | 120.53 (11) |
| C1—N1—Fe1  | 166.63 (14) | C16—C17—C18 | 118.90 (17) |
| C2—N2—Fe1  | 169.26 (14) | H9—C17—C18  | 120.55 (11) |
| C8—C7—N3   | 116.34 (14) | H9—C17—C16  | 120.55 (11) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C6—C7—N3        | 122.34 (16)  | H2—C4—C3        | 120.53 (11)  |
| C6—C7—C8        | 121.32 (15)  | C5—C4—C3        | 118.94 (17)  |
| C20—C19—N6      | 114.39 (14)  | C5—C4—H2        | 120.53 (11)  |
| C18—C19—N6      | 122.05 (15)  | C21—C22—H19     | 120.82 (12)  |
| C18—C19—C20     | 123.54 (15)  | C23—C22—H19     | 120.82 (13)  |
| N2—C2—S2        | 179.33 (15)  | C23—C22—C21     | 118.37 (19)  |
| C12—C13—N5      | 122.33 (16)  | C22—C21—Br3     | 119.79 (15)  |
| C14—C13—N5      | 115.24 (14)  | C26—C21—Br3     | 118.18 (16)  |
| C14—C13—C12     | 122.32 (15)  | C26—C21—C22     | 122.0 (2)    |
| C7—C8—N4        | 112.91 (13)  | C4—C5—C6        | 118.76 (16)  |
| H5—C8—N4        | 109.00 (8)   | H3—C5—C6        | 120.62 (11)  |
| H5—C8—C7        | 109.00 (9)   | H3—C5—C4        | 120.62 (11)  |
| H6—C8—N4        | 109.00 (9)   | H20—C23—C22     | 119.97 (13)  |
| H6—C8—C7        | 109.00 (9)   | C24—C23—C22     | 120.1 (2)    |
| H6—C8—H5        | 107.8        | C24—C23—H20     | 119.97 (15)  |
| N1—C1—S1        | 179.19 (16)  | H23—C26—C21     | 120.42 (13)  |
| H16—C12—C13     | 120.66 (10)  | C25—C26—C21     | 119.2 (2)    |
| C11—C12—C13     | 118.67 (17)  | C25—C26—H23     | 120.42 (14)  |
| C11—C12—H16     | 120.66 (11)  | H21—C24—C23     | 120.09 (15)  |
| C19—C20—N4      | 108.14 (13)  | C25—C24—C23     | 119.8 (2)    |
| H11—C20—N4      | 110.07 (8)   | C25—C24—H21     | 120.09 (15)  |
| H11—C20—C19     | 110.07 (9)   | C24—C25—C26     | 120.6 (2)    |
| H12—C20—N4      | 110.07 (8)   | H22—C25—C26     | 119.72 (14)  |
| H12—C20—C19     | 110.07 (9)   | H22—C25—C24     | 119.72 (15)  |
| H12—C20—H11     | 108.4        |                 |              |
| Fe1—N5—C13—C12  | 177.82 (11)  | C7—C8—N4—C14    | −117.03 (14) |
| Fe1—N5—C13—C14  | −5.82 (11)   | C7—C6—C5—C4     | −0.0 (2)     |
| Fe1—N5—C9—C10   | −178.60 (14) | C19—N6—C15—C16  | 1.93 (17)    |
| Fe1—N3—C7—C8    | −3.56 (12)   | C19—C20—N4—C8   | −77.88 (14)  |
| Fe1—N3—C7—C6    | 176.95 (12)  | C19—C20—N4—C14  | 156.91 (13)  |
| Fe1—N3—C3—C4    | −177.03 (15) | C19—C18—C17—C16 | 2.6 (2)      |
| Fe1—N4—C8—C7    | 0.90 (10)    | C13—N5—C9—C10   | −0.43 (17)   |
| Fe1—N4—C20—C19  | 41.55 (10)   | C13—C12—C11—C10 | −0.98 (19)   |
| Fe1—N4—C14—C13  | −36.96 (10)  | C13—C14—N4—C8   | 83.32 (14)   |
| Fe1—N6—C19—C20  | 3.16 (12)    | C13—C14—N4—C20  | −152.01 (13) |
| Fe1—N6—C19—C18  | −178.35 (11) | C8—C7—N3—C3     | 178.92 (15)  |
| Fe1—N6—C15—C16  | 179.19 (14)  | C8—C7—C6—C5     | −178.98 (17) |
| Br3—C21—C22—C23 | 178.68 (15)  | C12—C13—N5—C9   | −0.64 (19)   |
| Br3—C21—C26—C25 | −179.11 (17) | C12—C11—C10—C9  | −0.0 (2)     |
| N5—C13—C12—C11  | 1.34 (18)    | C20—C19—N6—C15  | −179.19 (13) |
| N5—C13—C14—N4   | 29.21 (14)   | C20—C19—C18—C17 | 176.80 (17)  |
| N5—C9—C10—C11   | 0.7 (2)      | C18—C19—N6—C15  | −0.70 (19)   |
| N3—C7—C8—N4     | 1.69 (16)    | C18—C17—C16—C15 | −1.4 (2)     |
| N3—C7—C6—C5     | 0.50 (19)    | C14—C13—N5—C9   | 175.73 (14)  |
| N3—C3—C4—C5     | 0.3 (2)      | C14—C13—C12—C11 | −174.77 (16) |
| N4—C8—C7—C6     | −178.81 (14) | C3—N3—C7—C6     | −0.57 (18)   |
| N4—C20—C19—N6   | −30.31 (15)  | C3—C4—C5—C6     | −0.4 (2)     |

|                |              |                 |          |
|----------------|--------------|-----------------|----------|
| N4—C20—C19—C18 | 151.23 (13)  | C22—C21—C26—C25 | 0.1 (2)  |
| N4—C14—C13—C12 | −154.42 (13) | C22—C23—C24—C25 | 0.1 (2)  |
| N6—C19—C18—C17 | −1.56 (19)   | C21—C22—C23—C24 | 0.4 (2)  |
| N6—C15—C16—C17 | −0.9 (2)     | C21—C26—C25—C24 | 0.4 (3)  |
| C7—N3—C3—C4    | 0.18 (18)    | C23—C22—C21—C26 | −0.6 (2) |
| C7—C8—N4—C20   | 117.61 (14)  | C23—C24—C25—C26 | −0.5 (2) |

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) bromobenzene monosolvate (br\_180K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 14.4172$  (4) Å

$b = 12.7845$  (3) Å

$c = 15.3183$  (4) Å

$\beta = 109.913$  (3)°

$V = 2654.61$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.550$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 30794 reflections

$\theta = 2.0$ – $30.2^\circ$

$\mu = 2.26$  mm<sup>−1</sup>

$T = 180$  K

Block, red

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.357$ ,  $T_{\max} = 1.000$

66099 measured reflections

6586 independent reflections

5362 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -19 \rightarrow 19$

$k = -17 \rightarrow 17$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.080$

$S = 1.04$

6586 reflections

335 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 1.1578P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.0004$

$\Delta\rho_{\max} = 1.09$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.48$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.660715 (17) | 0.337109 (19) | 0.319070 (16) | 0.02422 (7)                      |
| Br3 | 0.63695 (5)   | 0.03000 (5)   | 0.55744 (4)   | 0.06362 (10)                     |
| S2  | 0.47653 (4)   | 0.04422 (4)   | 0.16796 (4)   | 0.03815 (12)                     |
| S1  | 0.74459 (5)   | 0.30070 (4)   | 0.04690 (4)   | 0.04669 (14)                     |
| N6  | 0.76999 (10)  | 0.24353 (11)  | 0.39169 (9)   | 0.0262 (3)                       |
| N3  | 0.61034 (10)  | 0.34455 (11)  | 0.42354 (9)   | 0.0266 (3)                       |
| N5  | 0.75273 (10)  | 0.45110 (11)  | 0.38526 (9)   | 0.0245 (3)                       |

|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| N1  | 0.71128 (11) | 0.33520 (12)  | 0.21436 (10) | 0.0301 (3)  |
| N7  | 0.57465 (11) | 0.45667 (12)  | 0.26131 (9)  | 0.0275 (3)  |
| N2  | 0.57212 (11) | 0.22494 (13)  | 0.25743 (10) | 0.0325 (3)  |
| C7  | 0.65101 (12) | 0.41972 (14)  | 0.48626 (11) | 0.0264 (3)  |
| C19 | 0.62379 (13) | 0.54863 (14)  | 0.27104 (11) | 0.0274 (3)  |
| C13 | 0.85554 (12) | 0.29532 (14)  | 0.43220 (11) | 0.0268 (3)  |
| C2  | 0.53229 (12) | 0.15004 (14)  | 0.21975 (11) | 0.0279 (3)  |
| C8  | 0.73280 (13) | 0.48214 (14)  | 0.47146 (12) | 0.0311 (4)  |
| H5  | 0.80120 (13) | 0.47151 (14)  | 0.53234 (12) | 0.0373 (4)* |
| H6  | 0.71303 (13) | 0.56644 (14)  | 0.46705 (12) | 0.0373 (4)* |
| C1  | 0.72522 (13) | 0.32169 (13)  | 0.14469 (12) | 0.0294 (4)  |
| C12 | 0.94050 (14) | 0.24536 (15)  | 0.48883 (12) | 0.0326 (4)  |
| H16 | 1.00891 (14) | 0.29003 (15)  | 0.52220 (12) | 0.0391 (5)* |
| C14 | 0.85410 (12) | 0.40887 (14)  | 0.40712 (13) | 0.0294 (4)  |
| H18 | 0.87821 (12) | 0.41810 (14)  | 0.34596 (13) | 0.0353 (4)* |
| H17 | 0.90607 (12) | 0.45308 (14)  | 0.46601 (13) | 0.0353 (4)* |
| C9  | 0.76740 (14) | 0.13994 (14)  | 0.40605 (12) | 0.0313 (4)  |
| H13 | 0.69743 (14) | 0.09758 (14)  | 0.37353 (12) | 0.0375 (5)* |
| C18 | 0.57621 (14) | 0.64211 (15)  | 0.23737 (13) | 0.0340 (4)  |
| H10 | 0.61795 (14) | 0.71609 (15)  | 0.24782 (13) | 0.0408 (5)* |
| C20 | 0.73344 (13) | 0.54015 (13)  | 0.31900 (12) | 0.0288 (4)  |
| H11 | 0.76275 (13) | 0.61382 (13)  | 0.35681 (12) | 0.0345 (4)* |
| H12 | 0.77047 (13) | 0.52647 (13)  | 0.26718 (12) | 0.0345 (4)* |
| C15 | 0.47672 (13) | 0.45651 (16)  | 0.21853 (12) | 0.0343 (4)  |
| H7  | 0.43625 (13) | 0.38215 (16)  | 0.21265 (12) | 0.0412 (5)* |
| C3  | 0.53793 (13) | 0.28509 (15)  | 0.43484 (13) | 0.0335 (4)  |
| H1  | 0.50449 (13) | 0.22320 (15)  | 0.38361 (13) | 0.0402 (5)* |
| C6  | 0.62044 (14) | 0.43829 (16)  | 0.56154 (12) | 0.0352 (4)  |
| H4  | 0.65509 (14) | 0.50082 (16)  | 0.61165 (12) | 0.0423 (5)* |
| C10 | 0.84957 (15) | 0.08437 (15)  | 0.46029 (13) | 0.0373 (4)  |
| H14 | 0.84519 (15) | −0.00080 (15) | 0.46970 (13) | 0.0448 (5)* |
| C11 | 0.93753 (15) | 0.13786 (16)  | 0.50259 (13) | 0.0375 (4)  |
| H15 | 1.00388 (15) | 0.09581 (16)  | 0.54633 (13) | 0.0450 (5)* |
| C16 | 0.42435 (14) | 0.54635 (17)  | 0.18139 (13) | 0.0393 (4)  |
| H8  | 0.34385 (14) | 0.54323 (17)  | 0.14538 (13) | 0.0472 (5)* |
| C17 | 0.47529 (15) | 0.64096 (17)  | 0.19033 (14) | 0.0397 (4)  |
| H9  | 0.43586 (15) | 0.71354 (17)  | 0.16046 (14) | 0.0477 (5)* |
| C4  | 0.50414 (15) | 0.29911 (18)  | 0.50860 (14) | 0.0408 (4)  |
| H2  | 0.44503 (15) | 0.24897 (18)  | 0.51607 (14) | 0.0489 (5)* |
| C21 | 0.69514 (14) | 0.12383 (16)  | 0.65634 (15) | 0.0382 (4)  |
| C22 | 0.75250 (15) | 0.20496 (17)  | 0.64479 (15) | 0.0426 (5)  |
| H19 | 0.76630 (15) | 0.21536 (17)  | 0.57879 (15) | 0.0512 (6)* |
| C5  | 0.54593 (15) | 0.37742 (18)  | 0.57305 (14) | 0.0421 (5)  |
| H3  | 0.52024 (15) | 0.39080 (18)  | 0.63222 (14) | 0.0505 (6)* |
| C23 | 0.79295 (15) | 0.27405 (19)  | 0.71904 (19) | 0.0532 (6)  |
| H20 | 0.83896 (15) | 0.34014 (19)  | 0.71178 (19) | 0.0638 (7)* |
| C26 | 0.67734 (18) | 0.10848 (19)  | 0.73826 (18) | 0.0541 (6)  |
| H23 | 0.63136 (18) | 0.04239 (19)  | 0.74559 (18) | 0.0649 (7)* |

|     |              |            |              |             |
|-----|--------------|------------|--------------|-------------|
| C24 | 0.77517 (19) | 0.2594 (2) | 0.80173 (18) | 0.0608 (7)  |
| H21 | 0.80693 (19) | 0.3139 (2) | 0.86017 (18) | 0.0729 (8)* |
| C25 | 0.71176 (2)  | 0.1766 (2) | 0.81068 (18) | 0.0634 (7)  |
| H22 | 0.7039 (2)   | 0.1650 (2) | 0.87659 (18) | 0.0761 (9)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02555 (12) | 0.02687 (13) | 0.01915 (11) | −0.00324 (10) | 0.00621 (9)  | −0.00070 (9)  |
| Br3 | 0.06849 (19) | 0.04661 (15) | 0.06095 (17) | −0.00408 (12) | 0.00281 (14) | −0.00335 (11) |
| S2  | 0.0339 (2)   | 0.0361 (3)   | 0.0438 (3)   | −0.00716 (19) | 0.0124 (2)   | −0.0127 (2)   |
| S1  | 0.0730 (4)   | 0.0426 (3)   | 0.0322 (2)   | 0.0146 (3)    | 0.0279 (3)   | 0.0067 (2)    |
| N6  | 0.0302 (7)   | 0.0264 (7)   | 0.0211 (6)   | −0.0022 (6)   | 0.0077 (6)   | −0.0002 (5)   |
| N3  | 0.0273 (7)   | 0.0321 (7)   | 0.0200 (7)   | −0.0029 (6)   | 0.0074 (6)   | −0.0013 (6)   |
| N5  | 0.0239 (7)   | 0.0240 (7)   | 0.0229 (7)   | −0.0042 (5)   | 0.0046 (5)   | −0.0003 (5)   |
| N1  | 0.0361 (8)   | 0.0328 (8)   | 0.0232 (7)   | −0.0002 (6)   | 0.0124 (6)   | 0.0008 (6)    |
| N7  | 0.0272 (7)   | 0.0322 (8)   | 0.0200 (6)   | −0.0016 (6)   | 0.0039 (6)   | −0.0023 (6)   |
| N2  | 0.0336 (8)   | 0.0349 (8)   | 0.0262 (7)   | −0.0084 (7)   | 0.0066 (6)   | −0.0060 (6)   |
| C7  | 0.0268 (8)   | 0.0307 (8)   | 0.0196 (7)   | 0.0018 (7)    | 0.0052 (6)   | −0.0008 (6)   |
| C19 | 0.0287 (8)   | 0.0288 (9)   | 0.0221 (8)   | −0.0002 (7)   | 0.0054 (7)   | 0.0021 (6)    |
| C13 | 0.0272 (8)   | 0.0272 (8)   | 0.0241 (8)   | 0.0002 (7)    | 0.0061 (7)   | 0.0016 (6)    |
| C2  | 0.0274 (8)   | 0.0326 (9)   | 0.0227 (8)   | −0.0021 (7)   | 0.0072 (7)   | −0.0032 (7)   |
| C8  | 0.0333 (9)   | 0.0329 (9)   | 0.0240 (8)   | −0.0061 (7)   | 0.0058 (7)   | −0.0054 (7)   |
| C1  | 0.0364 (9)   | 0.0272 (8)   | 0.0253 (8)   | 0.0036 (7)    | 0.0113 (7)   | 0.0033 (7)    |
| C12 | 0.0305 (9)   | 0.0343 (10)  | 0.0294 (9)   | 0.0049 (8)    | 0.0056 (7)   | 0.0008 (7)    |
| C14 | 0.0257 (8)   | 0.0276 (9)   | 0.0322 (9)   | −0.0027 (7)   | 0.0061 (7)   | 0.0023 (7)    |
| C9  | 0.0411 (10)  | 0.0266 (9)   | 0.0262 (8)   | −0.0021 (7)   | 0.0117 (8)   | 0.0008 (7)    |
| C18 | 0.0351 (9)   | 0.0329 (10)  | 0.0317 (9)   | 0.0042 (8)    | 0.0086 (8)   | 0.0065 (7)    |
| C20 | 0.0296 (9)   | 0.0254 (8)   | 0.0286 (8)   | −0.0030 (7)   | 0.0062 (7)   | 0.0036 (7)    |
| C15 | 0.0268 (9)   | 0.0434 (11)  | 0.0271 (9)   | −0.0025 (8)   | 0.0019 (7)   | −0.0043 (8)   |
| C3  | 0.0304 (9)   | 0.0406 (10)  | 0.0296 (9)   | −0.0068 (8)   | 0.0102 (7)   | −0.0019 (8)   |
| C6  | 0.0377 (10)  | 0.0435 (11)  | 0.0243 (9)   | 0.0021 (8)    | 0.0103 (8)   | −0.0057 (8)   |
| C10 | 0.0504 (12)  | 0.0277 (9)   | 0.0336 (9)   | 0.0041 (8)    | 0.0139 (9)   | 0.0036 (7)    |
| C11 | 0.0445 (11)  | 0.0346 (10)  | 0.0315 (9)   | 0.0099 (8)    | 0.0107 (8)   | 0.0048 (8)    |
| C16 | 0.0289 (9)   | 0.0524 (12)  | 0.0299 (9)   | 0.0048 (9)    | 0.0011 (7)   | −0.0008 (8)   |
| C17 | 0.0387 (10)  | 0.0440 (11)  | 0.0328 (9)   | 0.0114 (9)    | 0.0075 (8)   | 0.0070 (8)    |
| C4  | 0.0361 (10)  | 0.0557 (12)  | 0.0342 (10)  | −0.0048 (9)   | 0.0168 (8)   | 0.0025 (9)    |
| C21 | 0.0356 (10)  | 0.0354 (10)  | 0.0431 (11)  | 0.0023 (8)    | 0.0127 (8)   | 0.0055 (8)    |
| C22 | 0.0401 (11)  | 0.0444 (11)  | 0.0474 (12)  | 0.0013 (9)    | 0.0202 (9)   | 0.0094 (10)   |
| C5  | 0.0421 (11)  | 0.0593 (13)  | 0.0302 (10)  | 0.0023 (10)   | 0.0191 (9)   | −0.0022 (9)   |
| C23 | 0.0318 (10)  | 0.0450 (12)  | 0.0768 (17)  | −0.0011 (9)   | 0.0106 (11)  | 0.0024 (12)   |
| C26 | 0.0633 (14)  | 0.0473 (13)  | 0.0636 (15)  | 0.0060 (11)   | 0.0369 (13)  | 0.0158 (12)   |
| C24 | 0.0585 (15)  | 0.0596 (16)  | 0.0495 (14)  | 0.0215 (12)   | −0.0008 (11) | −0.0080 (12)  |
| C25 | 0.090 (2)    | 0.0609 (16)  | 0.0471 (14)  | 0.0240 (15)   | 0.0331 (14)  | 0.0093 (12)   |



*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N6    | 1.9892 (14) | C9—H13      | 1.1030      |
| Fe1—N3    | 1.9721 (14) | C9—C10      | 1.389 (3)   |
| Fe1—N5    | 1.9988 (14) | C18—H10     | 1.1030      |
| Fe1—N1    | 1.9751 (15) | C18—C17     | 1.386 (3)   |
| Fe1—N7    | 1.9777 (15) | C20—H11     | 1.1110      |
| Fe1—N2    | 1.9386 (15) | C20—H12     | 1.1110      |
| Br3—C21   | 1.890 (2)   | C15—H7      | 1.1030      |
| S2—C2     | 1.6345 (18) | C15—C16     | 1.386 (3)   |
| S1—C1     | 1.6367 (18) | C3—H1       | 1.1030      |
| N6—C13    | 1.350 (2)   | C3—C4       | 1.386 (3)   |
| N6—C9     | 1.345 (2)   | C6—H4       | 1.1030      |
| N3—C7     | 1.344 (2)   | C6—C5       | 1.386 (3)   |
| N3—C3     | 1.349 (2)   | C10—H14     | 1.1030      |
| N5—C8     | 1.497 (2)   | C10—C11     | 1.391 (3)   |
| N5—C14    | 1.485 (2)   | C11—H15     | 1.1030      |
| N5—C20    | 1.487 (2)   | C16—H8      | 1.1030      |
| N1—C1     | 1.164 (2)   | C16—C17     | 1.397 (3)   |
| N7—C19    | 1.354 (2)   | C17—H9      | 1.1030      |
| N7—C15    | 1.339 (2)   | C4—H2       | 1.1030      |
| N2—C2     | 1.164 (2)   | C4—C5       | 1.391 (3)   |
| C7—C8     | 1.504 (2)   | C21—C22     | 1.375 (3)   |
| C7—C6     | 1.388 (2)   | C21—C26     | 1.378 (3)   |
| C19—C18   | 1.387 (2)   | C22—H19     | 1.1030      |
| C19—C20   | 1.503 (2)   | C22—C23     | 1.401 (3)   |
| C13—C12   | 1.392 (2)   | C5—H3       | 1.1030      |
| C13—C14   | 1.500 (2)   | C23—H20     | 1.1030      |
| C8—H5     | 1.1110      | C23—C24     | 1.388 (4)   |
| C8—H6     | 1.1110      | C26—H23     | 1.1030      |
| C12—H16   | 1.1030      | C26—C25     | 1.374 (4)   |
| C12—C11   | 1.393 (3)   | C24—H21     | 1.1030      |
| C14—H18   | 1.1110      | C24—C25     | 1.381 (4)   |
| C14—H17   | 1.1110      | C25—H22     | 1.1030      |
| N3—Fe1—N6 | 90.90 (6)   | H13—C9—N6   | 118.79 (10) |
| N5—Fe1—N6 | 83.82 (6)   | C10—C9—N6   | 122.41 (18) |
| N5—Fe1—N3 | 85.26 (6)   | C10—C9—H13  | 118.79 (11) |
| N1—Fe1—N6 | 90.32 (6)   | H10—C18—C19 | 120.57 (11) |
| N1—Fe1—N3 | 177.94 (6)  | C17—C18—C19 | 118.86 (18) |
| N1—Fe1—N5 | 93.22 (6)   | C17—C18—H10 | 120.57 (12) |
| N7—Fe1—N6 | 166.35 (6)  | C19—C20—N5  | 108.04 (13) |
| N7—Fe1—N3 | 88.71 (6)   | H11—C20—N5  | 110.09 (9)  |
| N7—Fe1—N5 | 82.55 (6)   | H11—C20—C19 | 110.09 (9)  |
| N7—Fe1—N1 | 89.72 (6)   | H12—C20—N5  | 110.09 (9)  |
| N2—Fe1—N6 | 95.31 (6)   | H12—C20—C19 | 110.09 (9)  |
| N2—Fe1—N3 | 93.86 (6)   | H12—C20—H11 | 108.4       |
| N2—Fe1—N5 | 178.75 (6)  | H7—C15—N7   | 118.80 (11) |

|                |             |              |             |
|----------------|-------------|--------------|-------------|
| N2—Fe1—N1      | 87.68 (6)   | C16—C15—N7   | 122.41 (18) |
| N2—Fe1—N7      | 98.33 (6)   | C16—C15—H7   | 118.80 (12) |
| C13—N6—Fe1     | 112.80 (11) | H1—C3—N3     | 118.87 (10) |
| C9—N6—Fe1      | 128.52 (13) | C4—C3—N3     | 122.26 (18) |
| C9—N6—C13      | 118.66 (15) | C4—C3—H1     | 118.87 (12) |
| C7—N3—Fe1      | 115.02 (11) | H4—C6—C7     | 120.36 (11) |
| C3—N3—Fe1      | 126.28 (12) | C5—C6—C7     | 119.27 (18) |
| C3—N3—C7       | 118.64 (15) | C5—C6—H4     | 120.36 (11) |
| C8—N5—Fe1      | 110.31 (10) | H14—C10—C9   | 120.53 (11) |
| C14—N5—Fe1     | 106.34 (10) | C11—C10—C9   | 118.93 (18) |
| C14—N5—C8      | 111.54 (13) | C11—C10—H14  | 120.53 (11) |
| C20—N5—Fe1     | 105.93 (10) | C10—C11—C12  | 118.98 (18) |
| C20—N5—C8      | 110.58 (13) | H15—C11—C12  | 120.51 (12) |
| C20—N5—C14     | 111.90 (13) | H15—C11—C10  | 120.51 (11) |
| C1—N1—Fe1      | 166.65 (15) | H8—C16—C15   | 120.58 (12) |
| C19—N7—Fe1     | 113.37 (11) | C17—C16—C15  | 118.84 (18) |
| C15—N7—Fe1     | 127.89 (13) | C17—C16—H8   | 120.58 (11) |
| C15—N7—C19     | 118.69 (16) | C16—C17—C18  | 118.93 (18) |
| C2—N2—Fe1      | 169.21 (15) | H9—C17—C18   | 120.54 (12) |
| C8—C7—N3       | 116.43 (14) | H9—C17—C16   | 120.54 (11) |
| C6—C7—N3       | 122.11 (17) | H2—C4—C3     | 120.52 (12) |
| C6—C7—C8       | 121.45 (16) | C5—C4—C3     | 118.95 (18) |
| C18—C19—N7     | 122.21 (16) | C5—C4—H2     | 120.52 (11) |
| C20—C19—N7     | 114.51 (15) | C22—C21—Br3  | 119.86 (17) |
| C20—C19—C18    | 123.26 (16) | C26—C21—Br3  | 118.04 (17) |
| C12—C13—N6     | 122.25 (17) | C26—C21—C22  | 122.1 (2)   |
| C14—C13—N6     | 115.22 (15) | H19—C22—C21  | 120.87 (13) |
| C14—C13—C12    | 122.43 (16) | C23—C22—C21  | 118.3 (2)   |
| N2—C2—S2       | 179.35 (16) | C23—C22—H19  | 120.87 (14) |
| C7—C8—N5       | 112.84 (13) | C4—C5—C6     | 118.76 (17) |
| H5—C8—N5       | 109.02 (9)  | H3—C5—C6     | 120.62 (11) |
| H5—C8—C7       | 109.02 (9)  | H3—C5—C4     | 120.62 (11) |
| H6—C8—N5       | 109.02 (9)  | H20—C23—C22  | 119.97 (14) |
| H6—C8—C7       | 109.02 (10) | C24—C23—C22  | 120.1 (2)   |
| H6—C8—H5       | 107.8       | C24—C23—H20  | 119.97 (16) |
| N1—C1—S1       | 179.09 (16) | H23—C26—C21  | 120.44 (14) |
| H16—C12—C13    | 120.62 (11) | C25—C26—C21  | 119.1 (2)   |
| C11—C12—C13    | 118.76 (18) | C25—C26—H23  | 120.44 (15) |
| C11—C12—H16    | 120.62 (12) | H21—C24—C23  | 120.07 (16) |
| C13—C14—N5     | 109.64 (13) | C25—C24—C23  | 119.9 (2)   |
| H18—C14—N5     | 109.74 (9)  | C25—C24—H21  | 120.07 (16) |
| H18—C14—C13    | 109.74 (9)  | C24—C25—C26  | 120.6 (2)   |
| H17—C14—N5     | 109.74 (9)  | H22—C25—C26  | 119.70 (15) |
| H17—C14—C13    | 109.74 (9)  | H22—C25—C24  | 119.70 (16) |
| H17—C14—H18    | 108.2       |              |             |
| Fe1—N6—C13—C12 | 177.75 (11) | C7—C8—N5—C20 | 117.54 (15) |
| Fe1—N6—C13—C14 | −5.85 (12)  | C7—C6—C5—C4  | 0.2 (2)     |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Fe1—N6—C9—C10   | −178.65 (14) | C19—N7—C15—C16  | 2.06 (18)    |
| Fe1—N3—C7—C8    | −3.82 (13)   | C19—C18—C17—C16 | 2.3 (2)      |
| Fe1—N3—C7—C6    | 177.01 (12)  | C19—C20—N5—C8   | −78.15 (15)  |
| Fe1—N3—C3—C4    | −176.96 (16) | C19—C20—N5—C14  | 156.85 (13)  |
| Fe1—N5—C8—C7    | 0.69 (11)    | C13—N6—C9—C10   | −0.53 (18)   |
| Fe1—N5—C14—C13  | −37.04 (10)  | C13—C12—C11—C10 | −0.8 (2)     |
| Fe1—N5—C20—C19  | 41.37 (10)   | C13—C14—N5—C8   | 83.27 (15)   |
| Fe1—N7—C19—C18  | −178.38 (12) | C13—C14—N5—C20  | −152.26 (14) |
| Fe1—N7—C19—C20  | 2.98 (12)    | C8—C7—N3—C3     | 178.75 (16)  |
| Fe1—N7—C15—C16  | 179.36 (15)  | C8—C7—C6—C5     | −178.86 (18) |
| Br3—C21—C22—C23 | 178.85 (16)  | C12—C13—N6—C9   | −0.6 (2)     |
| Br3—C21—C26—C25 | −179.04 (18) | C12—C11—C10—C9  | −0.3 (2)     |
| N6—C13—C12—C11  | 1.32 (19)    | C14—C13—N6—C9   | 175.75 (14)  |
| N6—C13—C14—N5   | 29.28 (15)   | C14—C13—C12—C11 | −174.82 (17) |
| N6—C9—C10—C11   | 1.0 (2)      | C18—C19—N7—C15  | −0.7 (2)     |
| N3—C7—C8—N5     | 2.01 (16)    | C18—C17—C16—C15 | −1.0 (2)     |
| N3—C7—C6—C5     | 0.3 (2)      | C20—C19—N7—C15  | −179.34 (14) |
| N3—C3—C4—C5     | 0.3 (2)      | C20—C19—C18—C17 | 177.04 (18)  |
| N5—C8—C7—C6     | −178.81 (14) | C3—N3—C7—C6     | −0.42 (19)   |
| N5—C14—C13—C12  | −154.34 (13) | C3—C4—C5—C6     | −0.4 (2)     |
| N5—C20—C19—N7   | −30.07 (16)  | C21—C22—C23—C24 | 0.3 (2)      |
| N5—C20—C19—C18  | 151.30 (14)  | C21—C26—C25—C24 | 0.1 (3)      |
| N7—C19—C18—C17  | −1.5 (2)     | C22—C21—C26—C25 | 0.4 (3)      |
| N7—C15—C16—C17  | −1.2 (2)     | C22—C23—C24—C25 | 0.1 (3)      |
| C7—N3—C3—C4     | 0.14 (19)    | C23—C22—C21—C26 | −0.5 (2)     |
| C7—C8—N5—C14    | −117.26 (15) | C23—C24—C25—C26 | −0.3 (3)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate (br\_190K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 15.0867$  (4) Å

$b = 12.2943$  (2) Å

$c = 15.5270$  (3) Å

$\beta = 109.989$  (3)°

$V = 2706.46$  (11) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.520$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 39296 reflections

$\theta = 2.1$ – $31.1$ °

$\mu = 2.22$  mm<sup>−1</sup>

$T = 190$  K

Block, yellow

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.347$ ,  $T_{\max} = 1.000$

66000 measured reflections

6728 independent reflections

5883 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 28.3$ °,  $\theta_{\min} = 2.2$ °

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$  $wR(F^2) = 0.066$  $S = 1.03$ 

6728 reflections

387 parameters

30 restraints

55 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0195P)^2 + 1.7242P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.82 \text{ e } \text{\AA}^{-3}$ *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 | 0.352041 (15) | 0.317695 (17) | 0.697360 (14) | 0.02846 (6)                      | 0.658 (3) |
| Br3 | 0.35399 (5)   | 0.02592 (5)   | 0.44931 (4)   | 0.0626 (2)                       |           |
| S2  | 0.53408 (3)   | 0.00365 (4)   | 0.84203 (3)   | 0.04465 (11)                     |           |
| S1  | 0.25629 (11)  | 0.30409 (11)  | 0.96475 (8)   | 0.0729 (2)                       |           |
| N4  | 0.26106 (8)   | 0.45387 (10)  | 0.62094 (8)   | 0.0268 (2)                       |           |
| N3  | 0.39742 (8)   | 0.33338 (10)  | 0.57840 (8)   | 0.0295 (3)                       |           |
| N5  | 0.22328 (9)   | 0.23552 (10)  | 0.61030 (8)   | 0.0295 (3)                       |           |
| N6  | 0.43835 (9)   | 0.46516 (10)  | 0.74546 (8)   | 0.0308 (3)                       |           |
| N1  | 0.29988 (11)  | 0.33243 (12)  | 0.80678 (10)  | 0.0411 (3)                       |           |
| N2  | 0.43542 (11)  | 0.18509 (13)  | 0.75152 (10)  | 0.0470 (4)                       |           |
| C19 | 0.38932 (11)  | 0.55844 (12)  | 0.73004 (9)   | 0.0296 (3)                       |           |
| C7  | 0.35739 (10)  | 0.41349 (12)  | 0.51973 (9)   | 0.0280 (3)                       |           |
| C13 | 0.14927 (10)  | 0.30153 (12)  | 0.57306 (10)  | 0.0297 (3)                       |           |
| C14 | 0.16281 (10)  | 0.41882 (12)  | 0.60285 (11)  | 0.0331 (3)                       |           |
| H18 | 0.14470 (10)  | 0.42921 (12)  | 0.66596 (11)  | 0.0397 (4)*                      |           |
| H17 | 0.11445 (10)  | 0.47084 (12)  | 0.54841 (11)  | 0.0397 (4)*                      |           |
| C8  | 0.27892 (11)  | 0.47816 (13)  | 0.53484 (10)  | 0.0339 (3)                       |           |
| H5  | 0.21290 (11)  | 0.46308 (13)  | 0.47626 (10)  | 0.0407 (4)*                      |           |
| H6  | 0.29588 (11)  | 0.56604 (13)  | 0.53429 (10)  | 0.0407 (4)*                      |           |
| C20 | 0.28426 (11)  | 0.54744 (12)  | 0.68455 (11)  | 0.0329 (3)                       |           |
| H11 | 0.25537 (11)  | 0.62315 (12)  | 0.64615 (11)  | 0.0395 (4)*                      |           |
| H12 | 0.25098 (11)  | 0.53603 (12)  | 0.73764 (11)  | 0.0395 (4)*                      |           |
| C18 | 0.43201 (12)  | 0.65876 (13)  | 0.75774 (11)  | 0.0381 (3)                       |           |
| H10 | 0.38996 (12)  | 0.73423 (13)  | 0.74302 (11)  | 0.0457 (4)*                      |           |
| C9  | 0.21325 (12)  | 0.12897 (12)  | 0.58982 (10)  | 0.0344 (3)                       |           |
| H13 | 0.27422 (12)  | 0.07465 (12)  | 0.61994 (10)  | 0.0413 (4)*                      |           |
| C2  | 0.47619 (11)  | 0.10885 (14)  | 0.78869 (11)  | 0.0354 (3)                       |           |
| C12 | 0.06316 (11)  | 0.26329 (14)  | 0.51394 (11)  | 0.0373 (3)                       |           |
| H16 | 0.00384 (11)  | 0.31976 (14)  | 0.48363 (11)  | 0.0448 (4)*                      |           |
| C1  | 0.28190 (12)  | 0.32090 (13)  | 0.87321 (11)  | 0.0373 (3)                       |           |
| C6  | 0.38469 (12)  | 0.43626 (15)  | 0.44446 (11)  | 0.0389 (4)                       |           |
| H4  | 0.35067 (12)  | 0.50273 (15)  | 0.39695 (11)  | 0.0467 (4)*                      |           |
| C10 | 0.12925 (13)  | 0.08449 (14)  | 0.53228 (11)  | 0.0424 (4)                       |           |
| H14 | 0.12332 (13)  | −0.00351 (14) | 0.51760 (11)  | 0.0509 (5)*                      |           |
| C3  | 0.46565 (11)  | 0.27385 (14)  | 0.56416 (11)  | 0.0381 (3)                       |           |
| H1  | 0.49852 (11)  | 0.20800 (14)  | 0.61285 (11)  | 0.0457 (4)*                      |           |

|      |               |              |              |              |           |
|------|---------------|--------------|--------------|--------------|-----------|
| C15  | 0.53183 (11)  | 0.46924 (15) | 0.78786 (11) | 0.0391 (4)   |           |
| H7   | 0.57249 (11)  | 0.39284 (15) | 0.79842 (11) | 0.0469 (4)*  |           |
| C11  | 0.05302 (13)  | 0.15311 (15) | 0.49369 (12) | 0.0433 (4)   |           |
| H15  | −0.01470 (13) | 0.12057 (15) | 0.44757 (12) | 0.0519 (5)*  |           |
| C17  | 0.52832 (13)  | 0.66215 (15) | 0.80415 (12) | 0.0458 (4)   |           |
| H9   | 0.56386 (13)  | 0.74025 (15) | 0.82898 (12) | 0.0550 (5)*  |           |
| C16  | 0.57966 (13)  | 0.56583 (16) | 0.81907 (12) | 0.0466 (4)   |           |
| H8   | 0.65665 (13)  | 0.56617 (16) | 0.85488 (12) | 0.0559 (5)*  |           |
| C4   | 0.49662 (13)  | 0.29144 (16) | 0.49099 (13) | 0.0460 (4)   |           |
| H2   | 0.55289 (13)  | 0.24077 (16) | 0.48144 (13) | 0.0552 (5)*  |           |
| C21  | 0.2984 (5)    | 0.1247 (6)   | 0.3524 (5)   | 0.0392 (14)  | 0.658 (3) |
| C5   | 0.45507 (13)  | 0.37438 (17) | 0.43007 (12) | 0.0477 (4)   |           |
| H3   | 0.47776 (13)  | 0.39087 (17) | 0.37093 (12) | 0.0573 (5)*  |           |
| C22  | 0.2432 (6)    | 0.2071 (5)   | 0.3650 (4)   | 0.0454 (12)  | 0.658 (3) |
| H20  | 0.2316 (6)    | 0.2183 (5)   | 0.4309 (4)   | 0.0545 (14)* | 0.658 (3) |
| C26  | 0.3137 (5)    | 0.1076 (5)   | 0.2712 (5)   | 0.0481 (12)  | 0.658 (3) |
| H28  | 0.3583 (5)    | 0.0398 (5)   | 0.2636 (5)   | 0.0577 (15)* | 0.658 (3) |
| C23  | 0.2025 (5)    | 0.2760 (5)   | 0.2915 (4)   | 0.0522 (13)  | 0.658 (3) |
| H22  | 0.1568 (5)    | 0.3427 (5)   | 0.2990 (4)   | 0.0627 (16)* | 0.658 (3) |
| C24  | 0.2173 (4)    | 0.2637 (6)   | 0.2092 (5)   | 0.0555 (15)  | 0.658 (3) |
| H24  | 0.1864 (4)    | 0.3214 (6)   | 0.1525 (5)   | 0.0665 (18)* | 0.658 (3) |
| C25  | 0.2717 (4)    | 0.1777 (5)   | 0.1995 (4)   | 0.0559 (14)  | 0.658 (3) |
| H26  | 0.2819 (4)    | 0.1648 (5)   | 0.1332 (4)   | 0.0670 (16)* | 0.658 (3) |
| C23a | 0.2012 (8)    | 0.2658 (10)  | 0.3304 (10)  | 0.091 (6)    | 0.342 (3) |
| H21  | 0.1625 (8)    | 0.3192 (10)  | 0.3625 (10)  | 0.110 (7)*   | 0.342 (3) |
| Br3a | 0.3949 (3)    | 0.01665 (9)  | 0.4138 (3)   | 0.1455 (19)  | 0.342 (3) |
| C21a | 0.3081 (11)   | 0.1169 (11)  | 0.3413 (8)   | 0.052 (5)    | 0.342 (3) |
| C22a | 0.2594 (11)   | 0.1841 (13)  | 0.3816 (6)   | 0.075 (5)    | 0.342 (3) |
| H19  | 0.2668 (11)   | 0.1727 (13)  | 0.4542 (6)   | 0.090 (6)*   | 0.342 (3) |
| C26a | 0.2987 (9)    | 0.1313 (11)  | 0.2498 (8)   | 0.062 (4)    | 0.342 (3) |
| H27  | 0.3374 (9)    | 0.0779 (11)  | 0.2178 (8)   | 0.074 (5)*   | 0.342 (3) |
| C25a | 0.2405 (7)    | 0.2129 (11)  | 0.1986 (6)   | 0.079 (6)    | 0.342 (3) |
| H25  | 0.2331 (7)    | 0.2243 (11)  | 0.1260 (6)   | 0.095 (7)*   | 0.342 (3) |
| C24a | 0.1918 (6)    | 0.2802 (7)   | 0.2389 (10)  | 0.075 (5)    | 0.342 (3) |
| H23  | 0.1456 (6)    | 0.3449 (7)   | 0.1983 (10)  | 0.090 (6)*   | 0.342 (3) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| Fe1 | 0.03025 (11) | 0.02973 (11) | 0.02521 (10) | 0.00052 (8)   | 0.00921 (8)  | −0.00133 (8) |
| Br3 | 0.0862 (3)   | 0.0429 (2)   | 0.0551 (2)   | −0.00122 (18) | 0.0195 (2)   | 0.00364 (16) |
| S2  | 0.0363 (2)   | 0.0435 (2)   | 0.0580 (3)   | 0.00730 (18)  | 0.02112 (19) | 0.0116 (2)   |
| S1  | 0.1172 (5)   | 0.0704 (4)   | 0.0476 (3)   | −0.0452 (4)   | 0.0495 (3)   | −0.0230 (3)  |
| N4  | 0.0270 (6)   | 0.0259 (6)   | 0.0275 (6)   | 0.0030 (5)    | 0.0092 (5)   | −0.0017 (5)  |
| N3  | 0.0288 (6)   | 0.0338 (6)   | 0.0266 (6)   | 0.0032 (5)    | 0.0105 (5)   | −0.0001 (5)  |
| N5  | 0.0332 (6)   | 0.0287 (6)   | 0.0263 (6)   | 0.0020 (5)    | 0.0096 (5)   | −0.0008 (5)  |
| N6  | 0.0320 (6)   | 0.0305 (6)   | 0.0265 (6)   | 0.0003 (5)    | 0.0056 (5)   | 0.0008 (5)   |
| N1  | 0.0534 (8)   | 0.0392 (7)   | 0.0357 (7)   | −0.0067 (6)   | 0.0217 (6)   | −0.0037 (6)  |

|      |             |             |            |              |            |              |
|------|-------------|-------------|------------|--------------|------------|--------------|
| N2   | 0.0462 (8)  | 0.0487 (9)  | 0.0426 (8) | 0.0097 (7)   | 0.0107 (7) | 0.0083 (7)   |
| C19  | 0.0342 (7)  | 0.0269 (7)  | 0.0268 (7) | −0.0013 (6)  | 0.0091 (6) | −0.0027 (5)  |
| C7   | 0.0288 (7)  | 0.0316 (7)  | 0.0235 (6) | −0.0017 (6)  | 0.0087 (5) | −0.0010 (5)  |
| C13  | 0.0289 (7)  | 0.0300 (7)  | 0.0297 (7) | −0.0007 (6)  | 0.0092 (6) | −0.0007 (6)  |
| C14  | 0.0276 (7)  | 0.0306 (7)  | 0.0402 (8) | 0.0037 (6)   | 0.0105 (6) | −0.0029 (6)  |
| C8   | 0.0362 (8)  | 0.0359 (8)  | 0.0294 (7) | 0.0076 (6)   | 0.0108 (6) | 0.0049 (6)   |
| C20  | 0.0346 (8)  | 0.0273 (7)  | 0.0365 (8) | 0.0033 (6)   | 0.0116 (6) | −0.0049 (6)  |
| C18  | 0.0434 (9)  | 0.0319 (8)  | 0.0376 (8) | −0.0059 (7)  | 0.0120 (7) | −0.0077 (6)  |
| C9   | 0.0459 (9)  | 0.0278 (7)  | 0.0302 (7) | 0.0018 (6)   | 0.0138 (7) | 0.0010 (6)   |
| C2   | 0.0317 (7)  | 0.0407 (9)  | 0.0343 (8) | 0.0037 (7)   | 0.0118 (6) | 0.0049 (7)   |
| C12  | 0.0318 (8)  | 0.0385 (9)  | 0.0372 (8) | −0.0049 (7)  | 0.0061 (6) | 0.0001 (7)   |
| C1   | 0.0489 (9)  | 0.0342 (8)  | 0.0318 (8) | −0.0110 (7)  | 0.0174 (7) | −0.0064 (6)  |
| C6   | 0.0424 (9)  | 0.0469 (9)  | 0.0306 (8) | −0.0026 (7)  | 0.0167 (7) | 0.0040 (7)   |
| C10  | 0.0567 (11) | 0.0314 (8)  | 0.0382 (8) | −0.0096 (7)  | 0.0151 (8) | −0.0040 (7)  |
| C3   | 0.0330 (8)  | 0.0441 (9)  | 0.0384 (8) | 0.0078 (7)   | 0.0139 (7) | −0.0014 (7)  |
| C15  | 0.0345 (8)  | 0.0429 (9)  | 0.0330 (8) | 0.0012 (7)   | 0.0027 (6) | 0.0031 (7)   |
| C11  | 0.0429 (9)  | 0.0420 (9)  | 0.0400 (9) | −0.0129 (8)  | 0.0079 (7) | −0.0029 (7)  |
| C17  | 0.0470 (10) | 0.0433 (10) | 0.0428 (9) | −0.0141 (8)  | 0.0099 (8) | −0.0105 (8)  |
| C16  | 0.0369 (9)  | 0.0548 (11) | 0.0397 (9) | −0.0081 (8)  | 0.0024 (7) | −0.0029 (8)  |
| C4   | 0.0395 (9)  | 0.0588 (11) | 0.0467 (9) | 0.0047 (8)   | 0.0236 (8) | −0.0078 (8)  |
| C21  | 0.042 (2)   | 0.035 (3)   | 0.045 (3)  | −0.0074 (18) | 0.021 (2)  | −0.0099 (19) |
| C5   | 0.0462 (9)  | 0.0665 (12) | 0.0398 (9) | −0.0038 (9)  | 0.0268 (8) | −0.0032 (9)  |
| C22  | 0.050 (3)   | 0.0392 (18) | 0.056 (2)  | −0.0067 (19) | 0.030 (2)  | −0.0117 (19) |
| C26  | 0.061 (3)   | 0.046 (2)   | 0.049 (3)  | −0.001 (2)   | 0.033 (3)  | −0.005 (2)   |
| C23  | 0.036 (2)   | 0.042 (2)   | 0.077 (4)  | 0.0046 (17)  | 0.019 (3)  | 0.002 (3)    |
| C24  | 0.041 (3)   | 0.056 (4)   | 0.061 (3)  | −0.010 (3)   | 0.007 (2)  | 0.009 (3)    |
| C25  | 0.068 (4)   | 0.055 (3)   | 0.044 (3)  | −0.009 (3)   | 0.019 (3)  | 0.004 (2)    |
| C23a | 0.031 (4)   | 0.100 (11)  | 0.136 (14) | −0.011 (5)   | 0.021 (7)  | −0.080 (10)  |
| Br3a | 0.169 (2)   | 0.0481 (5)  | 0.131 (2)  | 0.0105 (7)   | −0.063 (2) | 0.0103 (7)   |
| C21a | 0.069 (8)   | 0.033 (5)   | 0.046 (7)  | 0.001 (4)    | 0.012 (5)  | −0.005 (4)   |
| C22a | 0.072 (9)   | 0.111 (13)  | 0.057 (6)  | −0.044 (8)   | 0.041 (6)  | −0.042 (7)   |
| C26a | 0.056 (5)   | 0.089 (10)  | 0.043 (6)  | 0.026 (5)    | 0.019 (5)  | −0.004 (6)   |
| C25a | 0.067 (10)  | 0.093 (14)  | 0.068 (9)  | 0.008 (7)    | 0.011 (6)  | 0.041 (10)   |
| C24a | 0.036 (5)   | 0.048 (5)   | 0.115 (14) | −0.002 (4)   | −0.007 (7) | 0.022 (9)    |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N4  | 2.2319 (12) | C6—H4   | 1.1030    |
| Fe1—N3  | 2.1869 (12) | C6—C5   | 1.385 (2) |
| Fe1—N5  | 2.1969 (13) | C10—H14 | 1.1030    |
| Fe1—N6  | 2.2080 (13) | C10—C11 | 1.386 (3) |
| Fe1—N1  | 2.1101 (14) | C3—H1   | 1.1030    |
| Fe1—N2  | 2.0553 (15) | C3—C4   | 1.385 (2) |
| Br3—C21 | 1.893 (5)   | C15—H7  | 1.1030    |
| S2—C2   | 1.6208 (17) | C15—C16 | 1.388 (2) |
| S1—C1   | 1.6087 (19) | C11—H15 | 1.1030    |
| N4—C14  | 1.4754 (18) | C17—H9  | 1.1030    |
| N4—C8   | 1.4818 (18) | C17—C16 | 1.390 (3) |



|            |             |             |             |
|------------|-------------|-------------|-------------|
| N4—C20     | 1.4781 (18) | C16—H8      | 1.1030      |
| N3—C7      | 1.3373 (18) | C4—H2       | 1.1030      |
| N3—C3      | 1.3414 (19) | C4—C5       | 1.386 (3)   |
| N5—C13     | 1.3409 (19) | C21—C22     | 1.367 (7)   |
| N5—C9      | 1.3443 (19) | C21—C26     | 1.373 (7)   |
| N6—C19     | 1.3412 (19) | C5—H3       | 1.1030      |
| N6—C15     | 1.338 (2)   | C22—H20     | 1.1030      |
| N1—C1      | 1.162 (2)   | C22—C23     | 1.385 (8)   |
| N2—C2      | 1.161 (2)   | C26—H28     | 1.1030      |
| C19—C20    | 1.505 (2)   | C26—C25     | 1.380 (7)   |
| C19—C18    | 1.390 (2)   | C23—H22     | 1.1030      |
| C7—C8      | 1.509 (2)   | C23—C24     | 1.378 (8)   |
| C7—C6      | 1.394 (2)   | C24—H24     | 1.1030      |
| C13—C14    | 1.507 (2)   | C24—C25     | 1.378 (7)   |
| C13—C12    | 1.391 (2)   | C25—H26     | 1.1030      |
| C14—H18    | 1.1110      | C23a—H21    | 1.1030      |
| C14—H17    | 1.1110      | C23a—C22a   | 1.3900      |
| C8—H5      | 1.1110      | C23a—C24a   | 1.3900      |
| C8—H6      | 1.1110      | Br3a—C21a   | 1.869 (8)   |
| C20—H11    | 1.1110      | C21a—C22a   | 1.3900      |
| C20—H12    | 1.1110      | C21a—C26a   | 1.3900      |
| C18—H10    | 1.1030      | C22a—H19    | 1.1030      |
| C18—C17    | 1.384 (2)   | C26a—H27    | 1.1030      |
| C9—H13     | 1.1030      | C26a—C25a   | 1.3900      |
| C9—C10     | 1.389 (2)   | C25a—H25    | 1.1030      |
| C12—H16    | 1.1030      | C25a—C24a   | 1.3900      |
| C12—C11    | 1.387 (2)   | C24a—H23    | 1.1030      |
|            |             |             |             |
| N3—Fe1—N4  | 78.35 (4)   | C11—C12—C13 | 119.03 (16) |
| N5—Fe1—N4  | 76.35 (4)   | C11—C12—H16 | 120.48 (10) |
| N5—Fe1—N3  | 89.26 (4)   | N1—C1—S1    | 179.47 (15) |
| N6—Fe1—N4  | 75.42 (4)   | H4—C6—C7    | 120.41 (10) |
| N6—Fe1—N3  | 83.95 (5)   | C5—C6—C7    | 119.17 (16) |
| N6—Fe1—N5  | 151.75 (5)  | C5—C6—H4    | 120.41 (10) |
| N1—Fe1—N4  | 91.88 (5)   | H14—C10—C9  | 120.72 (10) |
| N1—Fe1—N3  | 169.45 (5)  | C11—C10—C9  | 118.56 (16) |
| N1—Fe1—N5  | 92.30 (5)   | C11—C10—H14 | 120.72 (10) |
| N1—Fe1—N6  | 89.79 (5)   | H1—C3—N3    | 118.56 (9)  |
| N2—Fe1—N4  | 172.61 (5)  | C4—C3—N3    | 122.89 (16) |
| N2—Fe1—N3  | 95.22 (5)   | C4—C3—H1    | 118.56 (11) |
| N2—Fe1—N5  | 100.13 (6)  | H7—C15—N6   | 118.71 (10) |
| N2—Fe1—N6  | 107.77 (5)  | C16—C15—N6  | 122.59 (16) |
| N2—Fe1—N1  | 94.77 (6)   | C16—C15—H7  | 118.71 (10) |
| C14—N4—Fe1 | 106.10 (9)  | C10—C11—C12 | 119.03 (16) |
| C8—N4—Fe1  | 111.24 (8)  | H15—C11—C12 | 120.49 (10) |
| C8—N4—C14  | 111.58 (12) | H15—C11—C10 | 120.49 (10) |
| C20—N4—Fe1 | 105.45 (8)  | H9—C17—C18  | 120.43 (10) |
| C20—N4—C14 | 110.34 (11) | C16—C17—C18 | 119.13 (16) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| C20—N4—C8   | 111.81 (12) | C16—C17—H9     | 120.43 (10) |
| C7—N3—Fe1   | 116.19 (9)  | C17—C16—C15    | 118.49 (16) |
| C3—N3—Fe1   | 124.79 (11) | H8—C16—C15     | 120.76 (10) |
| C3—N3—C7    | 118.87 (13) | H8—C16—C17     | 120.76 (10) |
| C13—N5—Fe1  | 114.69 (10) | H2—C4—C3       | 120.86 (11) |
| C9—N5—Fe1   | 126.72 (11) | C5—C4—C3       | 118.28 (16) |
| C9—N5—C13   | 118.56 (13) | C5—C4—H2       | 120.86 (10) |
| C19—N6—Fe1  | 114.59 (10) | C22—C21—Br3    | 119.2 (5)   |
| C15—N6—Fe1  | 126.71 (11) | C26—C21—Br3    | 117.6 (5)   |
| C15—N6—C19  | 118.69 (14) | C26—C21—C22    | 123.2 (5)   |
| C1—N1—Fe1   | 165.74 (14) | C4—C5—C6       | 119.13 (15) |
| C2—N2—Fe1   | 171.95 (14) | H3—C5—C6       | 120.44 (10) |
| C20—C19—N6  | 115.80 (13) | H3—C5—C4       | 120.44 (10) |
| C18—C19—N6  | 122.32 (14) | H20—C22—C21    | 121.5 (3)   |
| C18—C19—C20 | 121.83 (14) | C23—C22—C21    | 117.0 (4)   |
| C8—C7—N3    | 118.54 (12) | C23—C22—H20    | 121.5 (3)   |
| C6—C7—N3    | 121.66 (14) | H28—C26—C21    | 121.0 (3)   |
| C6—C7—C8    | 119.78 (14) | C25—C26—C21    | 118.0 (5)   |
| C14—C13—N5  | 116.19 (13) | C25—C26—H28    | 121.0 (3)   |
| C12—C13—N5  | 122.14 (14) | H22—C23—C22    | 119.0 (3)   |
| C12—C13—C14 | 121.60 (14) | C24—C23—C22    | 122.0 (5)   |
| C13—C14—N4  | 111.10 (12) | C24—C23—H22    | 119.0 (4)   |
| H18—C14—N4  | 109.42 (8)  | H24—C24—C23    | 120.7 (4)   |
| H18—C14—C13 | 109.42 (8)  | C25—C24—C23    | 118.5 (7)   |
| H17—C14—N4  | 109.42 (8)  | C25—C24—H24    | 120.7 (5)   |
| H17—C14—C13 | 109.42 (8)  | C24—C25—C26    | 121.1 (7)   |
| H17—C14—H18 | 108.0       | H26—C25—C26    | 119.4 (3)   |
| C7—C8—N4    | 115.36 (12) | H26—C25—C24    | 119.4 (5)   |
| H5—C8—N4    | 108.44 (8)  | C22a—C23a—H21  | 120.0 (5)   |
| H5—C8—C7    | 108.44 (8)  | C24a—C23a—H21  | 120.0 (5)   |
| H6—C8—N4    | 108.44 (8)  | C24a—C23a—C22a | 120.0       |
| H6—C8—C7    | 108.44 (8)  | C22a—C21a—Br3a | 119.4       |
| H6—C8—H5    | 107.5       | C26a—C21a—Br3a | 120.4       |
| C19—C20—N4  | 110.76 (12) | C26a—C21a—C22a | 120.0       |
| H11—C20—N4  | 109.49 (8)  | C21a—C22a—C23a | 120.0       |
| H11—C20—C19 | 109.49 (8)  | H19—C22a—C23a  | 120.0 (5)   |
| H12—C20—N4  | 109.49 (8)  | H19—C22a—C21a  | 120.0 (5)   |
| H12—C20—C19 | 109.49 (8)  | H27—C26a—C21a  | 120.0 (5)   |
| H12—C20—H11 | 108.1       | C25a—C26a—C21a | 120.0       |
| H10—C18—C19 | 120.63 (9)  | C25a—C26a—H27  | 120.0 (5)   |
| C17—C18—C19 | 118.74 (16) | H25—C25a—C26a  | 120.0 (5)   |
| C17—C18—H10 | 120.63 (10) | C24a—C25a—C26a | 120.0       |
| H13—C9—N5   | 118.66 (9)  | C24a—C25a—H25  | 120.0 (5)   |
| C10—C9—N5   | 122.68 (15) | C25a—C24a—C23a | 120.0       |
| C10—C9—H13  | 118.66 (10) | H23—C24a—C23a  | 120.0 (5)   |
| N2—C2—S2    | 179.06 (16) | H23—C24a—C25a  | 120.0 (5)   |
| H16—C12—C13 | 120.48 (10) |                |             |

|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| Fe1—N4—C14—C13  | −42.56 (9)   | C7—C8—N4—C20        | 113.57 (13)  |
| Fe1—N4—C8—C7    | −4.04 (9)    | C7—C6—C5—C4         | −0.07 (19)   |
| Fe1—N4—C20—C19  | 45.27 (9)    | C13—N5—C9—C10       | −0.49 (16)   |
| Fe1—N3—C7—C8    | −5.85 (11)   | C13—C14—N4—C8       | 78.76 (14)   |
| Fe1—N3—C7—C6    | 175.85 (10)  | C13—C14—N4—C20      | −156.31 (12) |
| Fe1—N3—C3—C4    | −175.47 (13) | C13—C12—C11—C10     | −0.60 (18)   |
| Fe1—N5—C13—C14  | −5.23 (11)   | C14—C13—N5—C9       | 176.47 (13)  |
| Fe1—N5—C13—C12  | 178.01 (10)  | C14—C13—C12—C11     | −175.76 (15) |
| Fe1—N5—C9—C10   | −178.56 (12) | C8—C7—N3—C3         | 178.42 (14)  |
| Fe1—N6—C19—C20  | 1.12 (11)    | C8—C7—C6—C5         | −178.30 (15) |
| Fe1—N6—C19—C18  | 178.46 (10)  | C20—C19—N6—C15      | −178.31 (13) |
| Fe1—N6—C15—C16  | −177.27 (13) | C20—C19—C18—C17     | 176.12 (15)  |
| Br3—C21—C22—C23 | −178.7 (4)   | C18—C19—N6—C15      | −0.97 (18)   |
| Br3—C21—C26—C25 | 178.6 (3)    | C18—C17—C16—C15     | −1.0 (2)     |
| N4—C14—C13—N5   | 33.48 (14)   | C9—N5—C13—C12       | −0.29 (16)   |
| N4—C14—C13—C12  | −149.74 (12) | C9—C10—C11—C12      | −0.13 (19)   |
| N4—C8—C7—N3     | 6.70 (16)    | C6—C7—N3—C3         | 0.11 (18)    |
| N4—C8—C7—C6     | −174.96 (13) | C6—C5—C4—C3         | 0.1 (2)      |
| N4—C20—C19—N6   | −32.47 (14)  | C21—C22—C23—C24     | −0.8 (7)     |
| N4—C20—C19—C18  | 150.18 (12)  | C21—C26—C25—C24     | 1.2 (6)      |
| N3—C7—C6—C5     | −0.01 (17)   | C22—C21—C26—C25     | 0.4 (7)      |
| N3—C3—C4—C5     | 0.1 (2)      | C22—C23—C24—C25     | 2.3 (7)      |
| N5—C13—C12—C11  | 0.84 (17)    | C26—C21—C22—C23     | −0.6 (7)     |
| N5—C9—C10—C11   | 0.70 (18)    | C26—C25—C24—C23     | −2.5 (7)     |
| N6—C19—C18—C17  | −1.06 (17)   | C23a—C22a—C21a—Br3a | 174.1 (3)    |
| N6—C15—C16—C17  | −1.11 (19)   | C23a—C22a—C21a—C26a | 0.0 (11)     |
| C19—N6—C15—C16  | 2.08 (16)    | C23a—C24a—C25a—C26a | −0.0 (11)    |
| C19—C20—N4—C14  | 159.43 (12)  | Br3a—C21a—C26a—C25a | −174.0 (3)   |
| C19—C20—N4—C8   | −75.76 (13)  | C21a—C22a—C23a—C24a | 0.0 (11)     |
| C19—C18—C17—C16 | 1.99 (19)    | C21a—C26a—C25a—C24a | 0.0 (10)     |
| C7—N3—C3—C4     | −0.13 (17)   | C22a—C23a—C24a—C25a | −0.0 (10)    |
| C7—C8—N4—C14    | −122.31 (13) | C22a—C21a—C26a—C25a | −0.0 (11)    |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate**  
(br\_230K)

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 15.1940$  (4) Å

$b = 12.2919$  (3) Å

$c = 15.5626$  (4) Å

$\beta = 110.062$  (3)°

$V = 2730.16$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.507$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 35116 reflections

$\theta = 2.1$ – $30.6^\circ$

$\mu = 2.20$  mm<sup>−1</sup>

$T = 230$  K

Block, yellow

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector  
Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source  
Mirror monochromator  
Detector resolution: 10.0000 pixels mm<sup>-1</sup>  
 $\omega$  scans  
Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.338$ ,  $T_{\max} = 1.000$   
68518 measured reflections  
6781 independent reflections  
5758 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -20 \rightarrow 20$   
 $k = -16 \rightarrow 16$   
 $l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.081$   
 $S = 1.03$   
6781 reflections  
358 parameters  
264 restraints

60 constraints  
Primary atom site location: dual  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 1.4297P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.90 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.73 \text{ e } \text{\AA}^{-3}$

*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  | 0.648702 (15) | 0.317621 (18) | 0.301956 (14) | 0.03324 (7)                      | 0.696 (4) |
| Br3  | 0.64663 (17)  | 0.02690 (10)  | 0.55410 (12)  | 0.0874 (5)                       |           |
| S2   | 0.46632 (3)   | 0.00423 (4)   | 0.15865 (4)   | 0.05309 (13)                     |           |
| S1   | 0.74381 (6)   | 0.30545 (6)   | 0.03460 (4)   | 0.0909 (3)                       |           |
| N3   | 0.60360 (9)   | 0.33336 (11)  | 0.42135 (9)   | 0.0358 (3)                       |           |
| N4   | 0.73908 (9)   | 0.45463 (10)  | 0.37896 (8)   | 0.0328 (3)                       |           |
| N5   | 0.77803 (9)   | 0.23628 (11)  | 0.38938 (8)   | 0.0352 (3)                       |           |
| N6   | 0.56240 (9)   | 0.46565 (11)  | 0.25418 (9)   | 0.0371 (3)                       |           |
| N1   | 0.70032 (12)  | 0.33302 (13)  | 0.19239 (10)  | 0.0486 (4)                       |           |
| N2   | 0.56584 (12)  | 0.18515 (14)  | 0.24842 (11)  | 0.0547 (4)                       |           |
| C7   | 0.64358 (10)  | 0.41331 (13)  | 0.47991 (10)  | 0.0344 (3)                       |           |
| C19  | 0.61068 (11)  | 0.55917 (13)  | 0.27061 (10)  | 0.0360 (3)                       |           |
| C13  | 0.85114 (11)  | 0.30304 (13)  | 0.42598 (11)  | 0.0368 (3)                       |           |
| C14  | 0.83665 (11)  | 0.42036 (13)  | 0.39623 (12)  | 0.0402 (4)                       |           |
| H12  | 0.85370 (11)  | 0.43100 (13)  | 0.33346 (12)  | 0.0483 (4)*                      |           |
| H11  | 0.88435 (11)  | 0.47220 (13)  | 0.44982 (12)  | 0.0483 (4)*                      |           |
| C8   | 0.72215 (12)  | 0.47735 (14)  | 0.46534 (11)  | 0.0414 (4)                       |           |
| H5   | 0.78717 (12)  | 0.46058 (14)  | 0.52282 (11)  | 0.0496 (4)*                      |           |
| H6   | 0.70666 (12)  | 0.56461 (14)  | 0.46739 (11)  | 0.0496 (4)*                      |           |
| C9   | 0.78883 (13)  | 0.12990 (13)  | 0.41003 (11)  | 0.0408 (4)                       |           |
| H7   | 0.72920 (13)  | 0.07542 (13)  | 0.38052 (11)  | 0.0489 (4)*                      |           |
| C2   | 0.52474 (11)  | 0.10885 (14)  | 0.21163 (12)  | 0.0411 (4)                       |           |
| C20  | 0.71508 (11)  | 0.54874 (13)  | 0.31622 (12)  | 0.0405 (3)                       |           |
| H00e | 0.74298 (11)  | 0.62359 (13)  | 0.35481 (12)  | 0.0486 (4)*                      |           |
| H00f | 0.74795 (11)  | 0.53886 (13)  | 0.26371 (12)  | 0.0486 (4)*                      |           |
| C12  | 0.93717 (12)  | 0.26575 (15)  | 0.48465 (12)  | 0.0455 (4)                       |           |
| H10  | 0.99527 (12)  | 0.32227 (15)  | 0.51412 (12)  | 0.0546 (5)*                      |           |

|      |              |               |              |             |           |
|------|--------------|---------------|--------------|-------------|-----------|
| C3   | 0.53542 (12) | 0.27430 (16)  | 0.43557 (12) | 0.0460 (4)  |           |
| H1   | 0.50277 (12) | 0.20909 (16)  | 0.38744 (12) | 0.0552 (5)* |           |
| C18  | 0.56798 (13) | 0.65929 (14)  | 0.24395 (12) | 0.0457 (4)  |           |
| H16  | 0.60910 (13) | 0.73427 (14)  | 0.25955 (12) | 0.0548 (5)* |           |
| C10  | 0.87263 (14) | 0.08649 (15)  | 0.46729 (12) | 0.0499 (4)  |           |
| H8   | 0.87904 (14) | −0.00062 (15) | 0.48225 (12) | 0.0599 (5)* |           |
| C1   | 0.71835 (13) | 0.32239 (14)  | 0.12640 (12) | 0.0448 (4)  |           |
| C6   | 0.61640 (13) | 0.43642 (17)  | 0.55474 (12) | 0.0481 (4)  |           |
| H4   | 0.65003 (13) | 0.50227 (17)  | 0.60173 (12) | 0.0577 (5)* |           |
| C15  | 0.46961 (12) | 0.46958 (16)  | 0.21177 (12) | 0.0472 (4)  |           |
| H13  | 0.42980 (12) | 0.39366 (16)  | 0.20055 (12) | 0.0567 (5)* |           |
| C11  | 0.94808 (14) | 0.15553 (16)  | 0.50520 (13) | 0.0515 (4)  |           |
| H9   | 1.01512 (14) | 0.12379 (16)  | 0.55056 (13) | 0.0618 (5)* |           |
| C17  | 0.47235 (14) | 0.66262 (17)  | 0.19723 (14) | 0.0551 (5)  |           |
| H15  | 0.43719 (14) | 0.74007 (17)  | 0.17315 (14) | 0.0661 (6)* |           |
| C16  | 0.42180 (14) | 0.56623 (18)  | 0.18142 (13) | 0.0556 (5)  |           |
| H14  | 0.34602 (14) | 0.56639 (18)  | 0.14587 (13) | 0.0667 (6)* |           |
| C4   | 0.50480 (14) | 0.29228 (19)  | 0.50816 (14) | 0.0573 (5)  |           |
| H2   | 0.44917 (14) | 0.24244 (19)  | 0.51746 (14) | 0.0688 (6)* |           |
| C21  | 0.7035 (4)   | 0.1253 (4)    | 0.6479 (4)   | 0.0671 (6)  | 0.696 (4) |
| C5   | 0.54622 (15) | 0.3750 (2)    | 0.56891 (14) | 0.0594 (5)  |           |
| H3   | 0.52385 (15) | 0.3915 (2)    | 0.62730 (14) | 0.0713 (6)* |           |
| C26  | 0.6863 (3)   | 0.1092 (4)    | 0.7292 (4)   | 0.0671 (6)  | 0.696 (4) |
| H26  | 0.6406 (3)   | 0.0435 (4)    | 0.7360 (4)   | 0.0805 (7)* | 0.696 (4) |
| C22  | 0.7616 (4)   | 0.2082 (4)    | 0.6389 (3)   | 0.0671 (6)  | 0.696 (4) |
| H18  | 0.7767 (4)   | 0.2181 (4)    | 0.5755 (3)   | 0.0805 (7)* | 0.696 (4) |
| C23  | 0.7996 (3)   | 0.2772 (4)    | 0.7109 (4)   | 0.0671 (6)  | 0.696 (4) |
| H20  | 0.8434 (3)   | 0.3448 (4)    | 0.7040 (4)   | 0.0805 (7)* | 0.696 (4) |
| C24  | 0.7840 (3)   | 0.2633 (4)    | 0.7901 (3)   | 0.0671 (6)  | 0.696 (4) |
| H22  | 0.8155 (3)   | 0.3197 (4)    | 0.8465 (3)   | 0.0805 (7)* | 0.696 (4) |
| C25  | 0.7289 (3)   | 0.1793 (4)    | 0.8014 (3)   | 0.0671 (6)  | 0.696 (4) |
| H24  | 0.7190 (3)   | 0.1678 (4)    | 0.8671 (3)   | 0.0805 (7)* | 0.696 (4) |
| C22a | 0.7401 (4)   | 0.1855 (5)    | 0.6150 (3)   | 0.0509 (18) | 0.304 (4) |
| H17  | 0.7320 (4)   | 0.1743 (5)    | 0.5430 (3)   | 0.061 (2)*  | 0.304 (4) |
| Br3a | 0.5946 (3)   | 0.01489 (12)  | 0.5938 (3)   | 0.1274 (13) | 0.304 (4) |
| C21a | 0.6916 (5)   | 0.1188 (5)    | 0.6555 (3)   | 0.0439 (18) | 0.304 (4) |
| C26a | 0.7018 (4)   | 0.1331 (6)    | 0.7470 (3)   | 0.057 (2)   | 0.304 (4) |
| H25  | 0.6636 (4)   | 0.0806 (6)    | 0.7788 (3)   | 0.068 (2)*  | 0.304 (4) |
| C25a | 0.7605 (4)   | 0.2141 (6)    | 0.7980 (3)   | 0.066 (2)   | 0.304 (4) |
| H23  | 0.7686 (4)   | 0.2253 (6)    | 0.8700 (3)   | 0.079 (2)*  | 0.304 (4) |
| C23a | 0.7988 (3)   | 0.2665 (5)    | 0.6660 (5)   | 0.065 (2)   | 0.304 (4) |
| H19  | 0.8370 (3)   | 0.3189 (5)    | 0.6342 (5)   | 0.078 (2)*  | 0.304 (4) |
| C24a | 0.8091 (4)   | 0.2808 (4)    | 0.7575 (5)   | 0.0579 (19) | 0.304 (4) |
| H21  | 0.8552 (4)   | 0.3444 (4)    | 0.7977 (5)   | 0.069 (2)*  | 0.304 (4) |

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

|      | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------|--------------|--------------|--------------|--------------|--------------|--------------|
| Fe1  | 0.03626 (12) | 0.03280 (12) | 0.02954 (11) | −0.00229 (9) | 0.00985 (9)  | −0.00080 (8) |
| Br3  | 0.1201 (7)   | 0.0560 (3)   | 0.0754 (4)   | 0.0034 (3)   | 0.0199 (5)   | −0.0066 (2)  |
| S2   | 0.0446 (2)   | 0.0484 (3)   | 0.0703 (3)   | −0.0121 (2)  | 0.0250 (2)   | −0.0173 (2)  |
| S1   | 0.1482 (7)   | 0.0896 (5)   | 0.0557 (3)   | 0.0587 (5)   | 0.0617 (4)   | 0.0282 (3)   |
| N3   | 0.0348 (6)   | 0.0403 (7)   | 0.0331 (6)   | −0.0043 (5)  | 0.0126 (5)   | 0.0008 (5)   |
| N4   | 0.0328 (6)   | 0.0303 (6)   | 0.0344 (6)   | −0.0038 (5)  | 0.0105 (5)   | 0.0021 (5)   |
| N5   | 0.0385 (7)   | 0.0341 (7)   | 0.0321 (6)   | −0.0007 (5)  | 0.0109 (5)   | 0.0009 (5)   |
| N6   | 0.0383 (7)   | 0.0363 (7)   | 0.0323 (6)   | 0.0004 (5)   | 0.0066 (5)   | −0.0007 (5)  |
| N1   | 0.0653 (9)   | 0.0483 (8)   | 0.0380 (7)   | 0.0073 (7)   | 0.0252 (7)   | 0.0031 (6)   |
| N2   | 0.0568 (9)   | 0.0502 (9)   | 0.0526 (9)   | −0.0153 (7)  | 0.0128 (7)   | −0.0129 (7)  |
| C7   | 0.0356 (7)   | 0.0382 (8)   | 0.0292 (7)   | 0.0022 (6)   | 0.0107 (6)   | 0.0014 (6)   |
| C19  | 0.0413 (8)   | 0.0329 (8)   | 0.0327 (7)   | 0.0014 (6)   | 0.0113 (6)   | 0.0031 (6)   |
| C13  | 0.0349 (8)   | 0.0374 (8)   | 0.0368 (7)   | 0.0013 (6)   | 0.0108 (6)   | 0.0005 (6)   |
| C14  | 0.0337 (7)   | 0.0369 (8)   | 0.0488 (9)   | −0.0043 (6)  | 0.0126 (7)   | 0.0035 (7)   |
| C8   | 0.0442 (9)   | 0.0424 (9)   | 0.0366 (8)   | −0.0091 (7)  | 0.0128 (7)   | −0.0061 (7)  |
| C9   | 0.0523 (9)   | 0.0330 (8)   | 0.0360 (8)   | 0.0000 (7)   | 0.0139 (7)   | −0.0001 (6)  |
| C2   | 0.0376 (8)   | 0.0429 (9)   | 0.0428 (8)   | −0.0051 (7)  | 0.0138 (7)   | −0.0061 (7)  |
| C20  | 0.0415 (8)   | 0.0328 (8)   | 0.0460 (9)   | −0.0040 (6)  | 0.0136 (7)   | 0.0069 (7)   |
| C12  | 0.0368 (8)   | 0.0474 (10)  | 0.0468 (9)   | 0.0062 (7)   | 0.0072 (7)   | −0.0011 (8)  |
| C3   | 0.0402 (8)   | 0.0527 (10)  | 0.0474 (9)   | −0.0092 (7)  | 0.0181 (7)   | 0.0043 (8)   |
| C18  | 0.0528 (10)  | 0.0370 (9)   | 0.0454 (9)   | 0.0075 (7)   | 0.0145 (8)   | 0.0074 (7)   |
| C10  | 0.0635 (11)  | 0.0383 (9)   | 0.0451 (9)   | 0.0124 (8)   | 0.0152 (8)   | 0.0039 (7)   |
| C1   | 0.0602 (10)  | 0.0414 (9)   | 0.0355 (8)   | 0.0146 (8)   | 0.0200 (7)   | 0.0081 (7)   |
| C6   | 0.0526 (10)  | 0.0588 (11)  | 0.0367 (8)   | 0.0032 (8)   | 0.0201 (7)   | −0.0045 (8)  |
| C15  | 0.0414 (9)   | 0.0501 (10)  | 0.0412 (8)   | 0.0000 (7)   | 0.0026 (7)   | −0.0027 (7)  |
| C11  | 0.0498 (10)  | 0.0501 (10)  | 0.0480 (10)  | 0.0154 (8)   | 0.0081 (8)   | 0.0027 (8)   |
| C17  | 0.0564 (11)  | 0.0518 (11)  | 0.0522 (10)  | 0.0172 (9)   | 0.0124 (9)   | 0.0116 (8)   |
| C16  | 0.0450 (10)  | 0.0628 (12)  | 0.0483 (10)  | 0.0109 (9)   | 0.0024 (8)   | 0.0034 (9)   |
| C4   | 0.0495 (10)  | 0.0737 (13)  | 0.0584 (11)  | −0.0050 (9)  | 0.0309 (9)   | 0.0111 (10)  |
| C21  | 0.0622 (11)  | 0.0615 (11)  | 0.0737 (12)  | 0.0038 (8)   | 0.0182 (9)   | −0.0083 (9)  |
| C5   | 0.0590 (11)  | 0.0818 (15)  | 0.0488 (10)  | 0.0038 (10)  | 0.0331 (9)   | 0.0040 (10)  |
| C26  | 0.0647 (10)  | 0.0593 (10)  | 0.0802 (15)  | 0.0043 (7)   | 0.0285 (11)  | 0.0090 (10)  |
| C22  | 0.0639 (12)  | 0.0776 (14)  | 0.0617 (11)  | 0.0109 (10)  | 0.0240 (9)   | 0.0020 (9)   |
| C23  | 0.0566 (9)   | 0.0678 (10)  | 0.0771 (15)  | 0.0017 (8)   | 0.0230 (10)  | 0.0096 (11)  |
| C24  | 0.0613 (11)  | 0.0634 (11)  | 0.0679 (12)  | 0.0029 (8)   | 0.0110 (9)   | −0.0036 (9)  |
| C25  | 0.0712 (13)  | 0.0710 (14)  | 0.0591 (10)  | 0.0125 (10)  | 0.0223 (9)   | 0.0061 (8)   |
| C22a | 0.049 (4)    | 0.074 (4)    | 0.039 (3)    | 0.017 (3)    | 0.026 (2)    | 0.036 (3)    |
| Br3a | 0.151 (2)    | 0.0660 (7)   | 0.1218 (16)  | −0.0227 (9)  | −0.0096 (16) | −0.0013 (8)  |
| C21a | 0.069 (4)    | 0.030 (3)    | 0.035 (3)    | 0.015 (2)    | 0.022 (2)    | 0.0235 (19)  |
| C26a | 0.059 (4)    | 0.080 (5)    | 0.038 (3)    | −0.029 (3)   | 0.026 (3)    | −0.001 (3)   |
| C25a | 0.053 (4)    | 0.073 (5)    | 0.076 (4)    | −0.024 (3)   | 0.027 (3)    | −0.012 (3)   |
| C23a | 0.034 (3)    | 0.079 (4)    | 0.072 (4)    | 0.004 (3)    | 0.007 (3)    | 0.033 (3)    |
| C24a | 0.041 (3)    | 0.055 (4)    | 0.069 (4)    | −0.003 (2)   | 0.008 (3)    | 0.013 (3)    |



*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N3    | 2.1985 (13) | C18—H16     | 1.0930      |
| Fe1—N4    | 2.2425 (13) | C18—C17     | 1.384 (3)   |
| Fe1—N5    | 2.2086 (13) | C10—H8      | 1.0930      |
| Fe1—N6    | 2.2168 (14) | C10—C11     | 1.385 (3)   |
| Fe1—N1    | 2.1157 (15) | C6—H4       | 1.0930      |
| Fe1—N2    | 2.0521 (16) | C6—C5       | 1.385 (3)   |
| Br3—C21   | 1.865 (4)   | C15—H13     | 1.0930      |
| S2—C2     | 1.6184 (18) | C15—C16     | 1.388 (3)   |
| S1—C1     | 1.6166 (18) | C11—H9      | 1.0930      |
| N3—C7     | 1.336 (2)   | C17—H15     | 1.0930      |
| N3—C3     | 1.344 (2)   | C17—C16     | 1.387 (3)   |
| N4—C14    | 1.474 (2)   | C16—H14     | 1.0930      |
| N4—C8     | 1.479 (2)   | C4—H2       | 1.0930      |
| N4—C20    | 1.4766 (19) | C4—C5       | 1.384 (3)   |
| N5—C13    | 1.341 (2)   | C21—C26     | 1.392 (6)   |
| N5—C9     | 1.343 (2)   | C21—C22     | 1.386 (6)   |
| N6—C19    | 1.340 (2)   | C5—H3       | 1.0930      |
| N6—C15    | 1.337 (2)   | C26—H26     | 1.0930      |
| N1—C1     | 1.157 (2)   | C26—C25     | 1.387 (6)   |
| N2—C2     | 1.163 (2)   | C22—H18     | 1.0930      |
| C7—C8     | 1.510 (2)   | C22—C23     | 1.366 (6)   |
| C7—C6     | 1.392 (2)   | C23—H20     | 1.0930      |
| C19—C20   | 1.505 (2)   | C23—C24     | 1.343 (6)   |
| C19—C18   | 1.387 (2)   | C24—H22     | 1.0930      |
| C13—C14   | 1.507 (2)   | C24—C25     | 1.378 (6)   |
| C13—C12   | 1.390 (2)   | C25—H24     | 1.0930      |
| C14—H12   | 1.1010      | C22a—H17    | 1.0930      |
| C14—H11   | 1.1010      | C22a—C21a   | 1.3900      |
| C8—H5     | 1.1010      | C22a—C23a   | 1.3900      |
| C8—H6     | 1.1010      | Br3a—C21a   | 1.937 (4)   |
| C9—H7     | 1.0930      | C21a—C26a   | 1.3900      |
| C9—C10    | 1.386 (2)   | C26a—H25    | 1.0930      |
| C20—H00e  | 1.1010      | C26a—C25a   | 1.3900      |
| C20—H00f  | 1.1010      | C25a—H23    | 1.0930      |
| C12—H10   | 1.0930      | C25a—C24a   | 1.3900      |
| C12—C11   | 1.389 (3)   | C23a—H19    | 1.0930      |
| C3—H1     | 1.0930      | C23a—C24a   | 1.3900      |
| C3—C4     | 1.379 (3)   | C24a—H21    | 1.0930      |
| N4—Fe1—N3 | 78.08 (5)   | C4—C3—N3    | 122.95 (18) |
| N5—Fe1—N3 | 89.35 (5)   | C4—C3—H1    | 118.52 (12) |
| N5—Fe1—N4 | 76.02 (5)   | H16—C18—C19 | 120.58 (10) |
| N6—Fe1—N3 | 83.76 (5)   | C17—C18—C19 | 118.84 (17) |
| N6—Fe1—N4 | 75.29 (5)   | C17—C18—H16 | 120.58 (11) |
| N6—Fe1—N5 | 151.30 (5)  | H8—C10—C9   | 120.61 (11) |
| N1—Fe1—N3 | 169.28 (6)  | C11—C10—C9  | 118.78 (17) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N1—Fe1—N4   | 92.02 (6)   | C11—C10—H8     | 120.61 (11) |
| N1—Fe1—N5   | 92.26 (5)   | N1—C1—S1       | 179.07 (16) |
| N1—Fe1—N6   | 89.79 (5)   | H4—C6—C7       | 120.41 (11) |
| N2—Fe1—N3   | 95.00 (6)   | C5—C6—C7       | 119.18 (18) |
| N2—Fe1—N4   | 172.22 (6)  | C5—C6—H4       | 120.41 (12) |
| N2—Fe1—N5   | 100.55 (6)  | H13—C15—N6     | 118.69 (10) |
| N2—Fe1—N6   | 107.77 (6)  | C16—C15—N6     | 122.62 (18) |
| N2—Fe1—N1   | 95.12 (7)   | C16—C15—H13    | 118.69 (12) |
| C7—N3—Fe1   | 116.13 (10) | C10—C11—C12    | 118.92 (17) |
| C3—N3—Fe1   | 124.96 (12) | H9—C11—C12     | 120.54 (11) |
| C3—N3—C7    | 118.74 (14) | H9—C11—C10     | 120.54 (11) |
| C14—N4—Fe1  | 106.02 (9)  | H15—C17—C18    | 120.48 (11) |
| C8—N4—Fe1   | 111.22 (9)  | C16—C17—C18    | 119.04 (17) |
| C8—N4—C14   | 111.45 (13) | C16—C17—H15    | 120.48 (11) |
| C20—N4—Fe1  | 105.54 (9)  | C17—C16—C15    | 118.51 (17) |
| C20—N4—C14  | 110.43 (12) | H14—C16—C15    | 120.75 (12) |
| C20—N4—C8   | 111.87 (13) | H14—C16—C17    | 120.75 (11) |
| C13—N5—Fe1  | 114.65 (10) | H2—C4—C3       | 120.81 (12) |
| C9—N5—Fe1   | 126.73 (11) | C5—C4—C3       | 118.38 (18) |
| C9—N5—C13   | 118.61 (14) | C5—C4—H2       | 120.81 (11) |
| C19—N6—Fe1  | 114.71 (10) | C26—C21—Br3    | 116.0 (4)   |
| C15—N6—Fe1  | 126.69 (12) | C22—C21—Br3    | 122.5 (4)   |
| C15—N6—C19  | 118.60 (15) | C22—C21—C26    | 121.5 (4)   |
| C1—N1—Fe1   | 166.16 (16) | C4—C5—C6       | 119.11 (17) |
| C2—N2—Fe1   | 172.31 (16) | H3—C5—C6       | 120.45 (12) |
| C8—C7—N3    | 118.61 (13) | H3—C5—C4       | 120.45 (11) |
| C6—C7—N3    | 121.65 (15) | H26—C26—C21    | 121.1 (3)   |
| C6—C7—C8    | 119.72 (15) | C25—C26—C21    | 117.8 (4)   |
| C20—C19—N6  | 115.87 (14) | C25—C26—H26    | 121.1 (3)   |
| C18—C19—N6  | 122.33 (15) | H18—C22—C21    | 120.7 (3)   |
| C18—C19—C20 | 121.74 (15) | C23—C22—C21    | 118.5 (4)   |
| C14—C13—N5  | 116.27 (14) | C23—C22—H18    | 120.7 (3)   |
| C12—C13—N5  | 122.17 (16) | H20—C23—C22    | 119.5 (3)   |
| C12—C13—C14 | 121.49 (15) | C24—C23—C22    | 121.1 (4)   |
| C13—C14—N4  | 111.16 (13) | C24—C23—H20    | 119.5 (3)   |
| H12—C14—N4  | 109.40 (8)  | H22—C24—C23    | 119.4 (3)   |
| H12—C14—C13 | 109.40 (9)  | C25—C24—C23    | 121.2 (4)   |
| H11—C14—N4  | 109.40 (8)  | C25—C24—H22    | 119.4 (3)   |
| H11—C14—C13 | 109.40 (9)  | C24—C25—C26    | 119.8 (4)   |
| H11—C14—H12 | 108.0       | H24—C25—C26    | 120.1 (3)   |
| C7—C8—N4    | 115.51 (13) | H24—C25—C24    | 120.1 (3)   |
| H5—C8—N4    | 108.40 (8)  | C21a—C22a—H17  | 120.0 (2)   |
| H5—C8—C7    | 108.40 (9)  | C23a—C22a—H17  | 120.0 (2)   |
| H6—C8—N4    | 108.40 (8)  | C23a—C22a—C21a | 120.0       |
| H6—C8—C7    | 108.40 (9)  | Br3a—C21a—C22a | 126.6 (4)   |
| H6—C8—H5    | 107.5       | C26a—C21a—C22a | 120.0       |
| H7—C9—N5    | 118.71 (10) | C26a—C21a—Br3a | 112.9 (4)   |
| C10—C9—N5   | 122.58 (17) | H25—C26a—C21a  | 120.0 (2)   |

|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| C10—C9—H7       | 118.71 (11)  | C25a—C26a—C21a      | 120.0        |
| N2—C2—S2        | 178.81 (17)  | C25a—C26a—H25       | 120.0 (2)    |
| C19—C20—N4      | 111.10 (13)  | H23—C25a—C26a       | 120.0 (2)    |
| H00e—C20—N4     | 109.42 (8)   | C24a—C25a—C26a      | 120.0        |
| H00e—C20—C19    | 109.42 (9)   | C24a—C25a—H23       | 120.0 (2)    |
| H00f—C20—N4     | 109.42 (9)   | H19—C23a—C22a       | 120.0        |
| H00f—C20—C19    | 109.42 (9)   | C24a—C23a—C22a      | 120.0        |
| H00f—C20—H00e   | 108.0        | C24a—C23a—H19       | 120.0        |
| H10—C12—C13     | 120.53 (11)  | C23a—C24a—C25a      | 120.0        |
| C11—C12—C13     | 118.94 (17)  | H21—C24a—C25a       | 120.0 (2)    |
| C11—C12—H10     | 120.53 (11)  | H21—C24a—C23a       | 120.0 (2)    |
| H1—C3—N3        | 118.52 (10)  |                     |              |
| Fe1—N3—C7—C8    | −6.32 (12)   | C19—C20—N4—C8       | −76.29 (15)  |
| Fe1—N3—C7—C6    | 175.55 (12)  | C19—C18—C17—C16     | 2.3 (2)      |
| Fe1—N3—C3—C4    | −175.17 (15) | C13—N5—C9—C10       | −0.30 (17)   |
| Fe1—N4—C14—C13  | −42.98 (9)   | C13—C14—N4—C8       | 78.17 (15)   |
| Fe1—N4—C8—C7    | −5.40 (10)   | C13—C14—N4—C20      | −156.84 (14) |
| Fe1—N4—C20—C19  | 44.80 (10)   | C13—C12—C11—C10     | −0.6 (2)     |
| Fe1—N5—C13—C14  | −4.81 (11)   | C14—C13—N5—C9       | 176.65 (14)  |
| Fe1—N5—C13—C12  | 178.26 (11)  | C14—C13—C12—C11     | −176.03 (17) |
| Fe1—N5—C9—C10   | −178.65 (13) | C8—C7—N3—C3         | 178.20 (15)  |
| Fe1—N6—C19—C20  | 1.52 (12)    | C8—C7—C6—C5         | −178.05 (17) |
| Fe1—N6—C19—C18  | 178.91 (11)  | C9—N5—C13—C12       | −0.29 (17)   |
| Fe1—N6—C15—C16  | −177.71 (15) | C9—C10—C11—C12      | 0.1 (2)      |
| Br3—C21—C26—C25 | 178.6 (3)    | C20—C19—N6—C15      | −178.40 (14) |
| Br3—C21—C22—C23 | 179.4 (3)    | C20—C19—C18—C17     | 176.03 (17)  |
| N3—C7—C8—N4     | 7.99 (15)    | C3—N3—C7—C6         | 0.07 (18)    |
| N3—C7—C6—C5     | 0.06 (19)    | C3—C4—C5—C6         | 0.1 (2)      |
| N3—C3—C4—C5     | 0.1 (2)      | C18—C19—N6—C15      | −1.01 (19)   |
| N4—C14—C13—N5   | 33.54 (16)   | C18—C17—C16—C15     | −1.2 (2)     |
| N4—C14—C13—C12  | −149.50 (14) | C21—C26—C25—C24     | 1.9 (4)      |
| N4—C8—C7—C6     | −173.84 (14) | C21—C22—C23—C24     | 2.3 (5)      |
| N4—C20—C19—N6   | −32.49 (16)  | C26—C21—C22—C23     | −2.4 (6)     |
| N4—C20—C19—C18  | 150.10 (13)  | C26—C25—C24—C23     | −2.0 (4)     |
| N5—C13—C12—C11  | 0.75 (19)    | C22—C21—C26—C25     | 0.3 (5)      |
| N5—C9—C10—C11   | 0.4 (2)      | C22—C23—C24—C25     | −0.2 (5)     |
| N6—C19—C18—C17  | −1.21 (19)   | C22a—C21a—C26a—C25a | 0.0 (5)      |
| N6—C15—C16—C17  | −1.1 (2)     | C22a—C23a—C24a—C25a | −0.0 (6)     |
| C7—N3—C3—C4     | −0.13 (18)   | Br3a—C21a—C22a—C23a | 171.3 (2)    |
| C7—C8—N4—C14    | −123.47 (14) | Br3a—C21a—C26a—C25a | −172.4 (2)   |
| C7—C8—N4—C20    | 112.35 (15)  | C21a—C22a—C23a—C24a | 0.0 (5)      |
| C7—C6—C5—C4     | −0.1 (2)     | C21a—C26a—C25a—C24a | −0.0 (6)     |
| C19—N6—C15—C16  | 2.20 (18)    | C26a—C21a—C22a—C23a | −0.0 (5)     |
| C19—C20—N4—C14  | 158.96 (13)  | C26a—C25a—C24a—C23a | 0.0 (5)      |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) bromobenzene monosolvate  
(br\_270K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Br

$M_r = 619.39$

Monoclinic,  $P2_1/n$

$a = 15.2951$  (5) Å

$b = 12.3099$  (3) Å

$c = 15.5994$  (5) Å

$\beta = 110.210$  (4)°

$V = 2756.25$  (16) Å<sup>3</sup>

$Z = 4$

$F(000) = 1258.243$

$D_x = 1.493$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 30848 reflections

$\theta = 2.1$ – $28.9^\circ$

$\mu = 2.18$  mm<sup>-1</sup>

$T = 270$  K

Block, yellow

$0.36 \times 0.26 \times 0.09$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.338$ ,  $T_{\max} = 1.000$

68934 measured reflections

6846 independent reflections

5548 reflections with  $I \geq 2\sigma(I)$

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

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.077$

$S = 1.05$

6846 reflections

406 parameters

34 restraints

56 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.6102P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.46$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.37$  e Å<sup>-3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Fe1 | 0.649931 (14) | 0.318249 (16) | 0.301843 (13) | 0.04100 (7)                      | 0.582 (3) |
| Br3 | 0.65196 (15)  | 0.03168 (13)  | 0.55558 (10)  | 0.1028 (4)                       |           |
| S2  | 0.46841 (7)   | 0.00544 (9)   | 0.16241 (8)   | 0.06669 (14)                     |           |
| S1  | 0.74466 (10)  | 0.30986 (11)  | 0.03635 (8)   | 0.1121 (3)                       |           |
| N4  | 0.73971 (8)   | 0.45538 (9)   | 0.37922 (8)   | 0.0406 (2)                       |           |
| N3  | 0.60511 (8)   | 0.33384 (10)  | 0.42131 (8)   | 0.0441 (3)                       |           |
| N5  | 0.77928 (8)   | 0.23757 (9)   | 0.38911 (7)   | 0.0433 (3)                       |           |
| N6  | 0.56364 (8)   | 0.46609 (10)  | 0.25428 (8)   | 0.0458 (3)                       |           |
| N1  | 0.70102 (11)  | 0.33392 (12)  | 0.19234 (9)   | 0.0593 (3)                       |           |
| C7  | 0.64510 (9)   | 0.41353 (12)  | 0.47971 (9)   | 0.0431 (3)                       |           |
| C19 | 0.61138 (10)  | 0.55972 (12)  | 0.27169 (9)   | 0.0446 (3)                       |           |
| N2  | 0.56721 (11)  | 0.18617 (13)  | 0.24843 (10)  | 0.0664 (4)                       |           |
| C13 | 0.85197 (10)  | 0.30468 (12)  | 0.42543 (10)  | 0.0453 (3)                       |           |
| C14 | 0.83654 (10)  | 0.42183 (12)  | 0.39571 (11)  | 0.0498 (3)                       |           |

|      |              |              |              |              |           |
|------|--------------|--------------|--------------|--------------|-----------|
| H12  | 0.85251 (10) | 0.43266 (12) | 0.33328 (11) | 0.0597 (4)*  |           |
| H11  | 0.88370 (10) | 0.47337 (12) | 0.44849 (11) | 0.0597 (4)*  |           |
| C8   | 0.72340 (11) | 0.47674 (13) | 0.46570 (10) | 0.0518 (4)   |           |
| H5   | 0.78746 (11) | 0.45878 (13) | 0.52227 (10) | 0.0622 (4)*  |           |
| H6   | 0.70905 (11) | 0.56317 (13) | 0.46886 (10) | 0.0622 (4)*  |           |
| C9   | 0.79062 (11) | 0.13166 (12) | 0.40979 (10) | 0.0506 (3)   |           |
| H7   | 0.73190 (11) | 0.07757 (12) | 0.38099 (10) | 0.0607 (4)*  |           |
| C20  | 0.71505 (10) | 0.54980 (12) | 0.31701 (11) | 0.0506 (3)   |           |
| H17  | 0.74232 (10) | 0.62373 (12) | 0.35555 (11) | 0.0607 (4)*  |           |
| H18  | 0.74721 (10) | 0.54092 (12) | 0.26492 (11) | 0.0607 (4)*  |           |
| C18  | 0.56832 (12) | 0.65974 (13) | 0.24555 (11) | 0.0567 (4)   |           |
| H16  | 0.60841 (12) | 0.73421 (13) | 0.26153 (11) | 0.0681 (5)*  |           |
| C2   | 0.52591 (10) | 0.11017 (13) | 0.21230 (11) | 0.0499 (3)   |           |
| C12  | 0.93768 (11) | 0.26752 (14) | 0.48338 (11) | 0.0565 (4)   |           |
| H10  | 0.99501 (11) | 0.32346 (14) | 0.51221 (11) | 0.0678 (5)*  |           |
| C1   | 0.71878 (12) | 0.32403 (13) | 0.12652 (11) | 0.0550 (4)   |           |
| C3   | 0.53712 (11) | 0.27530 (15) | 0.43516 (12) | 0.0575 (4)   |           |
| H1   | 0.50482 (11) | 0.21080 (15) | 0.38753 (12) | 0.0690 (5)*  |           |
| C10  | 0.87422 (13) | 0.08837 (14) | 0.46645 (11) | 0.0616 (4)   |           |
| H8   | 0.88097 (13) | 0.00217 (14) | 0.48112 (11) | 0.0739 (5)*  |           |
| C6   | 0.61791 (12) | 0.43664 (16) | 0.55431 (11) | 0.0612 (4)   |           |
| H4   | 0.65130 (12) | 0.50149 (16) | 0.60108 (11) | 0.0735 (5)*  |           |
| C15  | 0.47153 (11) | 0.46977 (15) | 0.21130 (11) | 0.0577 (4)   |           |
| H13  | 0.43270 (11) | 0.39435 (15) | 0.19896 (11) | 0.0692 (5)*  |           |
| C11  | 0.94875 (13) | 0.15765 (15) | 0.50379 (12) | 0.0639 (4)   |           |
| H9   | 1.01506 (13) | 0.12652 (15) | 0.54847 (12) | 0.0767 (5)*  |           |
| C16  | 0.42370 (13) | 0.56567 (17) | 0.18190 (12) | 0.0678 (5)   |           |
| H14  | 0.34906 (13) | 0.56533 (17) | 0.14645 (12) | 0.0813 (6)*  |           |
| C17  | 0.47325 (13) | 0.66247 (16) | 0.19874 (13) | 0.0667 (5)   |           |
| H15  | 0.43806 (13) | 0.73898 (16) | 0.17560 (13) | 0.0800 (6)*  |           |
| C4   | 0.50663 (13) | 0.29372 (18) | 0.50738 (14) | 0.0719 (5)   |           |
| H2   | 0.45157 (13) | 0.24476 (18) | 0.51631 (14) | 0.0863 (6)*  |           |
| C5   | 0.54779 (14) | 0.37581 (19) | 0.56795 (13) | 0.0750 (5)   |           |
| H3   | 0.52554 (14) | 0.39248 (19) | 0.62544 (13) | 0.0901 (7)*  |           |
| C21  | 0.7058 (4)   | 0.1240 (4)   | 0.6474 (3)   | 0.0596 (13)  | 0.582 (3) |
| C26  | 0.6883 (4)   | 0.1098 (4)   | 0.7284 (4)   | 0.0718 (15)  | 0.582 (3) |
| H28  | 0.6441 (4)   | 0.0438 (4)   | 0.7347 (4)   | 0.0861 (18)* | 0.582 (3) |
| C23  | 0.8017 (3)   | 0.2792 (3)   | 0.7121 (3)   | 0.085 (2)    | 0.582 (3) |
| H22  | 0.8459 (3)   | 0.3452 (3)   | 0.7057 (3)   | 0.102 (2)*   | 0.582 (3) |
| C22  | 0.7625 (4)   | 0.2087 (4)   | 0.6392 (3)   | 0.0741 (15)  | 0.582 (3) |
| H20  | 0.7761 (4)   | 0.2198 (4)   | 0.5761 (3)   | 0.0889 (18)* | 0.582 (3) |
| C24  | 0.7842 (3)   | 0.2649 (3)   | 0.7930 (2)   | 0.0708 (12)  | 0.582 (3) |
| H24  | 0.8148 (3)   | 0.3198 (3)   | 0.8498 (2)   | 0.0850 (15)* | 0.582 (3) |
| C25  | 0.7275 (4)   | 0.1802 (4)   | 0.8012 (2)   | 0.0809 (14)  | 0.582 (3) |
| H26  | 0.7139 (4)   | 0.1692 (4)   | 0.8643 (2)   | 0.0971 (17)* | 0.582 (3) |
| C22a | 0.7366 (6)   | 0.1781 (9)   | 0.6183 (4)   | 0.100 (4)    | 0.418 (3) |
| H19  | 0.7282 (6)   | 0.1625 (9)   | 0.5475 (4)   | 0.120 (4)*   | 0.418 (3) |
| Br3a | 0.6034 (3)   | 0.01163 (18) | 0.5831 (2)   | 0.2060 (12)  | 0.418 (3) |

|      |            |            |            |            |           |
|------|------------|------------|------------|------------|-----------|
| C21a | 0.6867 (6) | 0.1173 (7) | 0.6609 (5) | 0.072 (2)  | 0.418 (3) |
| C26a | 0.6975 (5) | 0.1373 (7) | 0.7516 (5) | 0.079 (2)  | 0.418 (3) |
| H27  | 0.6587 (5) | 0.0899 (7) | 0.7848 (5) | 0.095 (3)* | 0.418 (3) |
| C25a | 0.7581 (5) | 0.2181 (7) | 0.7999 (3) | 0.113 (4)  | 0.418 (3) |
| H25  | 0.7665 (5) | 0.2336 (7) | 0.8706 (3) | 0.136 (5)* | 0.418 (3) |
| C23a | 0.7972 (4) | 0.2589 (8) | 0.6665 (6) | 0.129 (5)  | 0.418 (3) |
| H21  | 0.8360 (4) | 0.3063 (8) | 0.6333 (6) | 0.155 (6)* | 0.418 (3) |
| C24a | 0.8079 (4) | 0.2789 (5) | 0.7573 (6) | 0.085 (3)  | 0.418 (3) |
| H23  | 0.8551 (4) | 0.3418 (5) | 0.7948 (6) | 0.102 (4)* | 0.418 (3) |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1 | 0.04470 (12) | 0.03974 (11) | 0.03654 (11) | −0.00324 (8) | 0.01146 (8) | −0.00148 (8) |
| Br3 | 0.1457 (7)   | 0.0685 (4)   | 0.0871 (4)   | 0.0053 (4)   | 0.0312 (4)  | −0.0065 (3)  |
| S2  | 0.0569 (2)   | 0.0589 (3)   | 0.0900 (4)   | −0.0167 (2)  | 0.0327 (2)  | −0.0228 (2)  |
| S1  | 0.1817 (7)   | 0.1117 (5)   | 0.0690 (3)   | 0.0726 (5)   | 0.0764 (4)  | 0.0360 (3)   |
| N4  | 0.0406 (6)   | 0.0368 (6)   | 0.0431 (6)   | −0.0046 (5)  | 0.0129 (5)  | 0.0022 (5)   |
| N3  | 0.0436 (6)   | 0.0490 (7)   | 0.0405 (6)   | −0.0052 (5)  | 0.0156 (5)  | 0.0013 (5)   |
| N5  | 0.0470 (6)   | 0.0406 (6)   | 0.0405 (6)   | 0.0001 (5)   | 0.0128 (5)  | 0.0017 (5)   |
| N6  | 0.0474 (6)   | 0.0439 (7)   | 0.0405 (6)   | 0.0009 (5)   | 0.0083 (5)  | −0.0005 (5)  |
| N1  | 0.0778 (9)   | 0.0600 (8)   | 0.0468 (7)   | 0.0087 (7)   | 0.0301 (7)  | 0.0035 (6)   |
| C7  | 0.0445 (7)   | 0.0465 (7)   | 0.0367 (6)   | 0.0034 (6)   | 0.0122 (6)  | 0.0019 (6)   |
| C19 | 0.0509 (8)   | 0.0403 (7)   | 0.0404 (7)   | 0.0024 (6)   | 0.0130 (6)  | 0.0045 (6)   |
| N2  | 0.0705 (9)   | 0.0589 (9)   | 0.0632 (9)   | −0.0198 (7)  | 0.0148 (7)  | −0.0155 (7)  |
| C13 | 0.0435 (7)   | 0.0444 (8)   | 0.0456 (7)   | 0.0016 (6)   | 0.0124 (6)  | 0.0007 (6)   |
| C14 | 0.0414 (7)   | 0.0458 (8)   | 0.0600 (9)   | −0.0050 (6)  | 0.0148 (7)  | 0.0039 (7)   |
| C8  | 0.0563 (9)   | 0.0512 (9)   | 0.0456 (8)   | −0.0113 (7)  | 0.0145 (7)  | −0.0075 (6)  |
| C9  | 0.0637 (9)   | 0.0408 (8)   | 0.0455 (7)   | 0.0007 (7)   | 0.0168 (7)  | −0.0001 (6)  |
| C20 | 0.0513 (8)   | 0.0404 (7)   | 0.0577 (9)   | −0.0044 (6)  | 0.0159 (7)  | 0.0087 (6)   |
| C18 | 0.0649 (10)  | 0.0451 (9)   | 0.0579 (9)   | 0.0087 (7)   | 0.0184 (8)  | 0.0085 (7)   |
| C2  | 0.0472 (8)   | 0.0502 (9)   | 0.0528 (8)   | −0.0075 (7)  | 0.0179 (7)  | −0.0074 (7)  |
| C12 | 0.0459 (8)   | 0.0573 (10)  | 0.0587 (9)   | 0.0070 (7)   | 0.0085 (7)  | −0.0015 (8)  |
| C1  | 0.0727 (10)  | 0.0512 (8)   | 0.0438 (8)   | 0.0171 (7)   | 0.0237 (7)  | 0.0100 (7)   |
| C3  | 0.0511 (8)   | 0.0656 (10)  | 0.0586 (9)   | −0.0113 (8)  | 0.0226 (7)  | 0.0044 (8)   |
| C10 | 0.0770 (11)  | 0.0470 (9)   | 0.0566 (9)   | 0.0152 (8)   | 0.0177 (8)  | 0.0055 (7)   |
| C6  | 0.0668 (10)  | 0.0742 (11)  | 0.0462 (8)   | 0.0049 (9)   | 0.0241 (8)  | −0.0055 (8)  |
| C15 | 0.0489 (9)   | 0.0617 (10)  | 0.0518 (8)   | −0.0005 (7)  | 0.0038 (7)  | −0.0035 (7)  |
| C11 | 0.0616 (10)  | 0.0612 (11)  | 0.0604 (10)  | 0.0200 (8)   | 0.0101 (8)  | 0.0047 (8)   |
| C16 | 0.0532 (9)   | 0.0768 (13)  | 0.0607 (10)  | 0.0138 (9)   | 0.0036 (8)  | 0.0034 (9)   |
| C17 | 0.0671 (11)  | 0.0613 (11)  | 0.0651 (10)  | 0.0206 (9)   | 0.0144 (9)  | 0.0128 (8)   |
| C4  | 0.0618 (10)  | 0.0930 (14)  | 0.0732 (11)  | −0.0054 (10) | 0.0391 (9)  | 0.0146 (11)  |
| C5  | 0.0743 (12)  | 0.1040 (16)  | 0.0610 (10)  | 0.0051 (11)  | 0.0415 (10) | 0.0044 (11)  |
| C21 | 0.065 (2)    | 0.060 (2)    | 0.068 (3)    | 0.0143 (18)  | 0.040 (2)   | 0.019 (2)    |
| C26 | 0.103 (4)    | 0.064 (3)    | 0.071 (3)    | 0.001 (2)    | 0.058 (3)   | 0.001 (2)    |
| C23 | 0.058 (3)    | 0.077 (3)    | 0.105 (6)    | 0.004 (2)    | 0.009 (3)   | 0.008 (3)    |
| C22 | 0.060 (3)    | 0.068 (3)    | 0.106 (4)    | 0.011 (3)    | 0.045 (3)   | 0.023 (3)    |
| C24 | 0.049 (3)    | 0.072 (3)    | 0.086 (3)    | −0.004 (2)   | 0.017 (2)   | −0.004 (2)   |



|      |           |            |             |              |              |            |
|------|-----------|------------|-------------|--------------|--------------|------------|
| C25  | 0.093 (4) | 0.082 (4)  | 0.064 (3)   | 0.004 (3)    | 0.023 (3)    | −0.017 (2) |
| C22a | 0.082 (6) | 0.152 (10) | 0.081 (4)   | 0.040 (5)    | 0.047 (4)    | 0.066 (5)  |
| Br3a | 0.237 (2) | 0.0851 (8) | 0.2011 (16) | −0.0360 (10) | −0.0445 (15) | 0.0042 (9) |
| C21a | 0.095 (6) | 0.070 (4)  | 0.066 (4)   | 0.021 (3)    | 0.049 (3)    | 0.011 (3)  |
| C26a | 0.083 (4) | 0.096 (6)  | 0.069 (5)   | −0.007 (4)   | 0.040 (4)    | −0.003 (4) |
| C25a | 0.053 (4) | 0.090 (7)  | 0.175 (9)   | −0.019 (4)   | 0.012 (4)    | −0.040 (6) |
| C23a | 0.042 (4) | 0.130 (10) | 0.185 (12)  | −0.010 (4)   | 0.002 (5)    | 0.084 (8)  |
| C24a | 0.052 (4) | 0.074 (4)  | 0.113 (9)   | −0.000 (3)   | 0.006 (4)    | 0.020 (5)  |

*Geometric parameters (Å, °)*

|         |             |           |           |
|---------|-------------|-----------|-----------|
| Fe1—N4  | 2.2465 (11) | C3—H1     | 1.0830    |
| Fe1—N3  | 2.2051 (12) | C3—C4     | 1.379 (2) |
| Fe1—N5  | 2.2120 (12) | C10—H8    | 1.0830    |
| Fe1—N6  | 2.2199 (12) | C10—C11   | 1.381 (3) |
| Fe1—N1  | 2.1185 (14) | C6—H4     | 1.0830    |
| Fe1—N2  | 2.0519 (14) | C6—C5     | 1.383 (3) |
| Br3—C21 | 1.791 (3)   | C15—H13   | 1.0830    |
| S2—C2   | 1.6026 (18) | C15—C16   | 1.381 (2) |
| S1—C1   | 1.5970 (19) | C11—H9    | 1.0830    |
| N4—C14  | 1.4721 (18) | C16—H14   | 1.0830    |
| N4—C8   | 1.4776 (18) | C16—C17   | 1.388 (3) |
| N4—C20  | 1.4773 (17) | C17—H15   | 1.0830    |
| N3—C7   | 1.3347 (18) | C4—H2     | 1.0830    |
| N3—C3   | 1.3427 (18) | C4—C5     | 1.378 (3) |
| N5—C13  | 1.3432 (18) | C5—H3     | 1.0830    |
| N5—C9   | 1.3399 (19) | C21—C26   | 1.3900    |
| N6—C19  | 1.3411 (19) | C21—C22   | 1.3900    |
| N6—C15  | 1.3355 (19) | C26—H28   | 1.0830    |
| N1—C1   | 1.156 (2)   | C26—C25   | 1.3900    |
| C7—C8   | 1.506 (2)   | C23—H22   | 1.0830    |
| C7—C6   | 1.394 (2)   | C23—C22   | 1.3900    |
| C19—C20 | 1.501 (2)   | C23—C24   | 1.3900    |
| C19—C18 | 1.389 (2)   | C22—H20   | 1.0830    |
| N2—C2   | 1.160 (2)   | C24—H24   | 1.0830    |
| C13—C14 | 1.508 (2)   | C24—C25   | 1.3900    |
| C13—C12 | 1.388 (2)   | C25—H26   | 1.0830    |
| C14—H12 | 1.0910      | C22a—H19  | 1.0830    |
| C14—H11 | 1.0910      | C22a—C21a | 1.3900    |
| C8—H5   | 1.0910      | C22a—C23a | 1.3900    |
| C8—H6   | 1.0910      | Br3a—C21a | 1.929 (5) |
| C9—H7   | 1.0830      | C21a—C26a | 1.3900    |
| C9—C10  | 1.385 (2)   | C26a—H27  | 1.0830    |
| C20—H17 | 1.0910      | C26a—C25a | 1.3900    |
| C20—H18 | 1.0910      | C25a—H25  | 1.0830    |
| C18—H16 | 1.0830      | C25a—C24a | 1.3900    |
| C18—C17 | 1.384 (2)   | C23a—H21  | 1.0830    |
| C12—H10 | 1.0830      | C23a—C24a | 1.3900    |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C12—C11     | 1.386 (2)   | C24a—H23    | 1.0830      |
| N3—Fe1—N4   | 77.95 (4)   | C11—C12—C13 | 118.95 (16) |
| N5—Fe1—N4   | 75.87 (4)   | C11—C12—H10 | 120.52 (10) |
| N5—Fe1—N3   | 89.54 (4)   | N1—C1—S1    | 179.25 (17) |
| N6—Fe1—N4   | 75.29 (4)   | H1—C3—N3    | 118.57 (9)  |
| N6—Fe1—N3   | 83.60 (4)   | C4—C3—N3    | 122.86 (17) |
| N6—Fe1—N5   | 151.15 (4)  | C4—C3—H1    | 118.57 (11) |
| N1—Fe1—N4   | 92.15 (5)   | H8—C10—C9   | 120.76 (10) |
| N1—Fe1—N3   | 169.26 (5)  | C11—C10—C9  | 118.49 (16) |
| N1—Fe1—N5   | 92.09 (5)   | C11—C10—H8  | 120.76 (10) |
| N1—Fe1—N6   | 89.90 (5)   | H4—C6—C7    | 120.37 (10) |
| N2—Fe1—N4   | 172.01 (5)  | C5—C6—C7    | 119.26 (17) |
| N2—Fe1—N3   | 94.82 (5)   | C5—C6—H4    | 120.37 (11) |
| N2—Fe1—N5   | 100.90 (6)  | H13—C15—N6  | 118.60 (9)  |
| N2—Fe1—N6   | 107.57 (6)  | C16—C15—N6  | 122.80 (16) |
| N2—Fe1—N1   | 95.29 (6)   | C16—C15—H13 | 118.60 (11) |
| C14—N4—Fe1  | 105.93 (9)  | C10—C11—C12 | 119.21 (16) |
| C8—N4—Fe1   | 111.08 (8)  | H9—C11—C12  | 120.39 (10) |
| C8—N4—C14   | 111.55 (12) | H9—C11—C10  | 120.39 (10) |
| C20—N4—Fe1  | 105.56 (8)  | H14—C16—C15 | 120.69 (11) |
| C20—N4—C14  | 110.40 (11) | C17—C16—C15 | 118.62 (16) |
| C20—N4—C8   | 111.99 (12) | C17—C16—H14 | 120.69 (10) |
| C7—N3—Fe1   | 115.97 (9)  | C16—C17—C18 | 118.99 (16) |
| C3—N3—Fe1   | 125.00 (11) | H15—C17—C18 | 120.50 (11) |
| C3—N3—C7    | 118.84 (13) | H15—C17—C16 | 120.50 (10) |
| C13—N5—Fe1  | 114.67 (9)  | H2—C4—C3    | 120.74 (11) |
| C9—N5—Fe1   | 126.79 (10) | C5—C4—C3    | 118.52 (17) |
| C9—N5—C13   | 118.52 (13) | C5—C4—H2    | 120.74 (10) |
| C19—N6—Fe1  | 114.68 (9)  | C4—C5—C6    | 119.09 (17) |
| C15—N6—Fe1  | 126.72 (11) | H3—C5—C6    | 120.45 (11) |
| C15—N6—C19  | 118.60 (13) | H3—C5—C4    | 120.45 (10) |
| C1—N1—Fe1   | 166.46 (15) | C26—C21—Br3 | 117.9 (3)   |
| C8—C7—N3    | 118.71 (12) | C22—C21—Br3 | 122.1 (3)   |
| C6—C7—N3    | 121.43 (14) | C22—C21—C26 | 120.0       |
| C6—C7—C8    | 119.83 (14) | H28—C26—C21 | 120.0       |
| C20—C19—N6  | 115.98 (12) | C25—C26—C21 | 120.0       |
| C18—C19—N6  | 122.17 (14) | C25—C26—H28 | 120.0       |
| C18—C19—C20 | 121.78 (14) | C22—C23—H22 | 120.00 (18) |
| C2—N2—Fe1   | 172.81 (15) | C24—C23—H22 | 120.00 (18) |
| C14—C13—N5  | 116.08 (12) | C24—C23—C22 | 120.0       |
| C12—C13—N5  | 122.00 (14) | C23—C22—C21 | 120.0       |
| C12—C13—C14 | 121.85 (14) | H20—C22—C21 | 120.00 (18) |
| C13—C14—N4  | 111.29 (11) | H20—C22—C23 | 120.00 (18) |
| H12—C14—N4  | 109.37 (8)  | H24—C24—C23 | 120.00 (18) |
| H12—C14—C13 | 109.37 (8)  | C25—C24—C23 | 120.0       |
| H11—C14—N4  | 109.37 (8)  | C25—C24—H24 | 120.00 (18) |
| H11—C14—C13 | 109.37 (8)  | C24—C25—C26 | 120.0       |

|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| H11—C14—H12     | 108.0        | H26—C25—C26         | 120.00 (18)  |
| C7—C8—N4        | 115.73 (12)  | H26—C25—C24         | 120.00 (18)  |
| H5—C8—N4        | 108.35 (8)   | C21a—C22a—H19       | 120.0 (3)    |
| H5—C8—C7        | 108.35 (8)   | C23a—C22a—H19       | 120.0 (3)    |
| H6—C8—N4        | 108.35 (8)   | C23a—C22a—C21a      | 120.0        |
| H6—C8—C7        | 108.35 (8)   | Br3a—C21a—C22a      | 114.5 (6)    |
| H6—C8—H5        | 107.4        | C26a—C21a—C22a      | 120.0        |
| H7—C9—N5        | 118.59 (9)   | C26a—C21a—Br3a      | 125.4 (6)    |
| C10—C9—N5       | 122.82 (15)  | H27—C26a—C21a       | 120.0 (3)    |
| C10—C9—H7       | 118.59 (10)  | C25a—C26a—C21a      | 120.0        |
| C19—C20—N4      | 111.20 (11)  | C25a—C26a—H27       | 120.0 (3)    |
| H17—C20—N4      | 109.39 (8)   | H25—C25a—C26a       | 120.0 (3)    |
| H17—C20—C19     | 109.39 (8)   | C24a—C25a—C26a      | 120.0        |
| H18—C20—N4      | 109.39 (8)   | C24a—C25a—H25       | 120.0 (3)    |
| H18—C20—C19     | 109.39 (8)   | H21—C23a—C22a       | 120.0 (3)    |
| H18—C20—H17     | 108.0        | C24a—C23a—C22a      | 120.0        |
| H16—C18—C19     | 120.61 (10)  | C24a—C23a—H21       | 120.0 (3)    |
| C17—C18—C19     | 118.78 (16)  | C23a—C24a—C25a      | 120.0        |
| C17—C18—H16     | 120.61 (11)  | H23—C24a—C25a       | 120.0        |
| N2—C2—S2        | 179.72 (16)  | H23—C24a—C23a       | 120.0        |
| H10—C12—C13     | 120.52 (10)  |                     |              |
| Fe1—N4—C14—C13  | −43.38 (9)   | C19—C20—N4—C8       | −76.35 (14)  |
| Fe1—N4—C8—C7    | −6.42 (9)    | C19—C18—C17—C16     | 1.93 (19)    |
| Fe1—N4—C20—C19  | 44.65 (9)    | C13—N5—C9—C10       | −0.49 (15)   |
| Fe1—N3—C7—C8    | −6.70 (11)   | C13—C14—N4—C8       | 77.61 (14)   |
| Fe1—N3—C7—C6    | 175.38 (11)  | C13—C14—N4—C20      | −157.19 (12) |
| Fe1—N3—C3—C4    | −174.82 (14) | C13—C12—C11—C10     | −0.39 (19)   |
| Fe1—N5—C13—C14  | −4.50 (10)   | C14—C13—N5—C9       | 176.85 (13)  |
| Fe1—N5—C13—C12  | 178.58 (10)  | C14—C13—C12—C11     | −176.23 (16) |
| Fe1—N5—C9—C10   | −178.96 (12) | C8—C7—N3—C3         | 178.09 (14)  |
| Fe1—N6—C19—C20  | 2.17 (11)    | C8—C7—C6—C5         | −178.07 (16) |
| Fe1—N6—C19—C18  | 179.20 (10)  | C9—N5—C13—C12       | −0.08 (15)   |
| Fe1—N6—C15—C16  | −178.23 (14) | C9—C10—C11—C12      | −0.13 (19)   |
| Br3—C21—C26—C25 | −179.15 (12) | C20—C19—N6—C15      | −177.79 (13) |
| Br3—C21—C22—C23 | 179.11 (13)  | C20—C19—C18—C17     | 175.80 (16)  |
| N4—C14—C13—N5   | 33.62 (14)   | C18—C19—N6—C15      | −0.77 (18)   |
| N4—C14—C13—C12  | −149.45 (12) | C18—C17—C16—C15     | −1.0 (2)     |
| N4—C8—C7—N3     | 8.99 (16)    | C3—N3—C7—C6         | 0.17 (17)    |
| N4—C8—C7—C6     | −173.06 (13) | C3—C4—C5—C6         | 0.0 (2)      |
| N4—C20—C19—N6   | −32.85 (15)  | C21—C26—C25—C24     | −0.0 (5)     |
| N4—C20—C19—C18  | 150.11 (12)  | C21—C22—C23—C24     | −0.0 (4)     |
| N3—C7—C6—C5     | −0.17 (17)   | C26—C21—C22—C23     | 0.0 (4)      |
| N3—C3—C4—C5     | −0.0 (2)     | C26—C25—C24—C23     | 0.0 (4)      |
| N5—C13—C12—C11  | 0.52 (17)    | C22—C21—C26—C25     | −0.0 (4)     |
| N5—C9—C10—C11   | 0.59 (18)    | C22—C23—C24—C25     | −0.0 (5)     |
| N6—C19—C18—C17  | −1.05 (17)   | C22a—C21a—C26a—C25a | −0.0 (7)     |
| N6—C15—C16—C17  | −0.8 (2)     | C22a—C23a—C24a—C25a | 0.0 (7)      |

|                |              |                     |             |
|----------------|--------------|---------------------|-------------|
| C7—N3—C3—C4    | −0.09 (17)   | Br3a—C21a—C22a—C23a | 178.32 (19) |
| C7—C8—N4—C14   | −124.35 (13) | Br3a—C21a—C26a—C25a | −178.1 (2)  |
| C7—C8—N4—C20   | 111.34 (13)  | C21a—C22a—C23a—C24a | −0.0 (7)    |
| C7—C6—C5—C4    | 0.1 (2)      | C21a—C26a—C25a—C24a | 0.0 (7)     |
| C19—N6—C15—C16 | 1.73 (16)    | C26a—C21a—C22a—C23a | −0.0 (7)    |
| C19—C20—N4—C14 | 158.70 (13)  | C26a—C25a—C24a—C23a | −0.0 (7)    |

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) chlorobenzene monosolvate (cl\_100K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.3851$  (2) Å

$b = 12.6621$  (2) Å

$c = 15.2251$  (3) Å

$\beta = 110.094$  (2)°

$V = 2604.38$  (8) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.466$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 53306 reflections

$\theta = 2.0$ – $32.3^\circ$

$\mu = 0.87$  mm<sup>−1</sup>

$T = 100$  K

Block, red

$0.31 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.584$ ,  $T_{\max} = 1.000$

76006 measured reflections

6471 independent reflections

5904 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.065$

$S = 1.03$

6471 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.6817P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.85$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.57$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | $x$           | $y$           | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|--------------|----------------------------------|
| Fe1 | 0.339657 (10) | 0.337647 (11) | 0.680971 (9) | 0.01526 (5)                      |
| S2  | 0.52451 (2)   | 0.04013 (2)   | 0.83124 (2)  | 0.02399 (7)                      |
| S1  | 0.25347 (2)   | 0.30185 (2)   | 0.95375 (2)  | 0.02801 (7)                      |
| Cl1 | 0.35423 (3)   | 0.03585 (3)   | 0.44376 (2)  | 0.03973 (8)                      |
| N3  | 0.39006 (6)   | 0.34408 (7)   | 0.57653 (6)  | 0.01727 (17)                     |
| N4  | 0.24821 (6)   | 0.45289 (7)   | 0.61433 (6)  | 0.01640 (16)                     |
| N5  | 0.22997 (6)   | 0.24359 (7)   | 0.60791 (6)  | 0.01709 (17)                     |

|     |              |              |              |              |
|-----|--------------|--------------|--------------|--------------|
| N1  | 0.28910 (7)  | 0.33614 (7)  | 0.78587 (6)  | 0.01979 (18) |
| N6  | 0.42656 (6)  | 0.45760 (7)  | 0.73939 (6)  | 0.01770 (17) |
| N2  | 0.42783 (7)  | 0.22405 (7)  | 0.74341 (6)  | 0.02073 (18) |
| C19 | 0.37776 (8)  | 0.55069 (8)  | 0.72989 (7)  | 0.01796 (19) |
| C13 | 0.14412 (8)  | 0.29614 (8)  | 0.56644 (7)  | 0.01778 (19) |
| C7  | 0.34986 (7)  | 0.42038 (8)  | 0.51333 (7)  | 0.01729 (19) |
| C15 | 0.52538 (8)  | 0.45682 (9)  | 0.78238 (7)  | 0.0214 (2)   |
| C2  | 0.46841 (8)  | 0.14792 (8)  | 0.78067 (7)  | 0.01852 (19) |
| C14 | 0.14611 (7)  | 0.41102 (8)  | 0.59136 (7)  | 0.01941 (19) |
| C12 | 0.05857 (8)  | 0.24586 (8)  | 0.50935 (7)  | 0.0208 (2)   |
| C20 | 0.26770 (8)  | 0.54266 (8)  | 0.68153 (7)  | 0.0191 (2)   |
| C3  | 0.46174 (8)  | 0.28274 (9)  | 0.56497 (7)  | 0.0215 (2)   |
| C9  | 0.23209 (8)  | 0.13888 (8)  | 0.59368 (7)  | 0.0199 (2)   |
| C1  | 0.27432 (8)  | 0.32273 (8)  | 0.85592 (7)  | 0.0191 (2)   |
| C10 | 0.14924 (9)  | 0.08299 (9)  | 0.53864 (7)  | 0.0230 (2)   |
| C18 | 0.42598 (8)  | 0.64472 (9)  | 0.76487 (7)  | 0.0214 (2)   |
| C8  | 0.26858 (8)  | 0.48455 (8)  | 0.52785 (7)  | 0.0199 (2)   |
| C4  | 0.49592 (8)  | 0.29605 (10) | 0.49089 (8)  | 0.0255 (2)   |
| C11 | 0.06098 (9)  | 0.13712 (9)  | 0.49602 (7)  | 0.0229 (2)   |
| C16 | 0.57835 (8)  | 0.54753 (9)  | 0.82042 (7)  | 0.0247 (2)   |
| C6  | 0.38072 (8)  | 0.43860 (9)  | 0.43728 (7)  | 0.0231 (2)   |
| C17 | 0.52785 (8)  | 0.64297 (9)  | 0.81239 (8)  | 0.0244 (2)   |
| C5  | 0.45492 (9)  | 0.37591 (10) | 0.42601 (8)  | 0.0265 (2)   |
| C22 | 0.24601 (9)  | 0.20546 (10) | 0.35870 (9)  | 0.0276 (2)   |
| C21 | 0.30421 (9)  | 0.12179 (10) | 0.34998 (9)  | 0.0271 (2)   |
| C23 | 0.20719 (9)  | 0.27423 (11) | 0.28313 (10) | 0.0324 (3)   |
| C26 | 0.32414 (11) | 0.10448 (11) | 0.26842 (10) | 0.0359 (3)   |
| C24 | 0.22612 (10) | 0.25807 (12) | 0.20066 (9)  | 0.0370 (3)   |
| C25 | 0.28446 (12) | 0.17281 (12) | 0.19368 (10) | 0.0401 (3)   |
| H23 | 0.37081 (11) | 0.03743 (11) | 0.26298 (10) | 0.0430 (3)*  |
| H22 | 0.29922 (12) | 0.15956 (12) | 0.12807 (10) | 0.0481 (4)*  |
| H21 | 0.19519 (10) | 0.31235 (12) | 0.14105 (9)  | 0.0444 (4)*  |
| H20 | 0.16125 (9)  | 0.34175 (11) | 0.28903 (10) | 0.0389 (3)*  |
| H19 | 0.23058 (9)  | 0.21760 (10) | 0.42411 (9)  | 0.0332 (3)*  |
| H1  | 0.49433 (8)  | 0.21971 (9)  | 0.61634 (7)  | 0.0259 (2)*  |
| H2  | 0.55449 (8)  | 0.24439 (10) | 0.48341 (8)  | 0.0306 (3)*  |
| H3  | 0.48106 (9)  | 0.38901 (10) | 0.36673 (8)  | 0.0318 (3)*  |
| H4  | 0.34660 (8)  | 0.50196 (9)  | 0.38677 (7)  | 0.0278 (3)*  |
| H13 | 0.30204 (8)  | 0.09575 (8)  | 0.62684 (7)  | 0.0239 (2)*  |
| H14 | 0.15348 (9)  | −0.00297 (9) | 0.52892 (7)  | 0.0276 (3)*  |
| H15 | −0.00594 (9) | 0.09469 (9)  | 0.45244 (7)  | 0.0275 (3)*  |
| H16 | −0.00983 (8) | 0.29126 (8)  | 0.47531 (7)  | 0.0249 (2)*  |
| H7  | 0.56572 (8)  | 0.38167 (9)  | 0.78762 (7)  | 0.0257 (2)*  |
| H8  | 0.65918 (8)  | 0.54403 (9)  | 0.85643 (7)  | 0.0297 (3)*  |
| H9  | 0.56781 (8)  | 0.71587 (9)  | 0.84312 (8)  | 0.0292 (3)*  |
| H10 | 0.38438 (8)  | 0.71953 (9)  | 0.75521 (7)  | 0.0257 (2)*  |
| H18 | 0.12135 (7)  | 0.42098 (8)  | 0.65241 (7)  | 0.0233 (2)*  |
| H17 | 0.09464 (7)  | 0.45576 (8)  | 0.53158 (7)  | 0.0233 (2)*  |

|     |             |             |             |             |
|-----|-------------|-------------|-------------|-------------|
| H11 | 0.23842 (8) | 0.61725 (8) | 0.64371 (7) | 0.0229 (2)* |
| H12 | 0.23063 (8) | 0.52860 (8) | 0.73358 (7) | 0.0229 (2)* |
| H5  | 0.19992 (8) | 0.47488 (8) | 0.46621 (7) | 0.0239 (2)* |
| H6  | 0.28951 (8) | 0.56938 (8) | 0.53295 (7) | 0.0239 (2)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.01728 (8)  | 0.01698 (8)  | 0.01154 (7)  | 0.00202 (5)   | 0.00498 (6)  | 0.00064 (5)   |
| S2  | 0.02267 (13) | 0.02268 (13) | 0.02686 (14) | 0.00427 (10)  | 0.00884 (11) | 0.00739 (10)  |
| S1  | 0.04330 (17) | 0.02667 (15) | 0.01915 (13) | −0.00887 (12) | 0.01724 (12) | −0.00393 (10) |
| Cl1 | 0.04487 (19) | 0.03195 (16) | 0.03965 (18) | 0.00139 (13)  | 0.01100 (14) | 0.00237 (13)  |
| N3  | 0.0183 (4)   | 0.0205 (4)   | 0.0134 (4)   | 0.0015 (3)    | 0.0060 (3)   | 0.0004 (3)    |
| N4  | 0.0174 (4)   | 0.0164 (4)   | 0.0142 (4)   | 0.0016 (3)    | 0.0040 (3)   | −0.0004 (3)   |
| N5  | 0.0205 (4)   | 0.0170 (4)   | 0.0141 (4)   | 0.0009 (3)    | 0.0064 (3)   | −0.0000 (3)   |
| N1  | 0.0243 (4)   | 0.0211 (4)   | 0.0156 (4)   | −0.0004 (3)   | 0.0089 (3)   | −0.0002 (3)   |
| N6  | 0.0190 (4)   | 0.0202 (4)   | 0.0129 (4)   | 0.0006 (3)    | 0.0042 (3)   | 0.0007 (3)    |
| N2  | 0.0224 (4)   | 0.0221 (4)   | 0.0166 (4)   | 0.0046 (3)    | 0.0053 (3)   | 0.0033 (3)    |
| C19 | 0.0193 (5)   | 0.0198 (5)   | 0.0142 (4)   | −0.0004 (4)   | 0.0051 (4)   | −0.0011 (4)   |
| C13 | 0.0191 (5)   | 0.0186 (5)   | 0.0149 (4)   | −0.0006 (4)   | 0.0049 (4)   | −0.0009 (4)   |
| C7  | 0.0188 (4)   | 0.0202 (5)   | 0.0126 (4)   | −0.0008 (4)   | 0.0050 (4)   | 0.0005 (4)    |
| C15 | 0.0195 (5)   | 0.0260 (5)   | 0.0165 (4)   | 0.0011 (4)    | 0.0034 (4)   | 0.0026 (4)    |
| C2  | 0.0188 (5)   | 0.0207 (5)   | 0.0160 (4)   | 0.0023 (4)    | 0.0059 (4)   | 0.0024 (4)    |
| C14 | 0.0185 (4)   | 0.0187 (5)   | 0.0202 (5)   | 0.0021 (4)    | 0.0056 (4)   | −0.0013 (4)   |
| C12 | 0.0212 (5)   | 0.0214 (5)   | 0.0182 (5)   | −0.0026 (4)   | 0.0049 (4)   | −0.0005 (4)   |
| C20 | 0.0203 (5)   | 0.0185 (5)   | 0.0175 (5)   | 0.0015 (4)    | 0.0053 (4)   | −0.0020 (4)   |
| C3  | 0.0218 (5)   | 0.0268 (5)   | 0.0173 (5)   | 0.0052 (4)    | 0.0083 (4)   | 0.0012 (4)    |
| C9  | 0.0263 (5)   | 0.0177 (5)   | 0.0167 (5)   | 0.0015 (4)    | 0.0088 (4)   | 0.0006 (4)    |
| C1  | 0.0249 (5)   | 0.0185 (4)   | 0.0152 (5)   | −0.0019 (4)   | 0.0086 (4)   | −0.0019 (4)   |
| C10 | 0.0317 (6)   | 0.0186 (5)   | 0.0190 (5)   | −0.0022 (4)   | 0.0092 (4)   | −0.0014 (4)   |
| C18 | 0.0238 (5)   | 0.0214 (5)   | 0.0189 (5)   | −0.0028 (4)   | 0.0071 (4)   | −0.0038 (4)   |
| C8  | 0.0227 (5)   | 0.0207 (5)   | 0.0153 (4)   | 0.0028 (4)    | 0.0051 (4)   | 0.0031 (4)    |
| C4  | 0.0233 (5)   | 0.0356 (6)   | 0.0205 (5)   | 0.0037 (5)    | 0.0113 (4)   | −0.0007 (4)   |
| C11 | 0.0271 (5)   | 0.0217 (5)   | 0.0194 (5)   | −0.0051 (4)   | 0.0071 (4)   | −0.0021 (4)   |
| C16 | 0.0207 (5)   | 0.0316 (6)   | 0.0189 (5)   | −0.0033 (4)   | 0.0029 (4)   | 0.0011 (4)    |
| C6  | 0.0257 (5)   | 0.0289 (6)   | 0.0155 (5)   | −0.0011 (4)   | 0.0082 (4)   | 0.0032 (4)    |
| C17 | 0.0237 (5)   | 0.0275 (6)   | 0.0205 (5)   | −0.0061 (4)   | 0.0057 (4)   | −0.0039 (4)   |
| C5  | 0.0272 (5)   | 0.0379 (6)   | 0.0177 (5)   | −0.0004 (5)   | 0.0121 (4)   | 0.0013 (4)    |
| C22 | 0.0283 (6)   | 0.0301 (6)   | 0.0282 (6)   | −0.0028 (5)   | 0.0143 (5)   | −0.0063 (5)   |
| C21 | 0.0280 (5)   | 0.0260 (6)   | 0.0297 (6)   | −0.0039 (4)   | 0.0130 (5)   | −0.0054 (5)   |
| C23 | 0.0245 (6)   | 0.0320 (6)   | 0.0400 (7)   | −0.0012 (5)   | 0.0101 (5)   | −0.0011 (5)   |
| C26 | 0.0453 (7)   | 0.0308 (6)   | 0.0418 (7)   | −0.0061 (6)   | 0.0282 (6)   | −0.0108 (6)   |
| C24 | 0.0365 (7)   | 0.0412 (7)   | 0.0278 (6)   | −0.0139 (6)   | 0.0042 (5)   | 0.0024 (5)    |
| C25 | 0.0571 (9)   | 0.0403 (7)   | 0.0303 (7)   | −0.0174 (6)   | 0.0245 (6)   | −0.0105 (6)   |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N3    | 1.9629 (9)  | C20—H11     | 1.1110      |
| Fe1—N4    | 1.9930 (9)  | C20—H12     | 1.1110      |
| Fe1—N5    | 1.9848 (9)  | C3—C4       | 1.3867 (15) |
| Fe1—N1    | 1.9692 (9)  | C3—H1       | 1.1030      |
| Fe1—N6    | 1.9741 (9)  | C9—C10      | 1.3918 (15) |
| Fe1—N2    | 1.9372 (9)  | C9—H13      | 1.1030      |
| S2—C2     | 1.6371 (11) | C10—C11     | 1.3917 (16) |
| S1—C1     | 1.6387 (11) | C10—H14     | 1.1030      |
| Cl1—C21   | 1.7421 (13) | C18—C17     | 1.3939 (15) |
| N3—C7     | 1.3458 (13) | C18—H10     | 1.1030      |
| N3—C3     | 1.3498 (13) | C8—H5       | 1.1110      |
| N4—C14    | 1.4857 (13) | C8—H6       | 1.1110      |
| N4—C20    | 1.4900 (12) | C4—C5       | 1.3950 (17) |
| N4—C8     | 1.4984 (13) | C4—H2       | 1.1030      |
| N5—C13    | 1.3530 (13) | C11—H15     | 1.1030      |
| N5—C9     | 1.3456 (13) | C16—C17     | 1.3935 (17) |
| N1—C1     | 1.1694 (14) | C16—H8      | 1.1030      |
| N6—C19    | 1.3541 (13) | C6—C5       | 1.3873 (16) |
| N6—C15    | 1.3453 (13) | C6—H4       | 1.1030      |
| N2—C2     | 1.1680 (14) | C17—H9      | 1.1030      |
| C19—C20   | 1.5027 (14) | C5—H3       | 1.1030      |
| C19—C18   | 1.3887 (14) | C22—C21     | 1.3847 (17) |
| C13—C14   | 1.5011 (14) | C22—C23     | 1.3973 (18) |
| C13—C12   | 1.3930 (14) | C22—H19     | 1.1030      |
| C7—C8     | 1.5013 (14) | C21—C26     | 1.3844 (17) |
| C7—C6     | 1.3944 (14) | C23—C24     | 1.3883 (19) |
| C15—C16   | 1.3900 (16) | C23—H20     | 1.1030      |
| C15—H7    | 1.1030      | C26—C25     | 1.387 (2)   |
| C14—H18   | 1.1110      | C26—H23     | 1.1030      |
| C14—H17   | 1.1110      | C24—C25     | 1.394 (2)   |
| C12—C11   | 1.3940 (15) | C24—H21     | 1.1030      |
| C12—H16   | 1.1030      | C25—H22     | 1.1030      |
| N4—Fe1—N3 | 85.42 (4)   | H12—C20—C19 | 110.14 (5)  |
| N5—Fe1—N3 | 90.84 (3)   | H12—C20—H11 | 108.5       |
| N5—Fe1—N4 | 84.00 (4)   | C4—C3—N3    | 122.43 (10) |
| N1—Fe1—N3 | 178.18 (4)  | H1—C3—N3    | 118.78 (6)  |
| N1—Fe1—N4 | 93.26 (4)   | H1—C3—C4    | 118.78 (7)  |
| N1—Fe1—N5 | 90.27 (4)   | C10—C9—N5   | 122.40 (10) |
| N6—Fe1—N3 | 88.86 (3)   | H13—C9—N5   | 118.80 (6)  |
| N6—Fe1—N4 | 82.59 (4)   | H13—C9—C10  | 118.80 (6)  |
| N6—Fe1—N5 | 166.57 (4)  | N1—C1—S1    | 179.06 (10) |
| N6—Fe1—N1 | 89.73 (4)   | C11—C10—C9  | 119.06 (10) |
| N2—Fe1—N3 | 93.81 (4)   | H14—C10—C9  | 120.47 (6)  |
| N2—Fe1—N4 | 178.85 (4)  | H14—C10—C11 | 120.47 (6)  |
| N2—Fe1—N5 | 95.16 (4)   | C17—C18—C19 | 118.88 (10) |



|              |             |                 |             |
|--------------|-------------|-----------------|-------------|
| N2—Fe1—N1    | 87.52 (4)   | H10—C18—C19     | 120.56 (6)  |
| N2—Fe1—N6    | 98.26 (4)   | H10—C18—C17     | 120.56 (7)  |
| C7—N3—Fe1    | 114.92 (7)  | C7—C8—N4        | 112.45 (8)  |
| C3—N3—Fe1    | 126.37 (7)  | H5—C8—N4        | 109.11 (5)  |
| C3—N3—C7     | 118.66 (9)  | H5—C8—C7        | 109.11 (5)  |
| C14—N4—Fe1   | 106.55 (6)  | H6—C8—N4        | 109.11 (5)  |
| C20—N4—Fe1   | 105.87 (6)  | H6—C8—C7        | 109.11 (6)  |
| C20—N4—C14   | 111.80 (8)  | H6—C8—H5        | 107.8       |
| C8—N4—Fe1    | 110.43 (6)  | C5—C4—C3        | 118.83 (10) |
| C8—N4—C14    | 111.37 (8)  | H2—C4—C3        | 120.59 (7)  |
| C8—N4—C20    | 110.60 (8)  | H2—C4—C5        | 120.59 (6)  |
| C13—N5—Fe1   | 112.89 (7)  | C10—C11—C12     | 118.88 (10) |
| C9—N5—Fe1    | 128.60 (7)  | H15—C11—C12     | 120.56 (7)  |
| C9—N5—C13    | 118.49 (9)  | H15—C11—C10     | 120.56 (6)  |
| C1—N1—Fe1    | 167.07 (9)  | C17—C16—C15     | 119.04 (10) |
| C19—N6—Fe1   | 113.38 (7)  | H8—C16—C15      | 120.48 (7)  |
| C15—N6—Fe1   | 127.83 (8)  | H8—C16—C17      | 120.48 (6)  |
| C15—N6—C19   | 118.74 (9)  | C5—C6—C7        | 119.08 (10) |
| C2—N2—Fe1    | 170.00 (9)  | H4—C6—C7        | 120.46 (6)  |
| C20—C19—N6   | 114.44 (9)  | H4—C6—C5        | 120.46 (6)  |
| C18—C19—N6   | 122.21 (9)  | C16—C17—C18     | 118.86 (10) |
| C18—C19—C20  | 123.33 (9)  | H9—C17—C18      | 120.57 (7)  |
| C14—C13—N5   | 115.06 (9)  | H9—C17—C16      | 120.57 (6)  |
| C12—C13—N5   | 122.38 (10) | C6—C5—C4        | 118.90 (10) |
| C12—C13—C14  | 122.45 (9)  | H3—C5—C4        | 120.55 (6)  |
| C8—C7—N3     | 116.64 (9)  | H3—C5—C6        | 120.55 (6)  |
| C6—C7—N3     | 122.09 (10) | C23—C22—C21     | 118.78 (11) |
| C6—C7—C8     | 121.26 (9)  | H19—C22—C21     | 120.61 (7)  |
| C16—C15—N6   | 122.20 (10) | H19—C22—C23     | 120.61 (7)  |
| H7—C15—N6    | 118.90 (6)  | C22—C21—C11     | 119.50 (9)  |
| H7—C15—C16   | 118.90 (7)  | C26—C21—C11     | 118.80 (10) |
| N2—C2—S2     | 179.04 (10) | C26—C21—C22     | 121.70 (12) |
| C13—C14—N4   | 109.68 (8)  | C24—C23—C22     | 120.38 (12) |
| H18—C14—N4   | 109.73 (5)  | H20—C23—C22     | 119.81 (7)  |
| H18—C14—C13  | 109.73 (5)  | H20—C23—C24     | 119.81 (8)  |
| H17—C14—N4   | 109.73 (5)  | C25—C26—C21     | 118.92 (13) |
| H17—C14—C13  | 109.73 (5)  | H23—C26—C21     | 120.54 (8)  |
| H17—C14—H18  | 108.2       | H23—C26—C25     | 120.54 (8)  |
| C11—C12—C13  | 118.77 (10) | C25—C24—C23     | 119.57 (13) |
| H16—C12—C13  | 120.61 (6)  | H21—C24—C23     | 120.21 (8)  |
| H16—C12—C11  | 120.61 (7)  | H21—C24—C25     | 120.21 (8)  |
| C19—C20—N4   | 107.83 (8)  | C24—C25—C26     | 120.64 (12) |
| H11—C20—N4   | 110.14 (5)  | H22—C25—C26     | 119.68 (8)  |
| H11—C20—C19  | 110.14 (6)  | H22—C25—C24     | 119.68 (8)  |
| H12—C20—N4   | 110.14 (5)  |                 |             |
| Fe1—N3—C7—C8 | −3.78 (8)   | C19—C20—N4—C8   | −77.93 (9)  |
| Fe1—N3—C7—C6 | 177.07 (7)  | C19—C18—C17—C16 | 2.15 (12)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Fe1—N3—C3—C4    | −177.00 (9)  | C13—N5—C9—C10   | −0.49 (11)   |
| Fe1—N4—C14—C13  | −36.57 (6)   | C13—C14—N4—C20  | −151.79 (8)  |
| Fe1—N4—C20—C19  | 41.71 (6)    | C13—C14—N4—C8   | 83.91 (9)    |
| Fe1—N4—C8—C7    | 1.23 (7)     | C13—C12—C11—C10 | −1.31 (11)   |
| Fe1—N5—C13—C14  | −5.83 (7)    | C7—N3—C3—C4     | 0.53 (11)    |
| Fe1—N5—C13—C12  | 177.88 (7)   | C7—C8—N4—C14    | −116.95 (9)  |
| Fe1—N5—C9—C10   | −178.52 (8)  | C7—C8—N4—C20    | 118.08 (9)   |
| Fe1—N6—C19—C20  | 2.93 (7)     | C7—C6—C5—C4     | 0.43 (12)    |
| Fe1—N6—C19—C18  | −178.97 (7)  | C15—N6—C19—C20  | −179.60 (9)  |
| Fe1—N6—C15—C16  | 179.58 (9)   | C15—N6—C19—C18  | −1.49 (11)   |
| Cl1—C21—C22—C23 | 179.42 (9)   | C15—C16—C17—C18 | −1.20 (12)   |
| Cl1—C21—C26—C25 | −179.96 (10) | C14—C13—N5—C9   | 175.84 (8)   |
| N3—C7—C8—N4     | 1.61 (10)    | C14—C13—C12—C11 | −174.67 (10) |
| N3—C7—C6—C5     | 0.26 (12)    | C12—C13—N5—C9   | −0.45 (12)   |
| N3—C3—C4—C5     | 0.15 (13)    | C12—C11—C10—C9  | 0.43 (12)    |
| N4—C14—C13—N5   | 28.86 (9)    | C20—C19—C18—C17 | 177.10 (10)  |
| N4—C14—C13—C12  | −154.85 (8)  | C3—N3—C7—C8     | 178.41 (9)   |
| N4—C20—C19—N6   | −30.20 (9)   | C3—N3—C7—C6     | −0.74 (11)   |
| N4—C20—C19—C18  | 151.72 (8)   | C3—C4—C5—C6     | −0.63 (13)   |
| N4—C8—C7—C6     | −179.22 (8)  | C8—C7—C6—C5     | −178.85 (10) |
| N5—C13—C12—C11  | 1.35 (11)    | C22—C21—C26—C25 | −0.12 (14)   |
| N5—C9—C10—C11   | 0.49 (12)    | C22—C23—C24—C25 | −0.19 (14)   |
| N6—C19—C18—C17  | −0.84 (12)   | C21—C22—C23—C24 | 0.57 (14)    |
| N6—C15—C16—C17  | −1.18 (12)   | C21—C26—C25—C24 | 0.52 (15)    |
| C19—N6—C15—C16  | 2.52 (11)    | C23—C22—C21—C26 | −0.41 (14)   |
| C19—C20—N4—C14  | 157.34 (8)   | C23—C24—C25—C26 | −0.36 (14)   |

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) chlorobenzene monosolvate (cl\_110K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.3918$  (3) Å

$b = 12.6666$  (2) Å

$c = 15.2318$  (3) Å

$\beta = 110.085$  (2)°

$V = 2607.82$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.464$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 52338 reflections

$\theta = 2.0$ – $32.2^\circ$

$\mu = 0.87$  mm<sup>−1</sup>

$T = 110$  K

Block, red

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.556$ ,  $T_{\max} = 1.000$

75361 measured reflections

6480 independent reflections

5886 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.026$  $wR(F^2) = 0.069$  $S = 1.03$ 

6480 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 0.7833P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.95 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.339596 (11) | 0.337606 (12) | 0.680991 (10) | 0.01623 (5)                      |
| S2  | 0.52449 (2)   | 0.04036 (2)   | 0.83124 (2)   | 0.02548 (7)                      |
| S1  | 0.25354 (3)   | 0.30190 (3)   | 0.95370 (2)   | 0.03029 (8)                      |
| Cl1 | 0.35423 (3)   | 0.03589 (3)   | 0.44354 (3)   | 0.04325 (9)                      |
| N4  | 0.24816 (7)   | 0.45280 (7)   | 0.61436 (6)   | 0.01738 (17)                     |
| N3  | 0.39008 (7)   | 0.34406 (7)   | 0.57662 (6)   | 0.01846 (18)                     |
| N5  | 0.22995 (7)   | 0.24351 (7)   | 0.60799 (6)   | 0.01820 (18)                     |
| N1  | 0.28902 (7)   | 0.33611 (7)   | 0.78587 (7)   | 0.02073 (19)                     |
| N6  | 0.42649 (7)   | 0.45756 (7)   | 0.73932 (6)   | 0.01873 (18)                     |
| N2  | 0.42776 (7)   | 0.22403 (8)   | 0.74340 (7)   | 0.02207 (19)                     |
| C13 | 0.14415 (8)   | 0.29607 (9)   | 0.56653 (7)   | 0.0188 (2)                       |
| C19 | 0.37767 (8)   | 0.55058 (9)   | 0.72984 (7)   | 0.0193 (2)                       |
| C7  | 0.34981 (8)   | 0.42044 (9)   | 0.51342 (7)   | 0.0186 (2)                       |
| C20 | 0.26765 (8)   | 0.54259 (9)   | 0.68147 (8)   | 0.0198 (2)                       |
| C15 | 0.52528 (8)   | 0.45672 (10)  | 0.78235 (8)   | 0.0229 (2)                       |
| C14 | 0.14623 (8)   | 0.41092 (9)   | 0.59156 (8)   | 0.0206 (2)                       |
| C12 | 0.05863 (8)   | 0.24586 (9)   | 0.50950 (8)   | 0.0219 (2)                       |
| C2  | 0.46838 (8)   | 0.14807 (9)   | 0.78064 (8)   | 0.0195 (2)                       |
| C1  | 0.27440 (8)   | 0.32269 (9)   | 0.85598 (8)   | 0.0204 (2)                       |
| C9  | 0.23213 (9)   | 0.13886 (9)   | 0.59374 (8)   | 0.0213 (2)                       |
| C3  | 0.46165 (8)   | 0.28281 (10)  | 0.56506 (8)   | 0.0233 (2)                       |
| C8  | 0.26852 (8)   | 0.48433 (9)   | 0.52801 (7)   | 0.0211 (2)                       |
| C10 | 0.14934 (9)   | 0.08296 (9)   | 0.53873 (8)   | 0.0246 (2)                       |
| C18 | 0.42599 (9)   | 0.64454 (9)   | 0.76471 (8)   | 0.0229 (2)                       |
| C11 | 0.06111 (9)   | 0.13725 (9)   | 0.49625 (8)   | 0.0245 (2)                       |
| C6  | 0.38069 (9)   | 0.43856 (10)  | 0.43736 (8)   | 0.0245 (2)                       |
| C16 | 0.57811 (9)   | 0.54743 (10)  | 0.82027 (8)   | 0.0266 (2)                       |
| C4  | 0.49580 (9)   | 0.29610 (11)  | 0.49105 (9)   | 0.0273 (2)                       |
| C17 | 0.52766 (9)   | 0.64281 (10)  | 0.81212 (8)   | 0.0262 (2)                       |
| C22 | 0.24614 (9)   | 0.20519 (11)  | 0.35862 (9)   | 0.0297 (3)                       |
| C5  | 0.45490 (9)   | 0.37596 (11)  | 0.42621 (8)   | 0.0288 (3)                       |
| C21 | 0.30439 (9)   | 0.12185 (10)  | 0.34977 (9)   | 0.0288 (3)                       |
| C23 | 0.20714 (10)  | 0.27400 (11)  | 0.28301 (11)  | 0.0347 (3)                       |
| C26 | 0.32409 (11)  | 0.10460 (12)  | 0.26828 (11)  | 0.0386 (3)                       |
| C24 | 0.22629 (11)  | 0.25776 (13)  | 0.20077 (10)  | 0.0402 (3)                       |
| C25 | 0.28441 (13)  | 0.17292 (13)  | 0.19370 (11)  | 0.0434 (4)                       |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| H1  | 0.49422 (8)  | 0.21980 (10) | 0.61640 (8)  | 0.0279 (3)* |
| H2  | 0.55431 (9)  | 0.24441 (11) | 0.48355 (9)  | 0.0327 (3)* |
| H3  | 0.48117 (9)  | 0.38911 (11) | 0.36705 (8)  | 0.0345 (3)* |
| H4  | 0.34654 (9)  | 0.50180 (10) | 0.38679 (8)  | 0.0294 (3)* |
| H13 | 0.30208 (9)  | 0.09579 (9)  | 0.62684 (8)  | 0.0256 (3)* |
| H14 | 0.15354 (9)  | −0.00296 (9) | 0.52895 (8)  | 0.0295 (3)* |
| H15 | −0.00581 (9) | 0.09485 (9)  | 0.45275 (8)  | 0.0295 (3)* |
| H16 | −0.00973 (8) | 0.29124 (9)  | 0.47550 (8)  | 0.0263 (3)* |
| H7  | 0.56561 (8)  | 0.38159 (10) | 0.78770 (8)  | 0.0274 (3)* |
| H8  | 0.65889 (9)  | 0.54396 (10) | 0.85633 (8)  | 0.0320 (3)* |
| H9  | 0.56761 (9)  | 0.71572 (10) | 0.84272 (8)  | 0.0314 (3)* |
| H10 | 0.38445 (9)  | 0.71936 (9)  | 0.75499 (8)  | 0.0274 (3)* |
| H11 | 0.23844 (8)  | 0.61715 (9)  | 0.64364 (8)  | 0.0237 (2)* |
| H12 | 0.23057 (8)  | 0.52862 (9)  | 0.73349 (8)  | 0.0237 (2)* |
| H18 | 0.12162 (8)  | 0.42078 (9)  | 0.65269 (8)  | 0.0247 (3)* |
| H17 | 0.09468 (8)  | 0.45569 (9)  | 0.53192 (8)  | 0.0247 (3)* |
| H5  | 0.19991 (8)  | 0.47454 (9)  | 0.46642 (7)  | 0.0253 (3)* |
| H6  | 0.28929 (8)  | 0.56917 (9)  | 0.53303 (7)  | 0.0253 (3)* |
| H23 | 0.37064 (11) | 0.03754 (12) | 0.26271 (11) | 0.0463 (4)* |
| H22 | 0.29914 (13) | 0.15979 (13) | 0.12810 (11) | 0.0521 (4)* |
| H21 | 0.19544 (11) | 0.31205 (13) | 0.14120 (10) | 0.0482 (4)* |
| H20 | 0.16103 (10) | 0.34136 (11) | 0.28881 (11) | 0.0416 (3)* |
| H19 | 0.23073 (9)  | 0.21717 (11) | 0.42403 (9)  | 0.0356 (3)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.01832 (8)  | 0.01809 (9)  | 0.01246 (8)  | 0.00226 (5)   | 0.00552 (6)  | 0.00073 (5)   |
| S2  | 0.02393 (14) | 0.02399 (14) | 0.02900 (15) | 0.00466 (10)  | 0.00970 (11) | 0.00799 (11)  |
| S1  | 0.04719 (19) | 0.02871 (16) | 0.02075 (14) | −0.00994 (13) | 0.01911 (13) | −0.00432 (11) |
| Cl1 | 0.0488 (2)   | 0.03462 (18) | 0.0433 (2)   | 0.00150 (15)  | 0.01197 (16) | 0.00272 (14)  |
| N4  | 0.0189 (4)   | 0.0171 (4)   | 0.0153 (4)   | 0.0018 (3)    | 0.0048 (3)   | −0.0003 (3)   |
| N3  | 0.0196 (4)   | 0.0217 (4)   | 0.0145 (4)   | 0.0021 (3)    | 0.0064 (3)   | 0.0005 (3)    |
| N5  | 0.0215 (4)   | 0.0184 (4)   | 0.0153 (4)   | 0.0012 (3)    | 0.0071 (3)   | 0.0001 (3)    |
| N1  | 0.0254 (5)   | 0.0220 (5)   | 0.0163 (4)   | −0.0001 (3)   | 0.0092 (4)   | −0.0002 (3)   |
| N6  | 0.0199 (4)   | 0.0215 (4)   | 0.0136 (4)   | 0.0007 (3)    | 0.0043 (3)   | 0.0008 (3)    |
| N2  | 0.0240 (5)   | 0.0234 (5)   | 0.0179 (4)   | 0.0049 (4)    | 0.0062 (4)   | 0.0031 (4)    |
| C13 | 0.0201 (5)   | 0.0191 (5)   | 0.0167 (5)   | −0.0006 (4)   | 0.0057 (4)   | −0.0008 (4)   |
| C19 | 0.0213 (5)   | 0.0210 (5)   | 0.0155 (5)   | −0.0002 (4)   | 0.0063 (4)   | −0.0012 (4)   |
| C7  | 0.0205 (5)   | 0.0216 (5)   | 0.0134 (4)   | −0.0012 (4)   | 0.0052 (4)   | 0.0005 (4)    |
| C20 | 0.0209 (5)   | 0.0190 (5)   | 0.0189 (5)   | 0.0012 (4)    | 0.0061 (4)   | −0.0023 (4)   |
| C15 | 0.0205 (5)   | 0.0281 (6)   | 0.0177 (5)   | 0.0008 (4)    | 0.0037 (4)   | 0.0023 (4)    |
| C14 | 0.0190 (5)   | 0.0206 (5)   | 0.0213 (5)   | 0.0022 (4)    | 0.0058 (4)   | −0.0016 (4)   |
| C12 | 0.0218 (5)   | 0.0232 (5)   | 0.0191 (5)   | −0.0028 (4)   | 0.0051 (4)   | −0.0005 (4)   |
| C2  | 0.0195 (5)   | 0.0219 (5)   | 0.0172 (5)   | 0.0022 (4)    | 0.0066 (4)   | 0.0024 (4)    |
| C1  | 0.0263 (5)   | 0.0201 (5)   | 0.0163 (5)   | −0.0024 (4)   | 0.0091 (4)   | −0.0022 (4)   |
| C9  | 0.0288 (6)   | 0.0189 (5)   | 0.0174 (5)   | 0.0016 (4)    | 0.0095 (4)   | 0.0006 (4)    |
| C3  | 0.0229 (5)   | 0.0290 (6)   | 0.0191 (5)   | 0.0057 (4)    | 0.0088 (4)   | 0.0011 (4)    |

|     |             |            |            |             |            |             |
|-----|-------------|------------|------------|-------------|------------|-------------|
| C8  | 0.0245 (5)  | 0.0214 (5) | 0.0168 (5) | 0.0031 (4)  | 0.0063 (4) | 0.0030 (4)  |
| C10 | 0.0344 (6)  | 0.0192 (5) | 0.0210 (5) | −0.0024 (5) | 0.0105 (5) | −0.0016 (4) |
| C18 | 0.0254 (6)  | 0.0224 (5) | 0.0201 (5) | −0.0026 (4) | 0.0071 (4) | −0.0033 (4) |
| C11 | 0.0285 (6)  | 0.0231 (6) | 0.0213 (5) | −0.0056 (5) | 0.0076 (4) | −0.0022 (4) |
| C6  | 0.0271 (6)  | 0.0306 (6) | 0.0169 (5) | −0.0014 (5) | 0.0089 (4) | 0.0035 (4)  |
| C16 | 0.0219 (5)  | 0.0341 (7) | 0.0207 (5) | −0.0030 (5) | 0.0031 (4) | 0.0015 (5)  |
| C4  | 0.0251 (5)  | 0.0377 (7) | 0.0224 (6) | 0.0041 (5)  | 0.0125 (5) | −0.0007 (5) |
| C17 | 0.0255 (6)  | 0.0295 (6) | 0.0219 (5) | −0.0070 (5) | 0.0061 (5) | −0.0041 (5) |
| C22 | 0.0304 (6)  | 0.0327 (7) | 0.0303 (6) | −0.0031 (5) | 0.0160 (5) | −0.0066 (5) |
| C5  | 0.0304 (6)  | 0.0404 (7) | 0.0197 (5) | −0.0007 (5) | 0.0139 (5) | 0.0009 (5)  |
| C21 | 0.0302 (6)  | 0.0278 (6) | 0.0312 (6) | −0.0044 (5) | 0.0139 (5) | −0.0063 (5) |
| C23 | 0.0251 (6)  | 0.0344 (7) | 0.0435 (8) | −0.0013 (5) | 0.0105 (5) | −0.0012 (6) |
| C26 | 0.0491 (8)  | 0.0326 (7) | 0.0457 (8) | −0.0072 (6) | 0.0313 (7) | −0.0126 (6) |
| C24 | 0.0393 (7)  | 0.0459 (8) | 0.0299 (7) | −0.0150 (6) | 0.0049 (6) | 0.0026 (6)  |
| C25 | 0.0618 (10) | 0.0440 (8) | 0.0328 (7) | −0.0185 (7) | 0.0270 (7) | −0.0112 (6) |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| Fe1—N4  | 1.9934 (9)  | C12—C11 | 1.3928 (16) |
| Fe1—N3  | 1.9635 (9)  | C12—H16 | 1.1030      |
| Fe1—N5  | 1.9854 (9)  | C9—C10  | 1.3919 (16) |
| Fe1—N1  | 1.9699 (10) | C9—H13  | 1.1030      |
| Fe1—N6  | 1.9745 (9)  | C3—C4   | 1.3862 (16) |
| Fe1—N2  | 1.9377 (10) | C3—H1   | 1.1030      |
| S2—C2   | 1.6371 (11) | C8—H5   | 1.1110      |
| S1—C1   | 1.6375 (11) | C8—H6   | 1.1110      |
| C11—C21 | 1.7425 (14) | C10—C11 | 1.3927 (17) |
| N4—C20  | 1.4901 (13) | C10—H14 | 1.1030      |
| N4—C14  | 1.4847 (13) | C18—C17 | 1.3920 (17) |
| N4—C8   | 1.4966 (13) | C18—H10 | 1.1030      |
| N3—C7   | 1.3472 (14) | C11—H15 | 1.1030      |
| N3—C3   | 1.3485 (14) | C6—C5   | 1.3867 (18) |
| N5—C13  | 1.3532 (14) | C6—H4   | 1.1030      |
| N5—C9   | 1.3453 (14) | C16—C17 | 1.3929 (18) |
| N1—C1   | 1.1697 (15) | C16—H8  | 1.1030      |
| N6—C19  | 1.3540 (14) | C4—C5   | 1.3949 (18) |
| N6—C15  | 1.3457 (14) | C4—H2   | 1.1030      |
| N2—C2   | 1.1668 (15) | C17—H9  | 1.1030      |
| C13—C14 | 1.5015 (15) | C22—C21 | 1.3831 (18) |
| C13—C12 | 1.3927 (15) | C22—C23 | 1.399 (2)   |
| C19—C20 | 1.5029 (15) | C22—H19 | 1.1030      |
| C19—C18 | 1.3887 (15) | C5—H3   | 1.1030      |
| C7—C8   | 1.5006 (15) | C21—C26 | 1.3822 (18) |
| C7—C6   | 1.3949 (15) | C23—C24 | 1.387 (2)   |
| C20—H11 | 1.1110      | C23—H20 | 1.1030      |
| C20—H12 | 1.1110      | C26—C25 | 1.386 (2)   |
| C15—C16 | 1.3893 (17) | C26—H23 | 1.1030      |
| C15—H7  | 1.1030      | C24—C25 | 1.389 (2)   |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C14—H18     | 1.1110      | C24—H21     | 1.1030      |
| C14—H17     | 1.1110      | C25—H22     | 1.1030      |
| N3—Fe1—N4   | 85.43 (4)   | H16—C12—C11 | 120.64 (7)  |
| N5—Fe1—N4   | 84.00 (4)   | N2—C2—S2    | 179.03 (10) |
| N5—Fe1—N3   | 90.89 (4)   | N1—C1—S1    | 179.07 (10) |
| N1—Fe1—N4   | 93.26 (4)   | C10—C9—N5   | 122.40 (11) |
| N1—Fe1—N3   | 178.16 (4)  | H13—C9—N5   | 118.80 (7)  |
| N1—Fe1—N5   | 90.25 (4)   | H13—C9—C10  | 118.80 (7)  |
| N6—Fe1—N4   | 82.60 (4)   | C4—C3—N3    | 122.44 (11) |
| N6—Fe1—N3   | 88.79 (4)   | H1—C3—N3    | 118.78 (6)  |
| N6—Fe1—N5   | 166.58 (4)  | H1—C3—C4    | 118.78 (7)  |
| N6—Fe1—N1   | 89.76 (4)   | C7—C8—N4    | 112.59 (9)  |
| N2—Fe1—N4   | 178.84 (4)  | H5—C8—N4    | 109.08 (5)  |
| N2—Fe1—N3   | 93.79 (4)   | H5—C8—C7    | 109.08 (6)  |
| N2—Fe1—N5   | 95.14 (4)   | H6—C8—N4    | 109.08 (6)  |
| N2—Fe1—N1   | 87.54 (4)   | H6—C8—C7    | 109.08 (6)  |
| N2—Fe1—N6   | 98.26 (4)   | H6—C8—H5    | 107.8       |
| C20—N4—Fe1  | 105.88 (6)  | C11—C10—C9  | 118.95 (11) |
| C14—N4—Fe1  | 106.50 (7)  | H14—C10—C9  | 120.53 (7)  |
| C14—N4—C20  | 111.80 (8)  | H14—C10—C11 | 120.53 (7)  |
| C8—N4—Fe1   | 110.40 (7)  | C17—C18—C19 | 118.95 (11) |
| C8—N4—C20   | 110.59 (8)  | H10—C18—C19 | 120.52 (7)  |
| C8—N4—C14   | 111.45 (8)  | H10—C18—C17 | 120.52 (7)  |
| C7—N3—Fe1   | 114.86 (7)  | C10—C11—C12 | 119.01 (11) |
| C3—N3—Fe1   | 126.39 (8)  | H15—C11—C12 | 120.49 (7)  |
| C3—N3—C7    | 118.71 (10) | H15—C11—C10 | 120.49 (7)  |
| C13—N5—Fe1  | 112.85 (7)  | C5—C6—C7    | 119.08 (11) |
| C9—N5—Fe1   | 128.59 (8)  | H4—C6—C7    | 120.46 (7)  |
| C9—N5—C13   | 118.53 (10) | H4—C6—C5    | 120.46 (7)  |
| C1—N1—Fe1   | 166.97 (9)  | C17—C16—C15 | 119.09 (11) |
| C19—N6—Fe1  | 113.35 (7)  | H8—C16—C15  | 120.46 (7)  |
| C15—N6—Fe1  | 127.81 (8)  | H8—C16—C17  | 120.46 (7)  |
| C15—N6—C19  | 118.79 (10) | C5—C4—C3    | 118.85 (11) |
| C2—N2—Fe1   | 170.07 (9)  | H2—C4—C3    | 120.57 (7)  |
| C14—C13—N5  | 115.01 (9)  | H2—C4—C5    | 120.57 (7)  |
| C12—C13—N5  | 122.39 (10) | C16—C17—C18 | 118.87 (11) |
| C12—C13—C14 | 122.48 (10) | H9—C17—C18  | 120.57 (7)  |
| C20—C19—N6  | 114.49 (9)  | H9—C17—C16  | 120.57 (7)  |
| C18—C19—N6  | 122.12 (10) | C23—C22—C21 | 118.72 (12) |
| C18—C19—C20 | 123.37 (10) | H19—C22—C21 | 120.64 (8)  |
| C8—C7—N3    | 116.58 (9)  | H19—C22—C23 | 120.64 (8)  |
| C6—C7—N3    | 122.00 (10) | C4—C5—C6    | 118.91 (11) |
| C6—C7—C8    | 121.41 (10) | H3—C5—C6    | 120.54 (7)  |
| C19—C20—N4  | 107.83 (9)  | H3—C5—C4    | 120.54 (7)  |
| H11—C20—N4  | 110.14 (5)  | C22—C21—C11 | 119.33 (10) |
| H11—C20—C19 | 110.14 (6)  | C26—C21—C11 | 118.92 (11) |
| H12—C20—N4  | 110.14 (6)  | C26—C21—C22 | 121.75 (13) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| H12—C20—C19     | 110.14 (6)   | C24—C23—C22     | 120.20 (13)  |
| H12—C20—H11     | 108.5        | H20—C23—C22     | 119.90 (8)   |
| C16—C15—N6      | 122.12 (11)  | H20—C23—C24     | 119.90 (9)   |
| H7—C15—N6       | 118.94 (7)   | C25—C26—C21     | 118.93 (14)  |
| H7—C15—C16      | 118.94 (7)   | H23—C26—C21     | 120.54 (9)   |
| C13—C14—N4      | 109.73 (9)   | H23—C26—C25     | 120.54 (9)   |
| H18—C14—N4      | 109.72 (5)   | C25—C24—C23     | 119.77 (14)  |
| H18—C14—C13     | 109.72 (6)   | H21—C24—C23     | 120.11 (9)   |
| H17—C14—N4      | 109.72 (6)   | H21—C24—C25     | 120.11 (9)   |
| H17—C14—C13     | 109.72 (6)   | C24—C25—C26     | 120.62 (14)  |
| H17—C14—H18     | 108.2        | H22—C25—C26     | 119.69 (9)   |
| C11—C12—C13     | 118.71 (11)  | H22—C25—C24     | 119.69 (9)   |
| H16—C12—C13     | 120.64 (7)   |                 |              |
| Fe1—N4—C20—C19  | 41.65 (7)    | C13—C14—N4—C8   | 83.76 (9)    |
| Fe1—N4—C14—C13  | −36.70 (7)   | C13—C12—C11—C10 | −1.35 (12)   |
| Fe1—N4—C8—C7    | 1.14 (7)     | C19—N6—C15—C16  | 2.39 (11)    |
| Fe1—N3—C7—C8    | −3.82 (8)    | C19—C20—N4—C14  | 157.23 (8)   |
| Fe1—N3—C7—C6    | 177.13 (8)   | C19—C20—N4—C8   | −77.94 (9)   |
| Fe1—N3—C3—C4    | −177.04 (10) | C19—C18—C17—C16 | 2.14 (13)    |
| Fe1—N5—C13—C14  | −5.91 (8)    | C7—N3—C3—C4     | 0.53 (12)    |
| Fe1—N5—C13—C12  | 177.89 (7)   | C7—C8—N4—C20    | 117.98 (9)   |
| Fe1—N5—C9—C10   | −178.50 (9)  | C7—C8—N4—C14    | −117.00 (9)  |
| Fe1—N6—C19—C20  | 2.85 (8)     | C7—C6—C5—C4     | 0.57 (13)    |
| Fe1—N6—C19—C18  | −178.90 (7)  | C20—C19—N6—C15  | −179.56 (9)  |
| Fe1—N6—C15—C16  | 179.60 (9)   | C20—C19—C18—C17 | 177.14 (11)  |
| C11—C21—C22—C23 | 179.58 (10)  | C15—N6—C19—C18  | −1.31 (11)   |
| C11—C21—C26—C25 | 179.97 (11)  | C15—C16—C17—C18 | −1.13 (13)   |
| N4—C20—C19—N6   | −30.12 (10)  | C14—C13—N5—C9   | 175.85 (9)   |
| N4—C20—C19—C18  | 151.66 (8)   | C14—C13—C12—C11 | −174.61 (11) |
| N4—C14—C13—N5   | 29.01 (10)   | C12—C13—N5—C9   | −0.36 (13)   |
| N4—C14—C13—C12  | −154.79 (8)  | C12—C11—C10—C9  | 0.49 (13)    |
| N4—C8—C7—N3     | 1.71 (10)    | C3—N3—C7—C8     | 178.32 (10)  |
| N4—C8—C7—C6     | −179.25 (9)  | C3—N3—C7—C6     | −0.72 (12)   |
| N3—C7—C6—C5     | 0.16 (13)    | C3—C4—C5—C6     | −0.74 (14)   |
| N3—C3—C4—C5     | 0.20 (14)    | C8—C7—C6—C5     | −178.83 (11) |
| N5—C13—C12—C11  | 1.32 (12)    | C22—C21—C26—C25 | 0.11 (15)    |
| N5—C9—C10—C11   | 0.49 (13)    | C22—C23—C24—C25 | −0.04 (16)   |
| N6—C19—C18—C17  | −0.96 (12)   | C21—C22—C23—C24 | 0.52 (15)    |
| N6—C15—C16—C17  | −1.18 (13)   | C21—C26—C25—C24 | 0.39 (16)    |
| C13—N5—C9—C10   | −0.56 (11)   | C23—C22—C21—C26 | −0.57 (15)   |
| C13—C14—N4—C20  | −151.89 (9)  | C23—C24—C25—C26 | −0.42 (16)   |



**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_120K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.3978$  (3) Å

$b = 12.6710$  (2) Å

$c = 15.2389$  (3) Å

$\beta = 110.070$  (2)°

$V = 2611.28$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.462$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 51755 reflections

$\theta = 2.1\text{--}32.2^\circ$

$\mu = 0.87$  mm<sup>-1</sup>

$T = 120$  K

Block, red

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.557$ ,  $T_{\max} = 1.000$

76156 measured reflections

6487 independent reflections

5860 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.072$

$S = 1.03$

6487 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.8401P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.0001$

$\Delta\rho_{\max} = 1.04$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.71$  e Å<sup>-3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.660448 (11) | 0.337559 (12) | 0.318982 (10) | 0.01721 (5)                      |
| S2  | 0.47550 (2)   | 0.04059 (2)   | 0.16868 (2)   | 0.02705 (8)                      |
| S1  | 0.74643 (3)   | 0.30196 (3)   | 0.04640 (2)   | 0.03248 (8)                      |
| Cl1 | 0.64581 (3)   | 0.03588 (3)   | 0.55671 (3)   | 0.04662 (10)                     |
| N3  | 0.60996 (7)   | 0.34397 (8)   | 0.42332 (7)   | 0.01937 (19)                     |
| N4  | 0.75176 (7)   | 0.45269 (7)   | 0.38554 (6)   | 0.01833 (18)                     |
| N6  | 0.77005 (7)   | 0.24353 (8)   | 0.39194 (6)   | 0.01914 (18)                     |
| N1  | 0.71099 (8)   | 0.33609 (8)   | 0.21409 (7)   | 0.0220 (2)                       |
| N5  | 0.57355 (7)   | 0.45745 (8)   | 0.26066 (6)   | 0.01982 (19)                     |
| N2  | 0.57225 (7)   | 0.22410 (8)   | 0.25658 (7)   | 0.0234 (2)                       |
| C19 | 0.62232 (8)   | 0.55047 (9)   | 0.27018 (7)   | 0.0201 (2)                       |
| C13 | 0.85594 (8)   | 0.29612 (9)   | 0.43338 (8)   | 0.0198 (2)                       |
| C7  | 0.65024 (8)   | 0.42028 (9)   | 0.48647 (7)   | 0.0197 (2)                       |
| C20 | 0.73233 (8)   | 0.54244 (9)   | 0.31851 (8)   | 0.0214 (2)                       |

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|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| H11 | 0.76156 (8)  | 0.61695 (9)   | 0.35633 (8)  | 0.0256 (3)* |
| H12 | 0.76939 (8)  | 0.52846 (9)   | 0.26650 (8)  | 0.0256 (3)* |
| C15 | 0.47484 (9)  | 0.45660 (10)  | 0.21761 (8)  | 0.0242 (2)  |
| H7  | 0.43454 (9)  | 0.38146 (10)  | 0.21208 (8)  | 0.0290 (3)* |
| C2  | 0.53170 (8)  | 0.14816 (9)   | 0.21928 (8)  | 0.0203 (2)  |
| C1  | 0.72567 (9)  | 0.32273 (9)   | 0.14410 (8)  | 0.0214 (2)  |
| C12 | 0.94131 (9)  | 0.24588 (10)  | 0.49025 (8)  | 0.0236 (2)  |
| H16 | 1.00971 (9)  | 0.29117 (10)  | 0.52411 (8)  | 0.0283 (3)* |
| C14 | 0.85373 (8)  | 0.41094 (9)   | 0.40831 (8)  | 0.0216 (2)  |
| H18 | 0.87829 (8)  | 0.42083 (9)   | 0.34719 (8)  | 0.0259 (3)* |
| H17 | 0.90520 (8)  | 0.45576 (9)   | 0.46789 (8)  | 0.0259 (3)* |
| C9  | 0.76777 (9)  | 0.13893 (9)   | 0.40618 (8)  | 0.0224 (2)  |
| H13 | 0.69782 (9)  | 0.09589 (9)   | 0.37320 (8)  | 0.0269 (3)* |
| C3  | 0.53840 (9)  | 0.28287 (10)  | 0.43487 (8)  | 0.0245 (2)  |
| H1  | 0.50578 (9)  | 0.21993 (10)  | 0.38352 (8)  | 0.0294 (3)* |
| C8  | 0.73146 (9)  | 0.48429 (9)   | 0.47194 (8)  | 0.0223 (2)  |
| H5  | 0.80005 (9)  | 0.47455 (9)   | 0.53348 (8)  | 0.0267 (3)* |
| H6  | 0.71063 (9)  | 0.56908 (9)   | 0.46690 (8)  | 0.0267 (3)* |
| C10 | 0.85069 (10) | 0.08304 (10)  | 0.46114 (8)  | 0.0262 (2)  |
| H14 | 0.84659 (10) | −0.00288 (10) | 0.47083 (8)  | 0.0314 (3)* |
| C18 | 0.57410 (9)  | 0.64444 (10)  | 0.23541 (8)  | 0.0243 (2)  |
| H10 | 0.61561 (9)  | 0.71923 (10)  | 0.24515 (8)  | 0.0291 (3)* |
| C11 | 0.93878 (10) | 0.13734 (10)  | 0.50362 (8)  | 0.0260 (2)  |
| H15 | 1.00562 (10) | 0.09500 (10)  | 0.54725 (8)  | 0.0312 (3)* |
| C16 | 0.42207 (9)  | 0.54734 (11)  | 0.17990 (8)  | 0.0284 (3)  |
| H8  | 0.34132 (9)  | 0.54393 (11)  | 0.14399 (8)  | 0.0341 (3)* |
| C6  | 0.61942 (9)  | 0.43839 (10)  | 0.56249 (8)  | 0.0261 (2)  |
| H4  | 0.65363 (9)  | 0.50155 (10)  | 0.61306 (8)  | 0.0314 (3)* |
| C4  | 0.50426 (10) | 0.29614 (11)  | 0.50885 (9)  | 0.0289 (3)  |
| H2  | 0.44575 (10) | 0.24452 (11)  | 0.51632 (9)  | 0.0347 (3)* |
| C17 | 0.47247 (10) | 0.64263 (11)  | 0.18804 (9)  | 0.0276 (3)  |
| H9  | 0.43252 (10) | 0.71551 (11)  | 0.15747 (9)  | 0.0331 (3)* |
| C22 | 0.75376 (10) | 0.20500 (11)  | 0.64150 (10) | 0.0320 (3)  |
| H19 | 0.76906 (10) | 0.21691 (11)  | 0.57606 (10) | 0.0384 (3)* |
| C5  | 0.54517 (10) | 0.37589 (12)  | 0.57365 (9)  | 0.0305 (3)  |
| H3  | 0.51893 (10) | 0.38906 (12)  | 0.63280 (9)  | 0.0365 (3)* |
| C21 | 0.69558 (10) | 0.12172 (11)  | 0.65043 (10) | 0.0308 (3)  |
| C23 | 0.79285 (10) | 0.27390 (12)  | 0.71700 (11) | 0.0371 (3)  |
| H20 | 0.83904 (10) | 0.34112 (12)  | 0.71114 (11) | 0.0445 (4)* |
| C26 | 0.67582 (12) | 0.10470 (12)  | 0.73186 (12) | 0.0415 (4)  |
| H23 | 0.62918 (12) | 0.03778 (12)  | 0.73746 (12) | 0.0498 (4)* |
| C24 | 0.77364 (12) | 0.25783 (14)  | 0.79924 (11) | 0.0432 (4)  |
| H21 | 0.80440 (12) | 0.31222 (14)  | 0.85870 (11) | 0.0518 (4)* |
| C25 | 0.71554 (14) | 0.17302 (14)  | 0.80637 (12) | 0.0464 (4)  |
| H22 | 0.70087 (14) | 0.15993 (14)  | 0.87198 (12) | 0.0557 (5)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.01938 (9)  | 0.01920 (9)  | 0.01321 (8)  | −0.00243 (6)  | 0.00581 (6)  | −0.00078 (5)  |
| S2  | 0.02538 (15) | 0.02543 (15) | 0.03076 (16) | −0.00510 (11) | 0.01018 (12) | −0.00867 (12) |
| S1  | 0.0509 (2)   | 0.03065 (17) | 0.02227 (15) | 0.01067 (14)  | 0.02076 (14) | 0.00468 (12)  |
| Cl1 | 0.0528 (2)   | 0.0371 (2)   | 0.0466 (2)   | −0.00139 (16) | 0.01266 (18) | −0.00290 (16) |
| N3  | 0.0204 (4)   | 0.0233 (5)   | 0.0151 (4)   | −0.0021 (4)   | 0.0069 (4)   | −0.0005 (3)   |
| N4  | 0.0195 (4)   | 0.0180 (4)   | 0.0166 (4)   | −0.0023 (3)   | 0.0051 (3)   | 0.0001 (3)    |
| N6  | 0.0230 (5)   | 0.0189 (4)   | 0.0162 (4)   | −0.0015 (4)   | 0.0077 (4)   | −0.0003 (3)   |
| N1  | 0.0270 (5)   | 0.0234 (5)   | 0.0174 (5)   | 0.0003 (4)    | 0.0099 (4)   | 0.0004 (4)    |
| N5  | 0.0205 (5)   | 0.0229 (5)   | 0.0147 (4)   | −0.0008 (4)   | 0.0044 (3)   | −0.0011 (3)   |
| N2  | 0.0251 (5)   | 0.0251 (5)   | 0.0188 (5)   | −0.0055 (4)   | 0.0062 (4)   | −0.0038 (4)   |
| C19 | 0.0219 (5)   | 0.0223 (5)   | 0.0157 (5)   | 0.0001 (4)    | 0.0060 (4)   | 0.0013 (4)    |
| C13 | 0.0208 (5)   | 0.0202 (5)   | 0.0177 (5)   | 0.0005 (4)    | 0.0059 (4)   | 0.0008 (4)    |
| C7  | 0.0217 (5)   | 0.0229 (5)   | 0.0140 (5)   | 0.0015 (4)    | 0.0057 (4)   | −0.0003 (4)   |
| C20 | 0.0229 (5)   | 0.0204 (5)   | 0.0202 (5)   | −0.0015 (4)   | 0.0065 (4)   | 0.0026 (4)    |
| C15 | 0.0215 (5)   | 0.0297 (6)   | 0.0187 (5)   | −0.0013 (5)   | 0.0036 (4)   | −0.0030 (4)   |
| C2  | 0.0209 (5)   | 0.0228 (5)   | 0.0173 (5)   | −0.0027 (4)   | 0.0066 (4)   | −0.0028 (4)   |
| C1  | 0.0279 (6)   | 0.0207 (5)   | 0.0173 (5)   | 0.0026 (4)    | 0.0099 (4)   | 0.0019 (4)    |
| C12 | 0.0240 (6)   | 0.0251 (6)   | 0.0203 (5)   | 0.0031 (5)    | 0.0059 (4)   | 0.0004 (4)    |
| C14 | 0.0196 (5)   | 0.0218 (5)   | 0.0222 (5)   | −0.0024 (4)   | 0.0057 (4)   | 0.0012 (4)    |
| C9  | 0.0300 (6)   | 0.0197 (5)   | 0.0188 (5)   | −0.0016 (4)   | 0.0101 (5)   | −0.0003 (4)   |
| C3  | 0.0238 (5)   | 0.0306 (6)   | 0.0205 (5)   | −0.0061 (5)   | 0.0094 (4)   | −0.0011 (4)   |
| C8  | 0.0252 (5)   | 0.0232 (5)   | 0.0174 (5)   | −0.0037 (4)   | 0.0060 (4)   | −0.0035 (4)   |
| C10 | 0.0359 (7)   | 0.0207 (6)   | 0.0228 (6)   | 0.0025 (5)    | 0.0111 (5)   | 0.0014 (4)    |
| C18 | 0.0273 (6)   | 0.0237 (6)   | 0.0213 (5)   | 0.0031 (5)    | 0.0076 (5)   | 0.0034 (4)    |
| C11 | 0.0306 (6)   | 0.0247 (6)   | 0.0221 (5)   | 0.0057 (5)    | 0.0084 (5)   | 0.0020 (5)    |
| C16 | 0.0227 (6)   | 0.0369 (7)   | 0.0217 (5)   | 0.0037 (5)    | 0.0027 (4)   | −0.0011 (5)   |
| C6  | 0.0289 (6)   | 0.0329 (6)   | 0.0177 (5)   | 0.0013 (5)    | 0.0093 (5)   | −0.0035 (5)   |
| C4  | 0.0267 (6)   | 0.0402 (7)   | 0.0236 (6)   | −0.0044 (5)   | 0.0135 (5)   | 0.0007 (5)    |
| C17 | 0.0269 (6)   | 0.0308 (6)   | 0.0230 (6)   | 0.0074 (5)    | 0.0062 (5)   | 0.0047 (5)    |
| C22 | 0.0327 (7)   | 0.0345 (7)   | 0.0335 (7)   | 0.0033 (5)    | 0.0176 (6)   | 0.0078 (6)    |
| C5  | 0.0315 (6)   | 0.0436 (8)   | 0.0205 (6)   | 0.0014 (6)    | 0.0143 (5)   | −0.0005 (5)   |
| C21 | 0.0320 (6)   | 0.0290 (6)   | 0.0341 (7)   | 0.0050 (5)    | 0.0149 (5)   | 0.0063 (5)    |
| C23 | 0.0269 (6)   | 0.0359 (7)   | 0.0478 (9)   | 0.0012 (5)    | 0.0119 (6)   | 0.0017 (6)    |
| C26 | 0.0519 (9)   | 0.0357 (8)   | 0.0492 (9)   | 0.0069 (7)    | 0.0332 (7)   | 0.0126 (7)    |
| C24 | 0.0415 (8)   | 0.0480 (9)   | 0.0337 (7)   | 0.0166 (7)    | 0.0046 (6)   | −0.0021 (6)   |
| C25 | 0.0658 (11)  | 0.0472 (9)   | 0.0348 (8)   | 0.0202 (8)    | 0.0283 (8)   | 0.0119 (7)    |

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

|        |             |         |             |
|--------|-------------|---------|-------------|
| Fe1—N3 | 1.9638 (10) | C14—H18 | 1.1110      |
| Fe1—N4 | 1.9925 (9)  | C14—H17 | 1.1110      |
| Fe1—N6 | 1.9854 (10) | C9—H13  | 1.1030      |
| Fe1—N1 | 1.9705 (10) | C9—C10  | 1.3936 (17) |
| Fe1—N5 | 1.9748 (10) | C3—H1   | 1.1030      |
| Fe1—N2 | 1.9377 (10) | C3—C4   | 1.3860 (16) |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| S2—C2      | 1.6368 (12) | C8—H5       | 1.1110      |
| S1—C1      | 1.6373 (12) | C8—H6       | 1.1110      |
| C11—C21    | 1.7417 (15) | C10—H14     | 1.1030      |
| N3—C7      | 1.3470 (14) | C10—C11     | 1.3919 (18) |
| N3—C3      | 1.3477 (15) | C18—H10     | 1.1030      |
| N4—C20     | 1.4896 (14) | C18—C17     | 1.3920 (17) |
| N4—C14     | 1.4853 (14) | C11—H15     | 1.1030      |
| N4—C8      | 1.4977 (14) | C16—H8      | 1.1030      |
| N6—C13     | 1.3550 (14) | C16—C17     | 1.3922 (19) |
| N6—C9      | 1.3453 (15) | C6—H4       | 1.1030      |
| N1—C1      | 1.1686 (16) | C6—C5       | 1.3869 (19) |
| N5—C19     | 1.3540 (15) | C4—H2       | 1.1030      |
| N5—C15     | 1.3455 (15) | C4—C5       | 1.3943 (19) |
| N2—C2      | 1.1671 (16) | C17—H9      | 1.1030      |
| C19—C20    | 1.5037 (16) | C22—H19     | 1.1030      |
| C19—C18    | 1.3885 (16) | C22—C21     | 1.3827 (19) |
| C13—C12    | 1.3915 (16) | C22—C23     | 1.399 (2)   |
| C13—C14    | 1.5017 (16) | C5—H3       | 1.1030      |
| C7—C8      | 1.5007 (16) | C21—C26     | 1.382 (2)   |
| C7—C6      | 1.3944 (15) | C23—H20     | 1.1030      |
| C20—H11    | 1.1110      | C23—C24     | 1.388 (2)   |
| C20—H12    | 1.1110      | C26—H23     | 1.1030      |
| C15—H7     | 1.1030      | C26—C25     | 1.386 (3)   |
| C15—C16    | 1.3891 (18) | C24—H21     | 1.1030      |
| C12—H16    | 1.1030      | C24—C25     | 1.389 (3)   |
| C12—C11    | 1.3927 (17) | C25—H22     | 1.1030      |
|            |             |             |             |
| N4—Fe1—N3  | 85.44 (4)   | H17—C14—N4  | 109.72 (6)  |
| N6—Fe1—N3  | 90.88 (4)   | H17—C14—C13 | 109.72 (6)  |
| N6—Fe1—N4  | 84.00 (4)   | H17—C14—H18 | 108.2       |
| N1—Fe1—N3  | 178.17 (4)  | H13—C9—N6   | 118.85 (7)  |
| N1—Fe1—N4  | 93.27 (4)   | C10—C9—N6   | 122.29 (11) |
| N1—Fe1—N6  | 90.27 (4)   | C10—C9—H13  | 118.85 (7)  |
| N5—Fe1—N3  | 88.81 (4)   | H1—C3—N3    | 118.76 (7)  |
| N5—Fe1—N4  | 82.61 (4)   | C4—C3—N3    | 122.47 (11) |
| N5—Fe1—N6  | 166.59 (4)  | C4—C3—H1    | 118.76 (8)  |
| N5—Fe1—N1  | 89.74 (4)   | C7—C8—N4    | 112.47 (9)  |
| N2—Fe1—N3  | 93.77 (4)   | H5—C8—N4    | 109.10 (6)  |
| N2—Fe1—N4  | 178.86 (4)  | H5—C8—C7    | 109.10 (6)  |
| N2—Fe1—N6  | 95.20 (4)   | H6—C8—N4    | 109.10 (6)  |
| N2—Fe1—N1  | 87.54 (4)   | H6—C8—C7    | 109.10 (6)  |
| N2—Fe1—N5  | 98.20 (4)   | H6—C8—H5    | 107.8       |
| C7—N3—Fe1  | 114.83 (8)  | H14—C10—C9  | 120.51 (7)  |
| C3—N3—Fe1  | 126.44 (8)  | C11—C10—C9  | 118.97 (11) |
| C3—N3—C7   | 118.69 (10) | C11—C10—H14 | 120.51 (7)  |
| C20—N4—Fe1 | 105.90 (7)  | H10—C18—C19 | 120.57 (7)  |
| C14—N4—Fe1 | 106.54 (7)  | C17—C18—C19 | 118.87 (12) |
| C14—N4—C20 | 111.72 (9)  | C17—C18—H10 | 120.57 (8)  |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C8—N4—Fe1      | 110.46 (7)   | C10—C11—C12     | 119.04 (12)  |
| C8—N4—C20      | 110.57 (9)   | H15—C11—C12     | 120.48 (7)   |
| C8—N4—C14      | 111.43 (9)   | H15—C11—C10     | 120.48 (7)   |
| C13—N6—Fe1     | 112.88 (8)   | H8—C16—C15      | 120.44 (7)   |
| C9—N6—Fe1      | 128.52 (8)   | C17—C16—C15     | 119.12 (11)  |
| C9—N6—C13      | 118.57 (10)  | C17—C16—H8      | 120.44 (7)   |
| C1—N1—Fe1      | 167.03 (10)  | H4—C6—C7        | 120.45 (7)   |
| C19—N5—Fe1     | 113.35 (7)   | C5—C6—C7        | 119.10 (12)  |
| C15—N5—Fe1     | 127.83 (8)   | C5—C6—H4        | 120.45 (7)   |
| C15—N5—C19     | 118.77 (10)  | H2—C4—C3        | 120.57 (8)   |
| C2—N2—Fe1      | 169.98 (10)  | C5—C4—C3        | 118.85 (12)  |
| C20—C19—N5     | 114.45 (10)  | C5—C4—H2        | 120.57 (7)   |
| C18—C19—N5     | 122.18 (11)  | C16—C17—C18     | 118.91 (12)  |
| C18—C19—C20    | 123.34 (11)  | H9—C17—C18      | 120.55 (8)   |
| C12—C13—N6     | 122.36 (11)  | H9—C17—C16      | 120.55 (7)   |
| C14—C13—N6     | 114.94 (10)  | C21—C22—H19     | 120.61 (8)   |
| C14—C13—C12    | 122.59 (10)  | C23—C22—H19     | 120.61 (8)   |
| C8—C7—N3       | 116.67 (10)  | C23—C22—C21     | 118.79 (13)  |
| C6—C7—N3       | 122.00 (11)  | C4—C5—C6        | 118.88 (11)  |
| C6—C7—C8       | 121.33 (10)  | H3—C5—C6        | 120.56 (7)   |
| C19—C20—N4     | 107.81 (9)   | H3—C5—C4        | 120.56 (7)   |
| H11—C20—N4     | 110.14 (6)   | C22—C21—C11     | 119.24 (11)  |
| H11—C20—C19    | 110.14 (6)   | C26—C21—C11     | 119.03 (12)  |
| H12—C20—N4     | 110.14 (6)   | C26—C21—C22     | 121.72 (14)  |
| H12—C20—C19    | 110.14 (6)   | H20—C23—C22     | 119.92 (8)   |
| H12—C20—H11    | 108.5        | C24—C23—C22     | 120.15 (14)  |
| H7—C15—N5      | 118.95 (7)   | C24—C23—H20     | 119.92 (10)  |
| C16—C15—N5     | 122.09 (12)  | H23—C26—C21     | 120.52 (10)  |
| C16—C15—H7     | 118.95 (7)   | C25—C26—C21     | 118.95 (15)  |
| N2—C2—S2       | 179.03 (11)  | C25—C26—H23     | 120.52 (9)   |
| N1—C1—S1       | 179.07 (11)  | H21—C24—C23     | 120.13 (10)  |
| H16—C12—C13    | 120.62 (7)   | C25—C24—C23     | 119.75 (15)  |
| C11—C12—C13    | 118.75 (11)  | C25—C24—H21     | 120.13 (10)  |
| C11—C12—H16    | 120.62 (7)   | C24—C25—C26     | 120.63 (15)  |
| C13—C14—N4     | 109.72 (9)   | H22—C25—C26     | 119.68 (9)   |
| H18—C14—N4     | 109.72 (6)   | H22—C25—C24     | 119.68 (10)  |
| H18—C14—C13    | 109.72 (6)   |                 |              |
| Fe1—N3—C7—C8   | −3.77 (9)    | C19—C20—N4—C8   | −77.99 (10)  |
| Fe1—N3—C7—C6   | 177.14 (8)   | C19—C18—C17—C16 | 2.17 (13)    |
| Fe1—N3—C3—C4   | −177.03 (10) | C13—N6—C9—C10   | −0.64 (12)   |
| Fe1—N4—C20—C19 | 41.70 (7)    | C13—C12—C11—C10 | −1.14 (13)   |
| Fe1—N4—C14—C13 | −36.73 (7)   | C13—C14—N4—C20  | −151.94 (9)  |
| Fe1—N4—C8—C7   | 1.17 (7)     | C13—C14—N4—C8   | 83.82 (10)   |
| Fe1—N6—C13—C12 | 177.93 (7)   | C7—N3—C3—C4     | 0.48 (13)    |
| Fe1—N6—C13—C14 | −5.86 (8)    | C7—C8—N4—C20    | 118.07 (10)  |
| Fe1—N6—C9—C10  | −178.56 (9)  | C7—C8—N4—C14    | −117.05 (10) |
| Fe1—N5—C19—C20 | 2.91 (8)     | C7—C6—C5—C4     | 0.59 (14)    |

|                 |             |                 |              |
|-----------------|-------------|-----------------|--------------|
| Fe1—N5—C19—C18  | −178.87 (8) | C20—C19—N5—C15  | −179.50 (9)  |
| Fe1—N5—C15—C16  | 179.46 (10) | C20—C19—C18—C17 | 177.12 (11)  |
| C11—C21—C22—C23 | 179.59 (11) | C15—N5—C19—C18  | −1.28 (12)   |
| C11—C21—C26—C25 | 179.96 (12) | C15—C16—C17—C18 | −1.25 (14)   |
| N3—C7—C8—N4     | 1.66 (11)   | C12—C13—N6—C9   | −0.31 (13)   |
| N3—C7—C6—C5     | 0.11 (13)   | C12—C11—C10—C9  | 0.26 (13)    |
| N3—C3—C4—C5     | 0.22 (15)   | C14—C13—N6—C9   | 175.90 (9)   |
| N4—C20—C19—N5   | −30.17 (10) | C14—C13—C12—C11 | −174.72 (11) |
| N4—C20—C19—C18  | 151.63 (9)  | C3—N3—C7—C8     | 178.44 (11)  |
| N4—C14—C13—N6   | 28.97 (10)  | C3—N3—C7—C6     | −0.65 (13)   |
| N4—C14—C13—C12  | −154.83 (9) | C3—C4—C5—C6     | −0.75 (15)   |
| N4—C8—C7—C6     | −179.25 (9) | C8—C7—C6—C5     | −178.93 (12) |
| N6—C13—C12—C11  | 1.20 (13)   | C22—C21—C26—C25 | −0.04 (16)   |
| N6—C9—C10—C11   | 0.66 (13)   | C22—C23—C24—C25 | 0.07 (16)    |
| N5—C19—C18—C17  | −0.94 (13)  | C21—C22—C23—C24 | 0.39 (16)    |
| N5—C15—C16—C17  | −1.01 (14)  | C21—C26—C25—C24 | 0.52 (17)    |
| C19—N5—C15—C16  | 2.27 (12)   | C23—C22—C21—C26 | −0.41 (16)   |
| C19—C20—N4—C14  | 157.30 (9)  | C23—C24—C25—C26 | −0.54 (17)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N, $N'$ , $N''$ , $N'''$ ]iron(II) chlorobenzene monosolvate (cl\_130K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.4057$  (3) Å

$b = 12.6757$  (2) Å

$c = 15.2461$  (3) Å

$\beta = 110.058$  (2)°

$V = 2615.11$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.460$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 50890 reflections

$\theta = 2.0$ – $32.0^\circ$

$\mu = 0.87$  mm<sup>−1</sup>

$T = 130$  K

Block, red

$0.32 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.570$ ,  $T_{\max} = 1.000$

76285 measured reflections

6493 independent reflections

5859 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.077$

$S = 1.04$

6493 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.8555P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 1.24 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.80 \text{ e } \text{\AA}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.339532 (12) | 0.337497 (13) | 0.681067 (11) | 0.01836 (6)                      |
| S2  | 0.52450 (2)   | 0.04078 (3)   | 0.83134 (2)   | 0.02882 (8)                      |
| S1  | 0.25365 (3)   | 0.30201 (3)   | 0.95353 (2)   | 0.03485 (9)                      |
| Cl1 | 0.35411 (4)   | 0.03593 (3)   | 0.44308 (3)   | 0.05044 (11)                     |
| N3  | 0.38996 (8)   | 0.34398 (8)   | 0.57673 (7)   | 0.0209 (2)                       |
| N4  | 0.24821 (7)   | 0.45262 (8)   | 0.61454 (7)   | 0.01953 (19)                     |
| N5  | 0.22994 (8)   | 0.24353 (8)   | 0.60813 (7)   | 0.0204 (2)                       |
| N6  | 0.42638 (7)   | 0.45740 (8)   | 0.73937 (7)   | 0.0212 (2)                       |
| N1  | 0.28903 (8)   | 0.33606 (8)   | 0.78592 (7)   | 0.0235 (2)                       |
| N2  | 0.42767 (8)   | 0.22410 (9)   | 0.74339 (7)   | 0.0248 (2)                       |
| C19 | 0.37762 (9)   | 0.55044 (10)  | 0.72982 (8)   | 0.0213 (2)                       |
| C13 | 0.14421 (9)   | 0.29617 (10)  | 0.56686 (8)   | 0.0209 (2)                       |
| C7  | 0.34969 (9)   | 0.42015 (10)  | 0.51367 (8)   | 0.0210 (2)                       |
| C14 | 0.14627 (9)   | 0.41092 (10)  | 0.59184 (9)   | 0.0231 (2)                       |
| H18 | 0.12173 (9)   | 0.42081 (10)  | 0.65294 (9)   | 0.0277 (3)*                      |
| H17 | 0.09480 (9)   | 0.45568 (10)  | 0.53228 (9)   | 0.0277 (3)*                      |
| C15 | 0.52496 (9)   | 0.45650 (11)  | 0.78237 (8)   | 0.0257 (3)                       |
| H7  | 0.56519 (9)   | 0.38136 (11)  | 0.78790 (8)   | 0.0308 (3)*                      |
| C20 | 0.26770 (9)   | 0.54241 (10)  | 0.68147 (8)   | 0.0227 (2)                       |
| H11 | 0.23853 (9)   | 0.61687 (10)  | 0.64360 (8)   | 0.0273 (3)*                      |
| H12 | 0.23062 (9)   | 0.52856 (10)  | 0.73345 (8)   | 0.0273 (3)*                      |
| C1  | 0.27440 (9)   | 0.32274 (9)   | 0.85582 (9)   | 0.0228 (2)                       |
| C9  | 0.23199 (10)  | 0.13895 (10)  | 0.59379 (8)   | 0.0239 (2)                       |
| H13 | 0.30183 (10)  | 0.09584 (10)  | 0.62676 (8)   | 0.0287 (3)*                      |
| C12 | 0.05882 (9)   | 0.24585 (10)  | 0.50995 (9)   | 0.0249 (2)                       |
| H16 | −0.00951 (9)  | 0.29117 (10)  | 0.47614 (9)   | 0.0299 (3)*                      |
| C2  | 0.46825 (9)   | 0.14816 (10)  | 0.78065 (8)   | 0.0216 (2)                       |
| C3  | 0.46142 (9)   | 0.28289 (11)  | 0.56513 (9)   | 0.0264 (3)                       |
| H1  | 0.49396 (9)   | 0.21991 (11)  | 0.61639 (9)   | 0.0316 (3)*                      |
| C8  | 0.26834 (9)   | 0.48411 (10)  | 0.52810 (8)   | 0.0239 (2)                       |
| H5  | 0.19980 (9)   | 0.47414 (10)  | 0.46664 (8)   | 0.0287 (3)*                      |
| H6  | 0.28900 (9)   | 0.56891 (10)  | 0.53296 (8)   | 0.0287 (3)*                      |
| C10 | 0.14927 (10)  | 0.08316 (10)  | 0.53889 (9)   | 0.0280 (3)                       |
| H14 | 0.15341 (10)  | −0.00271 (10) | 0.52911 (9)   | 0.0335 (3)*                      |
| C18 | 0.42590 (10)  | 0.64432 (10)  | 0.76446 (9)   | 0.0260 (3)                       |
| H10 | 0.38443 (10)  | 0.71910 (10)  | 0.75467 (9)   | 0.0312 (3)*                      |
| C11 | 0.06120 (10)  | 0.13746 (11)  | 0.49649 (9)   | 0.0279 (3)                       |
| H15 | −0.00564 (10) | 0.09521 (11)  | 0.45292 (9)   | 0.0334 (3)*                      |
| C17 | 0.52742 (10)  | 0.64261 (11)  | 0.81172 (9)   | 0.0297 (3)                       |
| H9  | 0.56736 (10)  | 0.71552 (11)  | 0.84207 (9)   | 0.0356 (3)*                      |
| C6  | 0.38041 (10)  | 0.43840 (11)  | 0.43761 (9)   | 0.0280 (3)                       |



|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| H4  | 0.34624 (10) | 0.50156 (11) | 0.38709 (9)  | 0.0336 (3)* |
| C16 | 0.57776 (10) | 0.54715 (12) | 0.82005 (9)  | 0.0302 (3)  |
| H8  | 0.65844 (10) | 0.54365 (12) | 0.85609 (9)  | 0.0362 (3)* |
| C4  | 0.49558 (10) | 0.29622 (12) | 0.49123 (10) | 0.0308 (3)  |
| H2  | 0.55414 (10) | 0.24471 (12) | 0.48379 (10) | 0.0370 (3)* |
| C5  | 0.45466 (11) | 0.37581 (13) | 0.42652 (9)  | 0.0329 (3)  |
| H3  | 0.48085 (11) | 0.38890 (13) | 0.36737 (9)  | 0.0395 (4)* |
| C21 | 0.30447 (11) | 0.12174 (12) | 0.34933 (11) | 0.0329 (3)  |
| C22 | 0.24632 (11) | 0.20491 (12) | 0.35836 (11) | 0.0342 (3)  |
| H19 | 0.23103 (11) | 0.21682 (12) | 0.42378 (11) | 0.0410 (4)* |
| C23 | 0.20728 (11) | 0.27360 (13) | 0.28291 (12) | 0.0402 (3)  |
| H20 | 0.16100 (11) | 0.34070 (13) | 0.28878 (12) | 0.0483 (4)* |
| C26 | 0.32418 (13) | 0.10488 (13) | 0.26802 (13) | 0.0446 (4)  |
| H23 | 0.37086 (13) | 0.03805 (13) | 0.26243 (13) | 0.0535 (5)* |
| C24 | 0.22640 (13) | 0.25786 (15) | 0.20073 (12) | 0.0462 (4)  |
| H21 | 0.19567 (13) | 0.31232 (15) | 0.14137 (12) | 0.0554 (5)* |
| C25 | 0.28453 (15) | 0.17301 (15) | 0.19360 (13) | 0.0498 (5)  |
| H22 | 0.29919 (15) | 0.15988 (15) | 0.12802 (13) | 0.0598 (5)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02068 (9)  | 0.02040 (10) | 0.01416 (9)  | 0.00269 (6)   | 0.00617 (6)  | 0.00088 (6)   |
| S2  | 0.02708 (16) | 0.02707 (16) | 0.03287 (17) | 0.00555 (12)  | 0.01098 (13) | 0.00931 (12)  |
| S1  | 0.0547 (2)   | 0.03276 (18) | 0.02385 (16) | −0.01164 (16) | 0.02224 (16) | −0.00515 (13) |
| Cl1 | 0.0572 (3)   | 0.0401 (2)   | 0.0504 (2)   | 0.00148 (18)  | 0.0137 (2)   | 0.00320 (17)  |
| N3  | 0.0221 (5)   | 0.0246 (5)   | 0.0166 (5)   | 0.0021 (4)    | 0.0072 (4)   | 0.0004 (4)    |
| N4  | 0.0212 (5)   | 0.0193 (5)   | 0.0172 (4)   | 0.0026 (4)    | 0.0056 (4)   | −0.0003 (4)   |
| N5  | 0.0245 (5)   | 0.0202 (5)   | 0.0170 (4)   | 0.0012 (4)    | 0.0079 (4)   | 0.0001 (4)    |
| N6  | 0.0221 (5)   | 0.0243 (5)   | 0.0159 (4)   | 0.0010 (4)    | 0.0047 (4)   | 0.0010 (4)    |
| N1  | 0.0282 (5)   | 0.0255 (5)   | 0.0187 (5)   | −0.0002 (4)   | 0.0106 (4)   | −0.0006 (4)   |
| N2  | 0.0264 (5)   | 0.0267 (5)   | 0.0202 (5)   | 0.0058 (4)    | 0.0067 (4)   | 0.0039 (4)    |
| C19 | 0.0234 (6)   | 0.0233 (6)   | 0.0166 (5)   | −0.0000 (4)   | 0.0063 (4)   | −0.0012 (4)   |
| C13 | 0.0222 (5)   | 0.0216 (6)   | 0.0184 (5)   | −0.0006 (4)   | 0.0062 (4)   | −0.0008 (4)   |
| C7  | 0.0231 (5)   | 0.0244 (6)   | 0.0153 (5)   | −0.0010 (4)   | 0.0062 (4)   | 0.0006 (4)    |
| C14 | 0.0214 (5)   | 0.0228 (6)   | 0.0238 (6)   | 0.0023 (4)    | 0.0061 (4)   | −0.0021 (5)   |
| C15 | 0.0233 (6)   | 0.0315 (7)   | 0.0196 (5)   | 0.0015 (5)    | 0.0040 (4)   | 0.0036 (5)    |
| C20 | 0.0241 (6)   | 0.0221 (6)   | 0.0211 (5)   | 0.0019 (4)    | 0.0066 (5)   | −0.0025 (4)   |
| C1  | 0.0297 (6)   | 0.0223 (5)   | 0.0184 (6)   | −0.0027 (4)   | 0.0109 (5)   | −0.0023 (4)   |
| C9  | 0.0319 (6)   | 0.0208 (6)   | 0.0205 (5)   | 0.0023 (5)    | 0.0108 (5)   | 0.0005 (4)    |
| C12 | 0.0248 (6)   | 0.0259 (6)   | 0.0222 (6)   | −0.0035 (5)   | 0.0057 (5)   | −0.0006 (5)   |
| C2  | 0.0218 (5)   | 0.0243 (6)   | 0.0187 (5)   | 0.0028 (4)    | 0.0069 (4)   | 0.0025 (4)    |
| C3  | 0.0254 (6)   | 0.0331 (7)   | 0.0222 (6)   | 0.0066 (5)    | 0.0101 (5)   | 0.0014 (5)    |
| C8  | 0.0275 (6)   | 0.0245 (6)   | 0.0187 (5)   | 0.0039 (5)    | 0.0066 (5)   | 0.0043 (4)    |
| C10 | 0.0386 (7)   | 0.0220 (6)   | 0.0240 (6)   | −0.0030 (5)   | 0.0117 (5)   | −0.0022 (5)   |
| C18 | 0.0285 (6)   | 0.0259 (6)   | 0.0230 (6)   | −0.0034 (5)   | 0.0080 (5)   | −0.0039 (5)   |
| C11 | 0.0326 (7)   | 0.0265 (6)   | 0.0235 (6)   | −0.0065 (5)   | 0.0083 (5)   | −0.0023 (5)   |
| C17 | 0.0289 (7)   | 0.0333 (7)   | 0.0251 (6)   | −0.0081 (5)   | 0.0068 (5)   | −0.0052 (5)   |

|     |             |             |             |             |            |             |
|-----|-------------|-------------|-------------|-------------|------------|-------------|
| C6  | 0.0306 (6)  | 0.0350 (7)  | 0.0192 (6)  | −0.0015 (5) | 0.0098 (5) | 0.0042 (5)  |
| C16 | 0.0241 (6)  | 0.0394 (8)  | 0.0232 (6)  | −0.0037 (5) | 0.0031 (5) | 0.0012 (5)  |
| C4  | 0.0275 (6)  | 0.0437 (8)  | 0.0252 (6)  | 0.0044 (6)  | 0.0143 (5) | −0.0010 (6) |
| C5  | 0.0338 (7)  | 0.0465 (8)  | 0.0226 (6)  | −0.0008 (6) | 0.0151 (5) | 0.0011 (6)  |
| C21 | 0.0340 (7)  | 0.0312 (7)  | 0.0363 (7)  | −0.0054 (6) | 0.0156 (6) | −0.0071 (6) |
| C22 | 0.0351 (7)  | 0.0374 (8)  | 0.0355 (7)  | −0.0043 (6) | 0.0191 (6) | −0.0088 (6) |
| C23 | 0.0288 (7)  | 0.0386 (8)  | 0.0521 (9)  | −0.0016 (6) | 0.0124 (7) | −0.0020 (7) |
| C26 | 0.0551 (9)  | 0.0382 (8)  | 0.0537 (10) | −0.0076 (7) | 0.0357 (8) | −0.0152 (7) |
| C24 | 0.0453 (9)  | 0.0510 (10) | 0.0356 (8)  | −0.0176 (8) | 0.0054 (7) | 0.0024 (7)  |
| C25 | 0.0707 (12) | 0.0503 (10) | 0.0374 (9)  | −0.0217 (9) | 0.0301 (9) | −0.0129 (7) |

*Geometric parameters (Å, °)*

|           |             |            |             |
|-----------|-------------|------------|-------------|
| Fe1—N3    | 1.9641 (10) | C9—H13     | 1.1030      |
| Fe1—N4    | 1.9934 (10) | C9—C10     | 1.3916 (18) |
| Fe1—N5    | 1.9858 (10) | C12—H16    | 1.1030      |
| Fe1—N6    | 1.9754 (11) | C12—C11    | 1.3913 (18) |
| Fe1—N1    | 1.9705 (11) | C3—H1      | 1.1030      |
| Fe1—N2    | 1.9374 (11) | C3—C4      | 1.3858 (18) |
| S2—C2     | 1.6360 (13) | C8—H5      | 1.1110      |
| S1—C1     | 1.6380 (13) | C8—H6      | 1.1110      |
| Cl1—C21   | 1.7425 (16) | C10—H14    | 1.1030      |
| N3—C7     | 1.3456 (15) | C10—C11    | 1.392 (2)   |
| N3—C3     | 1.3474 (16) | C18—H10    | 1.1030      |
| N4—C14    | 1.4857 (15) | C18—C17    | 1.3912 (19) |
| N4—C20    | 1.4896 (15) | C11—H15    | 1.1030      |
| N4—C8     | 1.4975 (15) | C17—H9     | 1.1030      |
| N5—C13    | 1.3540 (15) | C17—C16    | 1.394 (2)   |
| N5—C9     | 1.3456 (16) | C6—H4      | 1.1030      |
| N6—C19    | 1.3548 (16) | C6—C5      | 1.388 (2)   |
| N6—C15    | 1.3446 (16) | C16—H8     | 1.1030      |
| N1—C1     | 1.1673 (17) | C4—H2      | 1.1030      |
| N2—C2     | 1.1676 (17) | C4—C5      | 1.393 (2)   |
| C19—C20   | 1.5035 (16) | C5—H3      | 1.1030      |
| C19—C18   | 1.3881 (17) | C21—C22    | 1.383 (2)   |
| C13—C14   | 1.5013 (17) | C21—C26    | 1.380 (2)   |
| C13—C12   | 1.3930 (17) | C22—H19    | 1.1030      |
| C7—C8     | 1.5019 (17) | C22—C23    | 1.398 (2)   |
| C7—C6     | 1.3950 (16) | C23—H20    | 1.1030      |
| C14—H18   | 1.1110      | C23—C24    | 1.386 (2)   |
| C14—H17   | 1.1110      | C26—H23    | 1.1030      |
| C15—H7    | 1.1030      | C26—C25    | 1.384 (3)   |
| C15—C16   | 1.3889 (19) | C24—H21    | 1.1030      |
| C20—H11   | 1.1110      | C24—C25    | 1.390 (3)   |
| C20—H12   | 1.1110      | C25—H22    | 1.1030      |
| N4—Fe1—N3 | 85.42 (4)   | C10—C9—N5  | 122.44 (12) |
| N5—Fe1—N3 | 90.88 (4)   | C10—C9—H13 | 118.78 (8)  |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N5—Fe1—N4   | 83.97 (4)   | H16—C12—C13 | 120.56 (7)  |
| N6—Fe1—N3   | 88.80 (4)   | C11—C12—C13 | 118.87 (12) |
| N6—Fe1—N4   | 82.61 (4)   | C11—C12—H16 | 120.56 (8)  |
| N6—Fe1—N5   | 166.56 (4)  | N2—C2—S2    | 179.12 (12) |
| N1—Fe1—N3   | 178.13 (4)  | H1—C3—N3    | 118.76 (7)  |
| N1—Fe1—N4   | 93.25 (4)   | C4—C3—N3    | 122.48 (12) |
| N1—Fe1—N5   | 90.28 (4)   | C4—C3—H1    | 118.76 (8)  |
| N1—Fe1—N6   | 89.73 (4)   | C7—C8—N4    | 112.42 (10) |
| N2—Fe1—N3   | 93.79 (4)   | H5—C8—N4    | 109.12 (6)  |
| N2—Fe1—N4   | 178.85 (4)  | H5—C8—C7    | 109.12 (6)  |
| N2—Fe1—N5   | 95.22 (4)   | H6—C8—N4    | 109.12 (6)  |
| N2—Fe1—N6   | 98.21 (5)   | H6—C8—C7    | 109.12 (7)  |
| N2—Fe1—N1   | 87.56 (4)   | H6—C8—H5    | 107.9       |
| C7—N3—Fe1   | 114.85 (8)  | H14—C10—C9  | 120.53 (8)  |
| C3—N3—Fe1   | 126.42 (9)  | C11—C10—C9  | 118.94 (12) |
| C3—N3—C7    | 118.70 (11) | C11—C10—H14 | 120.53 (8)  |
| C14—N4—Fe1  | 106.57 (7)  | H10—C18—C19 | 120.52 (7)  |
| C20—N4—Fe1  | 105.92 (7)  | C17—C18—C19 | 118.97 (12) |
| C20—N4—C14  | 111.74 (9)  | C17—C18—H10 | 120.52 (8)  |
| C8—N4—Fe1   | 110.47 (7)  | C10—C11—C12 | 118.97 (12) |
| C8—N4—C14   | 111.36 (9)  | H15—C11—C12 | 120.52 (8)  |
| C8—N4—C20   | 110.57 (9)  | H15—C11—C10 | 120.52 (8)  |
| C13—N5—Fe1  | 112.83 (8)  | H9—C17—C18  | 120.58 (8)  |
| C9—N5—Fe1   | 128.64 (9)  | C16—C17—C18 | 118.84 (12) |
| C9—N5—C13   | 118.51 (11) | C16—C17—H9  | 120.58 (8)  |
| C19—N6—Fe1  | 113.35 (8)  | H4—C6—C7    | 120.53 (8)  |
| C15—N6—Fe1  | 127.80 (9)  | C5—C6—C7    | 118.95 (12) |
| C15—N6—C19  | 118.80 (11) | C5—C6—H4    | 120.53 (8)  |
| C1—N1—Fe1   | 167.02 (10) | C17—C16—C15 | 119.11 (12) |
| C2—N2—Fe1   | 170.01 (10) | H8—C16—C15  | 120.45 (8)  |
| C20—C19—N6  | 114.45 (10) | H8—C16—C17  | 120.45 (8)  |
| C18—C19—N6  | 122.12 (11) | H2—C4—C3    | 120.59 (8)  |
| C18—C19—C20 | 123.41 (11) | C5—C4—C3    | 118.83 (13) |
| C14—C13—N5  | 115.09 (10) | C5—C4—H2    | 120.59 (8)  |
| C12—C13—N5  | 122.26 (11) | C4—C5—C6    | 118.98 (12) |
| C12—C13—C14 | 122.55 (11) | H3—C5—C6    | 120.51 (8)  |
| C8—C7—N3    | 116.71 (10) | H3—C5—C4    | 120.51 (8)  |
| C6—C7—N3    | 122.07 (12) | C22—C21—C11 | 119.11 (12) |
| C6—C7—C8    | 121.22 (11) | C26—C21—C11 | 119.16 (13) |
| C13—C14—N4  | 109.62 (9)  | C26—C21—C22 | 121.73 (15) |
| H18—C14—N4  | 109.75 (6)  | H19—C22—C21 | 120.67 (9)  |
| H18—C14—C13 | 109.75 (6)  | C23—C22—C21 | 118.65 (14) |
| H17—C14—N4  | 109.75 (6)  | C23—C22—H19 | 120.67 (9)  |
| H17—C14—C13 | 109.75 (6)  | H20—C23—C22 | 119.82 (9)  |
| H17—C14—H18 | 108.2       | C24—C23—C22 | 120.37 (15) |
| H7—C15—N6   | 118.94 (7)  | C24—C23—H20 | 119.82 (11) |
| C16—C15—N6  | 122.12 (12) | H23—C26—C21 | 120.47 (10) |
| C16—C15—H7  | 118.94 (8)  | C25—C26—C21 | 119.06 (16) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C19—C20—N4      | 107.87 (9)   | C25—C26—H23     | 120.47 (10)  |
| H11—C20—N4      | 110.13 (6)   | H21—C24—C23     | 120.21 (11)  |
| H11—C20—C19     | 110.13 (7)   | C25—C24—C23     | 119.59 (16)  |
| H12—C20—N4      | 110.13 (6)   | C25—C24—H21     | 120.21 (10)  |
| H12—C20—C19     | 110.13 (6)   | C24—C25—C26     | 120.60 (16)  |
| H12—C20—H11     | 108.4        | H22—C25—C26     | 119.70 (10)  |
| N1—C1—S1        | 179.07 (11)  | H22—C25—C24     | 119.70 (10)  |
| H13—C9—N5       | 118.78 (7)   |                 |              |
| Fe1—N3—C7—C8    | −3.82 (9)    | C19—C20—N4—C8   | −78.10 (10)  |
| Fe1—N3—C7—C6    | 177.13 (9)   | C19—C18—C17—C16 | 2.06 (14)    |
| Fe1—N3—C3—C4    | −176.99 (11) | C13—N5—C9—C10   | −0.52 (12)   |
| Fe1—N4—C14—C13  | −36.71 (7)   | C13—C14—N4—C20  | −151.97 (9)  |
| Fe1—N4—C20—C19  | 41.61 (7)    | C13—C14—N4—C8   | 83.82 (10)   |
| Fe1—N4—C8—C7    | 1.02 (8)     | C13—C12—C11—C10 | −1.19 (14)   |
| Fe1—N5—C13—C14  | −5.84 (8)    | C7—N3—C3—C4     | 0.61 (14)    |
| Fe1—N5—C13—C12  | 177.91 (8)   | C7—C8—N4—C14    | −117.20 (10) |
| Fe1—N5—C9—C10   | −178.51 (10) | C7—C8—N4—C20    | 117.95 (10)  |
| Fe1—N6—C19—C20  | 2.89 (8)     | C7—C6—C5—C4     | 0.52 (15)    |
| Fe1—N6—C19—C18  | −178.77 (8)  | C14—C13—N5—C9   | 175.87 (10)  |
| Fe1—N6—C15—C16  | 179.45 (10)  | C14—C13—C12—C11 | −174.73 (12) |
| Cl1—C21—C22—C23 | 179.67 (11)  | C15—N6—C19—C20  | −179.53 (11) |
| Cl1—C21—C26—C25 | 179.88 (13)  | C15—N6—C19—C18  | −1.19 (13)   |
| N3—C7—C8—N4     | 1.79 (12)    | C15—C16—C17—C18 | −1.06 (15)   |
| N3—C7—C6—C5     | 0.18 (14)    | C20—C19—C18—C17 | 177.21 (12)  |
| N3—C3—C4—C5     | 0.09 (16)    | C9—N5—C13—C12   | −0.39 (13)   |
| N4—C14—C13—N5   | 28.95 (11)   | C9—C10—C11—C12  | 0.34 (14)    |
| N4—C14—C13—C12  | −154.81 (9)  | C3—N3—C7—C8     | 178.31 (11)  |
| N4—C20—C19—N6   | −30.10 (11)  | C3—N3—C7—C6     | −0.74 (14)   |
| N4—C20—C19—C18  | 151.58 (9)   | C3—C4—C5—C6     | −0.65 (16)   |
| N4—C8—C7—C6     | −179.15 (10) | C8—C7—C6—C5     | −178.83 (13) |
| N5—C13—C12—C11  | 1.25 (13)    | C21—C22—C23—C24 | 0.31 (17)    |
| N5—C9—C10—C11   | 0.54 (14)    | C21—C26—C25—C24 | 0.58 (19)    |
| N6—C19—C18—C17  | −0.97 (14)   | C22—C21—C26—C25 | −0.07 (18)   |
| N6—C15—C16—C17  | −1.15 (14)   | C22—C23—C24—C25 | 0.18 (18)    |
| C19—N6—C15—C16  | 2.26 (13)    | C23—C22—C21—C26 | −0.37 (17)   |
| C19—C20—N4—C14  | 157.26 (9)   | C23—C24—C25—C26 | −0.63 (18)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_140K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.4125$  (3) Å

$b = 12.6776$  (2) Å

$c = 15.2525$  (3) Å

$\beta = 110.048$  (2)°

$V = 2618.01$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.459$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 50196 reflections

$\theta = 2.0$ – $31.9^\circ$

$\mu = 0.87 \text{ mm}^{-1}$   
 $T = 140 \text{ K}$

Block, red  
 $0.34 \times 0.21 \times 0.17 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.558$ ,  $T_{\max} = 1.000$   
 76522 measured reflections  
 6505 independent reflections  
 5848 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.025$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -16 \rightarrow 16$   
 $l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.081$   
 $S = 1.03$   
 6505 reflections  
 325 parameters  
 0 restraints

43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 1.0012P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = -0.001$   
 $\Delta\rho_{\max} = 1.31 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.87 \text{ e } \text{\AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.339480 (13) | 0.337432 (14) | 0.681111 (12) | 0.01938 (6)                      |
| S2  | 0.52451 (3)   | 0.04097 (3)   | 0.83141 (3)   | 0.03039 (9)                      |
| S1  | 0.25370 (3)   | 0.30207 (3)   | 0.95345 (3)   | 0.03710 (10)                     |
| Cl1 | 0.35403 (4)   | 0.03593 (4)   | 0.44281 (4)   | 0.05407 (12)                     |
| N4  | 0.24823 (8)   | 0.45262 (8)   | 0.61462 (7)   | 0.0205 (2)                       |
| N3  | 0.38988 (8)   | 0.34392 (8)   | 0.57682 (7)   | 0.0218 (2)                       |
| N6  | 0.22987 (8)   | 0.24350 (8)   | 0.60816 (7)   | 0.0214 (2)                       |
| N5  | 0.42622 (8)   | 0.45729 (9)   | 0.73933 (7)   | 0.0223 (2)                       |
| N1  | 0.28901 (8)   | 0.33597 (9)   | 0.78593 (8)   | 0.0244 (2)                       |
| N2  | 0.42757 (8)   | 0.22405 (9)   | 0.74345 (8)   | 0.0262 (2)                       |
| C19 | 0.37771 (9)   | 0.55031 (10)  | 0.72977 (8)   | 0.0225 (2)                       |
| C7  | 0.34968 (9)   | 0.42000 (10)  | 0.51376 (8)   | 0.0219 (2)                       |
| C13 | 0.14429 (9)   | 0.29608 (10)  | 0.56708 (8)   | 0.0221 (2)                       |
| C14 | 0.14646 (9)   | 0.41086 (10)  | 0.59202 (9)   | 0.0244 (2)                       |
| H18 | 0.12197 (9)   | 0.42080 (10)  | 0.65311 (9)   | 0.0292 (3)*                      |
| H17 | 0.09500 (9)   | 0.45559 (10)  | 0.53250 (9)   | 0.0292 (3)*                      |
| C15 | 0.52480 (10)  | 0.45640 (11)  | 0.78232 (9)   | 0.0273 (3)                       |
| H7  | 0.56502 (10)  | 0.38127 (11)  | 0.78791 (9)   | 0.0327 (3)*                      |
| C20 | 0.26779 (9)   | 0.54234 (10)  | 0.68145 (9)   | 0.0237 (2)                       |
| H11 | 0.23870 (9)   | 0.61679 (10)  | 0.64359 (9)   | 0.0285 (3)*                      |
| H12 | 0.23074 (9)   | 0.52857 (10)  | 0.73343 (9)   | 0.0285 (3)*                      |
| C1  | 0.27435 (10)  | 0.32272 (10)  | 0.85575 (9)   | 0.0239 (2)                       |
| C9  | 0.23200 (10)  | 0.13901 (10)  | 0.59388 (9)   | 0.0251 (3)                       |
| H13 | 0.30180 (10)  | 0.09592 (10)  | 0.62687 (9)   | 0.0302 (3)*                      |

|     |               |               |              |             |
|-----|---------------|---------------|--------------|-------------|
| C12 | 0.05901 (10)  | 0.24589 (11)  | 0.51025 (9)  | 0.0261 (3)  |
| H16 | −0.00927 (10) | 0.29125 (11)  | 0.47655 (9)  | 0.0313 (3)* |
| C3  | 0.46130 (10)  | 0.28290 (12)  | 0.56525 (9)  | 0.0278 (3)  |
| H1  | 0.49385 (10)  | 0.21996 (12)  | 0.61650 (9)  | 0.0333 (3)* |
| C2  | 0.46817 (9)   | 0.14829 (10)  | 0.78064 (9)  | 0.0227 (2)  |
| C8  | 0.26837 (10)  | 0.48386 (11)  | 0.52822 (9)  | 0.0252 (3)  |
| H5  | 0.19985 (10)  | 0.47377 (11)  | 0.46683 (9)  | 0.0302 (3)* |
| H6  | 0.28893 (10)  | 0.56867 (11)  | 0.53293 (9)  | 0.0302 (3)* |
| C18 | 0.42593 (10)  | 0.64419 (11)  | 0.76427 (9)  | 0.0275 (3)  |
| H10 | 0.38449 (10)  | 0.71897 (11)  | 0.75433 (9)  | 0.0330 (3)* |
| C10 | 0.14926 (11)  | 0.08324 (11)  | 0.53896 (9)  | 0.0293 (3)  |
| H14 | 0.15338 (11)  | −0.00261 (11) | 0.52913 (9)  | 0.0352 (3)* |
| C11 | 0.06130 (11)  | 0.13754 (11)  | 0.49666 (10) | 0.0294 (3)  |
| H15 | −0.00552 (11) | 0.09537 (11)  | 0.45307 (10) | 0.0353 (3)* |
| C17 | 0.52739 (11)  | 0.64249 (12)  | 0.81160 (10) | 0.0315 (3)  |
| H9  | 0.56735 (11)  | 0.71535 (12)  | 0.84199 (10) | 0.0378 (4)* |
| C6  | 0.38031 (10)  | 0.43821 (12)  | 0.43781 (9)  | 0.0295 (3)  |
| H4  | 0.34605 (10)  | 0.50131 (12)  | 0.38728 (9)  | 0.0354 (3)* |
| C16 | 0.57751 (10)  | 0.54704 (12)  | 0.81987 (10) | 0.0318 (3)  |
| H8  | 0.65815 (10)  | 0.54350 (12)  | 0.85583 (10) | 0.0381 (4)* |
| C4  | 0.49545 (11)  | 0.29622 (13)  | 0.49134 (10) | 0.0328 (3)  |
| H2  | 0.55398 (11)  | 0.24473 (13)  | 0.48387 (10) | 0.0394 (4)* |
| C5  | 0.45448 (11)  | 0.37586 (14)  | 0.42668 (10) | 0.0346 (3)  |
| H3  | 0.48066 (11)  | 0.38908 (14)  | 0.36757 (10) | 0.0415 (4)* |
| C21 | 0.30446 (11)  | 0.12161 (12)  | 0.34915 (11) | 0.0349 (3)  |
| C22 | 0.24645 (11)  | 0.20466 (13)  | 0.35828 (12) | 0.0365 (3)  |
| H19 | 0.23127 (11)  | 0.21651 (13)  | 0.42373 (12) | 0.0438 (4)* |
| C23 | 0.20728 (12)  | 0.27350 (14)  | 0.28287 (13) | 0.0428 (4)  |
| H20 | 0.16104 (12)  | 0.34060 (14)  | 0.28875 (13) | 0.0514 (5)* |
| C26 | 0.32417 (14)  | 0.10507 (14)  | 0.26788 (14) | 0.0473 (4)  |
| H23 | 0.37098 (14)  | 0.03842 (14)  | 0.26221 (14) | 0.0568 (5)* |
| C24 | 0.22647 (14)  | 0.25758 (16)  | 0.20074 (13) | 0.0491 (4)  |
| H21 | 0.19569 (14)  | 0.31190 (16)  | 0.14133 (13) | 0.0590 (5)* |
| C25 | 0.28447 (17)  | 0.17311 (16)  | 0.19369 (14) | 0.0532 (5)  |
| H22 | 0.29912 (17)  | 0.16005 (16)  | 0.12813 (14) | 0.0639 (6)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02175 (10) | 0.02149 (10) | 0.01506 (9)  | 0.00284 (6)   | 0.00652 (7)  | 0.00095 (6)   |
| S2  | 0.02836 (17) | 0.02856 (17) | 0.03489 (18) | 0.00588 (13)  | 0.01164 (14) | 0.00993 (13)  |
| S1  | 0.0585 (2)   | 0.0348 (2)   | 0.02533 (17) | −0.01262 (17) | 0.02384 (17) | −0.00556 (14) |
| Cl1 | 0.0612 (3)   | 0.0429 (2)   | 0.0540 (3)   | 0.0015 (2)    | 0.0145 (2)   | 0.00338 (19)  |
| N4  | 0.0220 (5)   | 0.0203 (5)   | 0.0181 (5)   | 0.0029 (4)    | 0.0053 (4)   | 0.0001 (4)    |
| N3  | 0.0229 (5)   | 0.0259 (5)   | 0.0170 (5)   | 0.0021 (4)    | 0.0073 (4)   | 0.0008 (4)    |
| N6  | 0.0258 (5)   | 0.0216 (5)   | 0.0177 (5)   | 0.0012 (4)    | 0.0086 (4)   | 0.0001 (4)    |
| N5  | 0.0235 (5)   | 0.0256 (5)   | 0.0166 (5)   | 0.0012 (4)    | 0.0051 (4)   | 0.0012 (4)    |
| N1  | 0.0294 (6)   | 0.0264 (5)   | 0.0195 (5)   | −0.0001 (4)   | 0.0113 (4)   | −0.0004 (4)   |

|     |             |             |             |              |            |             |
|-----|-------------|-------------|-------------|--------------|------------|-------------|
| N2  | 0.0284 (5)  | 0.0281 (6)  | 0.0210 (5)  | 0.0062 (4)   | 0.0071 (4) | 0.0043 (4)  |
| C19 | 0.0246 (6)  | 0.0251 (6)  | 0.0174 (5)  | −0.0001 (5)  | 0.0066 (5) | −0.0011 (4) |
| C7  | 0.0243 (6)  | 0.0255 (6)  | 0.0156 (5)  | −0.0009 (5)  | 0.0065 (4) | 0.0011 (4)  |
| C13 | 0.0231 (6)  | 0.0231 (6)  | 0.0198 (5)  | −0.0001 (5)  | 0.0071 (5) | −0.0005 (4) |
| C14 | 0.0227 (6)  | 0.0237 (6)  | 0.0257 (6)  | 0.0027 (5)   | 0.0070 (5) | −0.0016 (5) |
| C15 | 0.0244 (6)  | 0.0335 (7)  | 0.0212 (6)  | 0.0012 (5)   | 0.0043 (5) | 0.0036 (5)  |
| C20 | 0.0251 (6)  | 0.0227 (6)  | 0.0223 (6)  | 0.0022 (5)   | 0.0068 (5) | −0.0025 (5) |
| C1  | 0.0311 (6)  | 0.0233 (6)  | 0.0193 (6)  | −0.0035 (5)  | 0.0111 (5) | −0.0027 (5) |
| C9  | 0.0339 (7)  | 0.0218 (6)  | 0.0211 (6)  | 0.0020 (5)   | 0.0113 (5) | 0.0006 (5)  |
| C12 | 0.0258 (6)  | 0.0276 (7)  | 0.0231 (6)  | −0.0039 (5)  | 0.0059 (5) | −0.0010 (5) |
| C3  | 0.0269 (6)  | 0.0347 (7)  | 0.0233 (6)  | 0.0070 (5)   | 0.0107 (5) | 0.0015 (5)  |
| C2  | 0.0223 (6)  | 0.0256 (6)  | 0.0204 (6)  | 0.0027 (5)   | 0.0075 (5) | 0.0031 (5)  |
| C8  | 0.0290 (6)  | 0.0257 (6)  | 0.0199 (6)  | 0.0042 (5)   | 0.0071 (5) | 0.0042 (5)  |
| C18 | 0.0308 (7)  | 0.0265 (6)  | 0.0243 (6)  | −0.0035 (5)  | 0.0083 (5) | −0.0042 (5) |
| C10 | 0.0414 (8)  | 0.0225 (6)  | 0.0250 (6)  | −0.0035 (6)  | 0.0126 (6) | −0.0027 (5) |
| C11 | 0.0345 (7)  | 0.0282 (7)  | 0.0247 (6)  | −0.0071 (6)  | 0.0090 (5) | −0.0024 (5) |
| C17 | 0.0305 (7)  | 0.0355 (7)  | 0.0266 (6)  | −0.0084 (6)  | 0.0075 (5) | −0.0050 (6) |
| C6  | 0.0328 (7)  | 0.0370 (7)  | 0.0198 (6)  | −0.0017 (6)  | 0.0103 (5) | 0.0043 (5)  |
| C16 | 0.0249 (6)  | 0.0412 (8)  | 0.0249 (6)  | −0.0043 (6)  | 0.0032 (5) | 0.0010 (6)  |
| C4  | 0.0298 (7)  | 0.0460 (9)  | 0.0269 (7)  | 0.0047 (6)   | 0.0150 (6) | −0.0012 (6) |
| C5  | 0.0366 (7)  | 0.0489 (9)  | 0.0233 (6)  | −0.0015 (7)  | 0.0166 (6) | 0.0009 (6)  |
| C21 | 0.0366 (7)  | 0.0319 (7)  | 0.0388 (8)  | −0.0054 (6)  | 0.0166 (6) | −0.0073 (6) |
| C22 | 0.0367 (8)  | 0.0399 (8)  | 0.0384 (8)  | −0.0049 (6)  | 0.0199 (7) | −0.0098 (7) |
| C23 | 0.0297 (7)  | 0.0411 (9)  | 0.0562 (10) | −0.0019 (6)  | 0.0128 (7) | −0.0027 (8) |
| C26 | 0.0588 (10) | 0.0399 (9)  | 0.0580 (11) | −0.0088 (8)  | 0.0388 (9) | −0.0168 (8) |
| C24 | 0.0472 (9)  | 0.0549 (11) | 0.0376 (9)  | −0.0191 (8)  | 0.0046 (7) | 0.0025 (8)  |
| C25 | 0.0757 (13) | 0.0545 (11) | 0.0392 (9)  | −0.0240 (10) | 0.0323 (9) | −0.0132 (8) |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| Fe1—N4  | 1.9940 (10) | C9—H13  | 1.1030      |
| Fe1—N3  | 1.9639 (11) | C9—C10  | 1.3923 (19) |
| Fe1—N6  | 1.9866 (11) | C12—H16 | 1.1030      |
| Fe1—N5  | 1.9746 (11) | C12—C11 | 1.3912 (19) |
| Fe1—N1  | 1.9705 (11) | C3—H1   | 1.1030      |
| Fe1—N2  | 1.9376 (11) | C3—C4   | 1.3863 (19) |
| S2—C2   | 1.6368 (13) | C8—H5   | 1.1110      |
| S1—C1   | 1.6378 (13) | C8—H6   | 1.1110      |
| C11—C21 | 1.7411 (17) | C18—H10 | 1.1030      |
| N4—C14  | 1.4844 (16) | C18—C17 | 1.391 (2)   |
| N4—C20  | 1.4883 (15) | C10—H14 | 1.1030      |
| N4—C8   | 1.4967 (16) | C10—C11 | 1.392 (2)   |
| N3—C7   | 1.3451 (16) | C11—H15 | 1.1030      |
| N3—C3   | 1.3467 (17) | C17—H9  | 1.1030      |
| N6—C13  | 1.3523 (16) | C17—C16 | 1.393 (2)   |
| N6—C9   | 1.3445 (16) | C6—H4   | 1.1030      |
| N5—C19  | 1.3531 (16) | C6—C5   | 1.386 (2)   |
| N5—C15  | 1.3454 (16) | C16—H8  | 1.1030      |



|            |             |             |             |
|------------|-------------|-------------|-------------|
| N1—C1      | 1.1667 (18) | C4—H2       | 1.1030      |
| N2—C2      | 1.1658 (17) | C4—C5       | 1.393 (2)   |
| C19—C20    | 1.5041 (17) | C5—H3       | 1.1030      |
| C19—C18    | 1.3877 (18) | C21—C22     | 1.381 (2)   |
| C7—C8      | 1.5016 (18) | C21—C26     | 1.379 (2)   |
| C7—C6      | 1.3932 (17) | C22—H19     | 1.1030      |
| C13—C14    | 1.5016 (18) | C22—C23     | 1.399 (3)   |
| C13—C12    | 1.3915 (18) | C23—H20     | 1.1030      |
| C14—H18    | 1.1110      | C23—C24     | 1.387 (3)   |
| C14—H17    | 1.1110      | C26—H23     | 1.1030      |
| C15—H7     | 1.1030      | C26—C25     | 1.381 (3)   |
| C15—C16    | 1.388 (2)   | C24—H21     | 1.1030      |
| C20—H11    | 1.1110      | C24—C25     | 1.385 (3)   |
| C20—H12    | 1.1110      | C25—H22     | 1.1030      |
|            |             |             |             |
| N3—Fe1—N4  | 85.41 (4)   | C10—C9—N6   | 122.38 (13) |
| N6—Fe1—N4  | 83.97 (4)   | C10—C9—H13  | 118.81 (8)  |
| N6—Fe1—N3  | 90.88 (4)   | H16—C12—C13 | 120.55 (8)  |
| N5—Fe1—N4  | 82.57 (4)   | C11—C12—C13 | 118.90 (13) |
| N5—Fe1—N3  | 88.80 (4)   | C11—C12—H16 | 120.55 (8)  |
| N5—Fe1—N6  | 166.52 (4)  | H1—C3—N3    | 118.76 (7)  |
| N1—Fe1—N4  | 93.26 (4)   | C4—C3—N3    | 122.48 (13) |
| N1—Fe1—N3  | 178.14 (5)  | C4—C3—H1    | 118.76 (9)  |
| N1—Fe1—N6  | 90.28 (4)   | N2—C2—S2    | 179.16 (12) |
| N1—Fe1—N5  | 89.73 (4)   | C7—C8—N4    | 112.55 (10) |
| N2—Fe1—N4  | 178.89 (4)  | H5—C8—N4    | 109.09 (6)  |
| N2—Fe1—N3  | 93.81 (5)   | H5—C8—C7    | 109.09 (7)  |
| N2—Fe1—N6  | 95.26 (5)   | H6—C8—N4    | 109.09 (7)  |
| N2—Fe1—N5  | 98.21 (5)   | H6—C8—C7    | 109.09 (7)  |
| N2—Fe1—N1  | 87.53 (5)   | H6—C8—H5    | 107.8       |
| C14—N4—Fe1 | 106.49 (8)  | H10—C18—C19 | 120.52 (8)  |
| C20—N4—Fe1 | 105.92 (7)  | C17—C18—C19 | 118.96 (13) |
| C20—N4—C14 | 111.82 (10) | C17—C18—H10 | 120.52 (9)  |
| C8—N4—Fe1  | 110.39 (8)  | H14—C10—C9  | 120.53 (8)  |
| C8—N4—C14  | 111.37 (10) | C11—C10—C9  | 118.93 (13) |
| C8—N4—C20  | 110.64 (10) | C11—C10—H14 | 120.53 (8)  |
| C7—N3—Fe1  | 114.90 (9)  | C10—C11—C12 | 118.93 (13) |
| C3—N3—Fe1  | 126.42 (9)  | H15—C11—C12 | 120.53 (8)  |
| C3—N3—C7   | 118.64 (11) | H15—C11—C10 | 120.53 (8)  |
| C13—N6—Fe1 | 112.83 (8)  | H9—C17—C18  | 120.64 (9)  |
| C9—N6—Fe1  | 128.57 (9)  | C16—C17—C18 | 118.72 (13) |
| C9—N6—C13  | 118.58 (11) | C16—C17—H9  | 120.64 (8)  |
| C19—N5—Fe1 | 113.46 (8)  | H4—C6—C7    | 120.50 (8)  |
| C15—N5—Fe1 | 127.79 (10) | C5—C6—C7    | 119.00 (13) |
| C15—N5—C19 | 118.70 (12) | C5—C6—H4    | 120.50 (8)  |
| C1—N1—Fe1  | 167.07 (11) | C17—C16—C15 | 119.23 (13) |
| C2—N2—Fe1  | 170.09 (11) | H8—C16—C15  | 120.38 (8)  |
| C20—C19—N5 | 114.38 (11) | H8—C16—C17  | 120.38 (8)  |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C18—C19—N5      | 122.24 (12)  | H2—C4—C3        | 120.62 (9)   |
| C18—C19—C20     | 123.36 (12)  | C5—C4—C3        | 118.77 (13)  |
| C8—C7—N3        | 116.62 (11)  | C5—C4—H2        | 120.62 (8)   |
| C6—C7—N3        | 122.13 (12)  | C4—C5—C6        | 118.96 (13)  |
| C6—C7—C8        | 121.24 (12)  | H3—C5—C6        | 120.52 (8)   |
| C14—C13—N6      | 115.07 (11)  | H3—C5—C4        | 120.52 (8)   |
| C12—C13—N6      | 122.26 (12)  | C22—C21—C11     | 119.03 (13)  |
| C12—C13—C14     | 122.56 (12)  | C26—C21—C11     | 119.30 (14)  |
| C13—C14—N4      | 109.73 (10)  | C26—C21—C22     | 121.66 (17)  |
| H18—C14—N4      | 109.72 (6)   | H19—C22—C21     | 120.65 (10)  |
| H18—C14—C13     | 109.72 (7)   | C23—C22—C21     | 118.70 (15)  |
| H17—C14—N4      | 109.72 (6)   | C23—C22—H19     | 120.65 (10)  |
| H17—C14—C13     | 109.72 (7)   | H20—C23—C22     | 119.92 (10)  |
| H17—C14—H18     | 108.2        | C24—C23—C22     | 120.16 (17)  |
| H7—C15—N5       | 118.95 (8)   | C24—C23—H20     | 119.92 (12)  |
| C16—C15—N5      | 122.09 (13)  | H23—C26—C21     | 120.46 (11)  |
| C16—C15—H7      | 118.95 (8)   | C25—C26—C21     | 119.09 (17)  |
| C19—C20—N4      | 107.90 (10)  | C25—C26—H23     | 120.46 (11)  |
| H11—C20—N4      | 110.12 (6)   | H21—C24—C23     | 120.16 (12)  |
| H11—C20—C19     | 110.12 (7)   | C25—C24—C23     | 119.68 (18)  |
| H12—C20—N4      | 110.12 (7)   | C25—C24—H21     | 120.16 (11)  |
| H12—C20—C19     | 110.12 (7)   | C24—C25—C26     | 120.71 (17)  |
| H12—C20—H11     | 108.4        | H22—C25—C26     | 119.65 (11)  |
| N1—C1—S1        | 179.08 (12)  | H22—C25—C24     | 119.65 (11)  |
| H13—C9—N6       | 118.81 (8)   |                 |              |
| Fe1—N4—C14—C13  | −36.69 (8)   | C19—C20—N4—C8   | −78.06 (11)  |
| Fe1—N4—C20—C19  | 41.58 (8)    | C19—C18—C17—C16 | 2.18 (15)    |
| Fe1—N4—C8—C7    | 0.91 (8)     | C7—N3—C3—C4     | 0.60 (14)    |
| Fe1—N3—C7—C8    | −3.83 (10)   | C7—C8—N4—C14    | −117.16 (11) |
| Fe1—N3—C7—C6    | 177.15 (9)   | C7—C8—N4—C20    | 117.82 (11)  |
| Fe1—N3—C3—C4    | −177.00 (12) | C7—C6—C5—C4     | 0.60 (16)    |
| Fe1—N6—C13—C14  | −5.70 (9)    | C13—N6—C9—C10   | −0.42 (13)   |
| Fe1—N6—C13—C12  | 177.90 (8)   | C13—C14—N4—C20  | −151.94 (10) |
| Fe1—N6—C9—C10   | −178.51 (11) | C13—C14—N4—C8   | 83.70 (11)   |
| Fe1—N5—C19—C20  | 2.96 (9)     | C13—C12—C11—C10 | −1.15 (14)   |
| Fe1—N5—C19—C18  | −178.71 (9)  | C14—C13—N6—C9   | 175.92 (11)  |
| Fe1—N5—C15—C16  | 179.39 (11)  | C14—C13—C12—C11 | −174.86 (13) |
| Cl1—C21—C22—C23 | 179.68 (12)  | C15—N5—C19—C20  | −179.54 (11) |
| Cl1—C21—C26—C25 | 179.84 (14)  | C15—N5—C19—C18  | −1.21 (13)   |
| N4—C14—C13—N6   | 28.88 (12)   | C15—C16—C17—C18 | −1.16 (16)   |
| N4—C14—C13—C12  | −154.73 (10) | C20—C19—C18—C17 | 177.15 (13)  |
| N4—C20—C19—N5   | −30.15 (11)  | C9—N6—C13—C12   | −0.48 (13)   |
| N4—C20—C19—C18  | 151.54 (10)  | C9—C10—C11—C12  | 0.31 (15)    |
| N4—C8—C7—N3     | 1.87 (12)    | C3—N3—C7—C8     | 178.31 (12)  |
| N4—C8—C7—C6     | −179.10 (11) | C3—N3—C7—C6     | −0.72 (14)   |
| N3—C7—C6—C5     | 0.12 (15)    | C3—C4—C5—C6     | −0.72 (17)   |
| N3—C3—C4—C5     | 0.12 (17)    | C8—C7—C6—C5     | −178.86 (13) |

|                |             |                 |            |
|----------------|-------------|-----------------|------------|
| N6—C13—C12—C11 | 1.27 (14)   | C21—C22—C23—C24 | 0.30 (18)  |
| N6—C9—C10—C11  | 0.50 (15)   | C21—C26—C25—C24 | 0.7 (2)    |
| N5—C19—C18—C17 | −1.03 (15)  | C22—C21—C26—C25 | −0.28 (19) |
| N5—C15—C16—C17 | −1.12 (15)  | C22—C23—C24—C25 | 0.09 (19)  |
| C19—N5—C15—C16 | 2.30 (13)   | C23—C22—C21—C26 | −0.20 (18) |
| C19—C20—N4—C14 | 157.18 (10) | C23—C24—C25—C26 | −0.58 (19) |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_150K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.4232$  (3) Å

$b = 12.6801$  (2) Å

$c = 15.2608$  (3) Å

$\beta = 110.038$  (2)°

$V = 2622.06$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.456$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 49592 reflections

$\theta = 2.0$ – $32.3^\circ$

$\mu = 0.86$  mm<sup>−1</sup>

$T = 150$  K

Block, red

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.562$ ,  $T_{\max} = 1.000$

76025 measured reflections

6515 independent reflections

5823 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.084$

$S = 1.03$

6515 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 1.115P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.36$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.80$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.660533 (13) | 0.337356 (15) | 0.318832 (12) | 0.02065 (6)                      |
| S2  | 0.47546 (3)   | 0.04113 (3)   | 0.16854 (3)   | 0.03230 (9)                      |
| S1  | 0.74623 (4)   | 0.30213 (3)   | 0.04661 (3)   | 0.03976 (11)                     |
| Cl1 | 0.64604 (4)   | 0.03590 (4)   | 0.55741 (4)   | 0.05800 (14)                     |
| N3  | 0.61019 (8)   | 0.34385 (9)   | 0.42311 (8)   | 0.0230 (2)                       |
| N4  | 0.75167 (8)   | 0.45252 (9)   | 0.38533 (7)   | 0.0218 (2)                       |
| N5  | 0.77012 (8)   | 0.24350 (9)   | 0.39169 (7)   | 0.0228 (2)                       |

|     |              |               |              |             |
|-----|--------------|---------------|--------------|-------------|
| N1  | 0.71100 (9)  | 0.33601 (9)   | 0.21406 (8)  | 0.0260 (2)  |
| N6  | 0.57382 (8)  | 0.45722 (9)   | 0.26074 (7)  | 0.0237 (2)  |
| N2  | 0.57252 (9)  | 0.22392 (10)  | 0.25662 (8)  | 0.0279 (2)  |
| C19 | 0.62234 (10) | 0.55016 (11)  | 0.27034 (9)  | 0.0239 (3)  |
| C7  | 0.65038 (10) | 0.41986 (11)  | 0.48612 (9)  | 0.0232 (3)  |
| C13 | 0.85561 (10) | 0.29605 (11)  | 0.43274 (9)  | 0.0237 (3)  |
| C20 | 0.73212 (10) | 0.54232 (11)  | 0.31858 (9)  | 0.0252 (3)  |
| H11 | 0.76112 (10) | 0.61675 (11)  | 0.35648 (9)  | 0.0302 (3)* |
| H12 | 0.76917 (10) | 0.52866 (11)  | 0.26664 (9)  | 0.0302 (3)* |
| C12 | 0.94096 (10) | 0.24599 (11)  | 0.48966 (10) | 0.0275 (3)  |
| H16 | 1.00915 (10) | 0.29140 (11)  | 0.52338 (10) | 0.0330 (3)* |
| C1  | 0.72557 (10) | 0.32284 (10)  | 0.14427 (10) | 0.0253 (3)  |
| C15 | 0.47542 (10) | 0.45636 (12)  | 0.21776 (9)  | 0.0291 (3)  |
| H7  | 0.43525 (10) | 0.38122 (12)  | 0.21211 (9)  | 0.0349 (3)* |
| C14 | 0.85349 (10) | 0.41084 (11)  | 0.40783 (10) | 0.0256 (3)  |
| H18 | 0.87790 (10) | 0.42081 (11)  | 0.34673 (10) | 0.0308 (3)* |
| H17 | 0.90494 (10) | 0.45555 (11)  | 0.46731 (10) | 0.0308 (3)* |
| C9  | 0.76814 (11) | 0.13904 (11)  | 0.40613 (9)  | 0.0267 (3)  |
| H13 | 0.69839 (11) | 0.09591 (11)  | 0.37328 (9)  | 0.0320 (3)* |
| C2  | 0.53193 (10) | 0.14832 (11)  | 0.21929 (9)  | 0.0241 (3)  |
| C3  | 0.53871 (10) | 0.28278 (12)  | 0.43461 (10) | 0.0293 (3)  |
| H1  | 0.50619 (10) | 0.21984 (12)  | 0.38340 (10) | 0.0351 (3)* |
| C8  | 0.73168 (10) | 0.48374 (11)  | 0.47173 (9)  | 0.0268 (3)  |
| H5  | 0.80015 (10) | 0.47354 (11)  | 0.53305 (9)  | 0.0322 (3)* |
| H6  | 0.71120 (10) | 0.56855 (11)  | 0.46711 (9)  | 0.0322 (3)* |
| C18 | 0.57417 (11) | 0.64410 (12)  | 0.23584 (10) | 0.0289 (3)  |
| H10 | 0.61559 (11) | 0.71886 (12)  | 0.24573 (10) | 0.0346 (3)* |
| C11 | 0.93862 (12) | 0.13773 (12)  | 0.50317 (10) | 0.0312 (3)  |
| H15 | 1.00540 (12) | 0.09555 (12)  | 0.54670 (10) | 0.0374 (4)* |
| C10 | 0.85071 (12) | 0.08333 (12)  | 0.46093 (10) | 0.0314 (3)  |
| H14 | 0.84663 (12) | −0.00250 (12) | 0.47076 (10) | 0.0376 (4)* |
| C6  | 0.61973 (11) | 0.43810 (13)  | 0.56204 (10) | 0.0314 (3)  |
| H4  | 0.65402 (11) | 0.50115 (13)  | 0.61253 (10) | 0.0377 (4)* |
| C17 | 0.47264 (12) | 0.64238 (13)  | 0.18852 (11) | 0.0333 (3)  |
| H9  | 0.43266 (12) | 0.71521 (13)  | 0.15822 (11) | 0.0400 (4)* |
| C4  | 0.50458 (12) | 0.29623 (14)  | 0.50851 (11) | 0.0349 (3)  |
| H2  | 0.44605 (12) | 0.24481 (14)  | 0.51599 (11) | 0.0419 (4)* |
| C16 | 0.42267 (11) | 0.54697 (13)  | 0.18025 (10) | 0.0339 (3)  |
| H8  | 0.34210 (11) | 0.54337 (13)  | 0.14431 (10) | 0.0407 (4)* |
| C22 | 0.75357 (12) | 0.20441 (14)  | 0.64179 (12) | 0.0390 (4)  |
| H19 | 0.76882 (12) | 0.21604 (14)  | 0.57639 (12) | 0.0468 (4)* |
| C5  | 0.54556 (12) | 0.37589 (14)  | 0.57318 (11) | 0.0365 (3)  |
| H3  | 0.51938 (12) | 0.38918 (14)  | 0.63224 (11) | 0.0437 (4)* |
| C21 | 0.69540 (12) | 0.12163 (13)  | 0.65111 (12) | 0.0374 (3)  |
| C23 | 0.79276 (13) | 0.27328 (15)  | 0.71725 (15) | 0.0457 (4)  |
| H20 | 0.83918 (13) | 0.34019 (15)  | 0.71146 (15) | 0.0549 (5)* |
| C26 | 0.67589 (15) | 0.10519 (16)  | 0.73239 (15) | 0.0500 (5)  |
| H23 | 0.62919 (15) | 0.03854 (16)  | 0.73819 (15) | 0.0601 (6)* |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| C24 | 0.77337 (15) | 0.25756 (18) | 0.79912 (14) | 0.0528 (5)  |
| H21 | 0.80392 (15) | 0.31211 (18) | 0.85835 (14) | 0.0634 (6)* |
| C25 | 0.71556 (18) | 0.17322 (17) | 0.80638 (15) | 0.0563 (6)  |
| H22 | 0.70105 (18) | 0.16023 (17) | 0.87198 (15) | 0.0676 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02320 (10) | 0.02273 (11) | 0.01600 (10) | −0.00302 (7)  | 0.00670 (7)  | −0.00092 (6)  |
| S2  | 0.03032 (18) | 0.03019 (18) | 0.0369 (2)   | −0.00618 (14) | 0.01214 (15) | −0.01057 (14) |
| S1  | 0.0631 (3)   | 0.0369 (2)   | 0.02699 (18) | 0.01352 (19)  | 0.02541 (18) | 0.00598 (15)  |
| Cl1 | 0.0661 (3)   | 0.0452 (3)   | 0.0579 (3)   | −0.0016 (2)   | 0.0151 (2)   | −0.0038 (2)   |
| N3  | 0.0243 (5)   | 0.0273 (6)   | 0.0177 (5)   | −0.0022 (4)   | 0.0074 (4)   | −0.0005 (4)   |
| N4  | 0.0235 (5)   | 0.0211 (5)   | 0.0193 (5)   | −0.0029 (4)   | 0.0055 (4)   | 0.0004 (4)    |
| N5  | 0.0276 (5)   | 0.0221 (5)   | 0.0189 (5)   | −0.0013 (4)   | 0.0083 (4)   | −0.0002 (4)   |
| N1  | 0.0313 (6)   | 0.0278 (6)   | 0.0210 (6)   | 0.0002 (4)    | 0.0115 (5)   | 0.0004 (4)    |
| N6  | 0.0251 (5)   | 0.0269 (6)   | 0.0175 (5)   | −0.0011 (4)   | 0.0053 (4)   | −0.0012 (4)   |
| N2  | 0.0302 (6)   | 0.0296 (6)   | 0.0225 (5)   | −0.0064 (5)   | 0.0072 (5)   | −0.0047 (5)   |
| C19 | 0.0262 (6)   | 0.0255 (6)   | 0.0190 (6)   | 0.0003 (5)    | 0.0066 (5)   | 0.0010 (5)    |
| C7  | 0.0258 (6)   | 0.0268 (6)   | 0.0162 (5)   | 0.0013 (5)    | 0.0060 (5)   | −0.0010 (5)   |
| C13 | 0.0248 (6)   | 0.0248 (6)   | 0.0207 (6)   | 0.0008 (5)    | 0.0068 (5)   | 0.0010 (5)    |
| C20 | 0.0270 (6)   | 0.0237 (6)   | 0.0239 (6)   | −0.0024 (5)   | 0.0075 (5)   | 0.0028 (5)    |
| C12 | 0.0274 (7)   | 0.0286 (7)   | 0.0243 (6)   | 0.0039 (5)    | 0.0060 (5)   | 0.0004 (5)    |
| C1  | 0.0331 (7)   | 0.0240 (6)   | 0.0209 (6)   | 0.0036 (5)    | 0.0119 (5)   | 0.0027 (5)    |
| C15 | 0.0258 (7)   | 0.0358 (8)   | 0.0222 (6)   | −0.0018 (6)   | 0.0036 (5)   | −0.0035 (5)   |
| C14 | 0.0233 (6)   | 0.0252 (6)   | 0.0270 (6)   | −0.0029 (5)   | 0.0067 (5)   | 0.0018 (5)    |
| C9  | 0.0360 (7)   | 0.0237 (6)   | 0.0220 (6)   | −0.0027 (5)   | 0.0119 (5)   | −0.0007 (5)   |
| C2  | 0.0240 (6)   | 0.0273 (7)   | 0.0207 (6)   | −0.0028 (5)   | 0.0073 (5)   | −0.0030 (5)   |
| C3  | 0.0286 (7)   | 0.0371 (8)   | 0.0238 (6)   | −0.0070 (6)   | 0.0110 (5)   | −0.0015 (5)   |
| C8  | 0.0307 (7)   | 0.0270 (7)   | 0.0212 (6)   | −0.0044 (5)   | 0.0069 (5)   | −0.0047 (5)   |
| C18 | 0.0322 (7)   | 0.0282 (7)   | 0.0251 (6)   | 0.0039 (6)    | 0.0083 (6)   | 0.0049 (5)    |
| C11 | 0.0365 (8)   | 0.0289 (7)   | 0.0270 (7)   | 0.0076 (6)    | 0.0093 (6)   | 0.0030 (6)    |
| C10 | 0.0440 (8)   | 0.0237 (7)   | 0.0274 (7)   | 0.0028 (6)    | 0.0136 (6)   | 0.0023 (5)    |
| C6  | 0.0351 (7)   | 0.0393 (8)   | 0.0207 (6)   | 0.0022 (6)    | 0.0108 (6)   | −0.0042 (6)   |
| C17 | 0.0327 (8)   | 0.0373 (8)   | 0.0280 (7)   | 0.0088 (6)    | 0.0080 (6)   | 0.0057 (6)    |
| C4  | 0.0319 (7)   | 0.0491 (9)   | 0.0282 (7)   | −0.0052 (7)   | 0.0159 (6)   | 0.0011 (6)    |
| C16 | 0.0267 (7)   | 0.0436 (9)   | 0.0266 (7)   | 0.0039 (6)    | 0.0029 (5)   | −0.0011 (6)   |
| C22 | 0.0397 (8)   | 0.0429 (9)   | 0.0406 (9)   | 0.0055 (7)    | 0.0218 (7)   | 0.0109 (7)    |
| C5  | 0.0376 (8)   | 0.0516 (10)  | 0.0252 (7)   | 0.0019 (7)    | 0.0171 (6)   | −0.0008 (7)   |
| C21 | 0.0386 (8)   | 0.0349 (8)   | 0.0417 (9)   | 0.0063 (6)    | 0.0176 (7)   | 0.0075 (7)    |
| C23 | 0.0323 (8)   | 0.0429 (9)   | 0.0608 (12)  | 0.0013 (7)    | 0.0144 (8)   | 0.0034 (8)    |
| C26 | 0.0616 (11)  | 0.0431 (10)  | 0.0599 (12)  | 0.0083 (9)    | 0.0395 (10)  | 0.0172 (9)    |
| C24 | 0.0521 (11)  | 0.0576 (12)  | 0.0407 (10)  | 0.0206 (9)    | 0.0055 (8)   | −0.0027 (9)   |
| C25 | 0.0812 (15)  | 0.0559 (12)  | 0.0424 (10)  | 0.0249 (11)   | 0.0347 (10)  | 0.0139 (9)    |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N3    | 1.9642 (11) | C14—H18     | 1.1110      |
| Fe1—N4    | 1.9941 (11) | C14—H17     | 1.1110      |
| Fe1—N5    | 1.9866 (12) | C9—H13      | 1.1030      |
| Fe1—N1    | 1.9709 (12) | C9—C10      | 1.391 (2)   |
| Fe1—N6    | 1.9749 (12) | C3—H1       | 1.1030      |
| Fe1—N2    | 1.9380 (12) | C3—C4       | 1.387 (2)   |
| S2—C2     | 1.6365 (14) | C8—H5       | 1.1110      |
| S1—C1     | 1.6380 (14) | C8—H6       | 1.1110      |
| Cl1—C21   | 1.7423 (19) | C18—H10     | 1.1030      |
| N3—C7     | 1.3445 (17) | C18—C17     | 1.393 (2)   |
| N3—C3     | 1.3481 (17) | C11—H15     | 1.1030      |
| N4—C20    | 1.4889 (16) | C11—C10     | 1.392 (2)   |
| N4—C14    | 1.4860 (17) | C10—H14     | 1.1030      |
| N4—C8     | 1.4963 (17) | C6—H4       | 1.1030      |
| N5—C13    | 1.3518 (17) | C6—C5       | 1.386 (2)   |
| N5—C9     | 1.3447 (17) | C17—H9      | 1.1030      |
| N1—C1     | 1.1660 (19) | C17—C16     | 1.392 (2)   |
| N6—C19    | 1.3527 (17) | C4—H2       | 1.1030      |
| N6—C15    | 1.3442 (17) | C4—C5       | 1.394 (2)   |
| N2—C2     | 1.1653 (18) | C16—H8      | 1.1030      |
| C19—C20   | 1.5034 (18) | C22—H19     | 1.1030      |
| C19—C18   | 1.3886 (19) | C22—C21     | 1.381 (2)   |
| C7—C8     | 1.5020 (19) | C22—C23     | 1.401 (3)   |
| C7—C6     | 1.3936 (18) | C5—H3       | 1.1030      |
| C13—C12   | 1.3929 (19) | C21—C26     | 1.378 (2)   |
| C13—C14   | 1.5020 (19) | C23—H20     | 1.1030      |
| C20—H11   | 1.1110      | C23—C24     | 1.385 (3)   |
| C20—H12   | 1.1110      | C26—H23     | 1.1030      |
| C12—H16   | 1.1030      | C26—C25     | 1.380 (3)   |
| C12—C11   | 1.390 (2)   | C24—H21     | 1.1030      |
| C15—H7    | 1.1030      | C24—C25     | 1.384 (3)   |
| C15—C16   | 1.389 (2)   | C25—H22     | 1.1030      |
| N4—Fe1—N3 | 85.37 (5)   | H17—C14—C13 | 109.74 (7)  |
| N5—Fe1—N3 | 90.90 (5)   | H17—C14—H18 | 108.2       |
| N5—Fe1—N4 | 83.95 (5)   | H13—C9—N5   | 118.76 (8)  |
| N1—Fe1—N3 | 178.10 (5)  | C10—C9—N5   | 122.47 (14) |
| N1—Fe1—N4 | 93.26 (5)   | C10—C9—H13  | 118.76 (9)  |
| N1—Fe1—N5 | 90.27 (5)   | N2—C2—S2    | 179.05 (13) |
| N6—Fe1—N3 | 88.76 (5)   | H1—C3—N3    | 118.81 (8)  |
| N6—Fe1—N4 | 82.56 (5)   | C4—C3—N3    | 122.39 (14) |
| N6—Fe1—N5 | 166.48 (5)  | C4—C3—H1    | 118.81 (9)  |
| N6—Fe1—N1 | 89.75 (5)   | C7—C8—N4    | 112.46 (11) |
| N2—Fe1—N3 | 93.79 (5)   | H5—C8—N4    | 109.11 (7)  |
| N2—Fe1—N4 | 178.82 (5)  | H5—C8—C7    | 109.11 (7)  |
| N2—Fe1—N5 | 95.25 (5)   | H6—C8—N4    | 109.11 (7)  |

|              |             |                 |             |
|--------------|-------------|-----------------|-------------|
| N2—Fe1—N1    | 87.60 (5)   | H6—C8—C7        | 109.11 (7)  |
| N2—Fe1—N6    | 98.25 (5)   | H6—C8—H5        | 107.8       |
| C7—N3—Fe1    | 114.90 (9)  | H10—C18—C19     | 120.54 (8)  |
| C3—N3—Fe1    | 126.37 (10) | C17—C18—C19     | 118.91 (14) |
| C3—N3—C7     | 118.69 (12) | C17—C18—H10     | 120.54 (9)  |
| C20—N4—Fe1   | 105.94 (8)  | H15—C11—C12     | 120.49 (9)  |
| C14—N4—Fe1   | 106.50 (8)  | C10—C11—C12     | 119.01 (14) |
| C14—N4—C20   | 111.75 (10) | C10—C11—H15     | 120.49 (9)  |
| C8—N4—Fe1    | 110.47 (8)  | C11—C10—C9      | 118.89 (14) |
| C8—N4—C20    | 110.61 (11) | H14—C10—C9      | 120.56 (9)  |
| C8—N4—C14    | 111.35 (10) | H14—C10—C11     | 120.56 (9)  |
| C13—N5—Fe1   | 112.86 (9)  | H4—C6—C7        | 120.47 (9)  |
| C9—N5—Fe1    | 128.63 (10) | C5—C6—C7        | 119.06 (14) |
| C9—N5—C13    | 118.48 (12) | C5—C6—H4        | 120.47 (9)  |
| C1—N1—Fe1    | 167.03 (12) | H9—C17—C18      | 120.65 (9)  |
| C19—N6—Fe1   | 113.41 (9)  | C16—C17—C18     | 118.70 (14) |
| C15—N6—Fe1   | 127.82 (10) | C16—C17—H9      | 120.65 (9)  |
| C15—N6—C19   | 118.72 (12) | H2—C4—C3        | 120.59 (9)  |
| C2—N2—Fe1    | 170.12 (12) | C5—C4—C3        | 118.82 (14) |
| C20—C19—N6   | 114.48 (12) | C5—C4—H2        | 120.59 (9)  |
| C18—C19—N6   | 122.22 (13) | C17—C16—C15     | 119.24 (14) |
| C18—C19—C20  | 123.27 (12) | H8—C16—C15      | 120.38 (9)  |
| C8—C7—N3     | 116.67 (11) | H8—C16—C17      | 120.38 (9)  |
| C6—C7—N3     | 122.12 (13) | C21—C22—H19     | 120.74 (11) |
| C6—C7—C8     | 121.20 (12) | C23—C22—H19     | 120.74 (10) |
| C12—C13—N5   | 122.36 (13) | C23—C22—C21     | 118.51 (16) |
| C14—C13—N5   | 115.10 (11) | C4—C5—C6        | 118.91 (14) |
| C14—C13—C12  | 122.44 (12) | H3—C5—C6        | 120.55 (9)  |
| C19—C20—N4   | 107.84 (10) | H3—C5—C4        | 120.55 (9)  |
| H11—C20—N4   | 110.14 (7)  | C22—C21—C11     | 118.78 (14) |
| H11—C20—C19  | 110.14 (7)  | C26—C21—C11     | 119.48 (15) |
| H12—C20—N4   | 110.14 (7)  | C26—C21—C22     | 121.74 (18) |
| H12—C20—C19  | 110.14 (7)  | H20—C23—C22     | 119.92 (11) |
| H12—C20—H11  | 108.5       | C24—C23—C22     | 120.17 (18) |
| H16—C12—C13  | 120.62 (8)  | C24—C23—H20     | 119.92 (13) |
| C11—C12—C13  | 118.77 (14) | H23—C26—C21     | 120.44 (12) |
| C11—C12—H16  | 120.62 (9)  | C25—C26—C21     | 119.12 (19) |
| N1—C1—S1     | 179.00 (13) | C25—C26—H23     | 120.44 (12) |
| H7—C15—N6    | 118.92 (8)  | H21—C24—C23     | 120.10 (13) |
| C16—C15—N6   | 122.15 (14) | C25—C24—C23     | 119.80 (19) |
| C16—C15—H7   | 118.92 (9)  | C25—C24—H21     | 120.10 (12) |
| C13—C14—N4   | 109.63 (10) | C24—C25—C26     | 120.66 (18) |
| H18—C14—N4   | 109.74 (7)  | H22—C25—C26     | 119.67 (12) |
| H18—C14—C13  | 109.74 (7)  | H22—C25—C24     | 119.67 (12) |
| H17—C14—N4   | 109.74 (7)  |                 |             |
| Fe1—N3—C7—C8 | −3.84 (10)  | C19—C20—N4—C8   | −78.19 (11) |
| Fe1—N3—C7—C6 | 177.11 (10) | C19—C18—C17—C16 | 2.09 (16)   |



|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Fe1—N3—C3—C4    | −176.94 (12) | C7—N3—C3—C4     | 0.63 (15)    |
| Fe1—N4—C20—C19  | 41.55 (8)    | C7—C8—N4—C20    | 117.81 (12)  |
| Fe1—N4—C14—C13  | −36.75 (8)   | C7—C8—N4—C14    | −117.29 (11) |
| Fe1—N4—C8—C7    | 0.84 (9)     | C7—C6—C5—C4     | 0.63 (17)    |
| Fe1—N5—C13—C12  | 177.82 (9)   | C13—N5—C9—C10   | −0.51 (14)   |
| Fe1—N5—C13—C14  | −5.74 (9)    | C13—C12—C11—C10 | −1.23 (15)   |
| Fe1—N5—C9—C10   | −178.48 (11) | C13—C14—N4—C20  | −151.99 (11) |
| Fe1—N6—C19—C20  | 2.92 (9)     | C13—C14—N4—C8   | 83.75 (12)   |
| Fe1—N6—C19—C18  | −178.77 (9)  | C20—C19—N6—C15  | −179.50 (11) |
| Fe1—N6—C15—C16  | 179.42 (12)  | C20—C19—C18—C17 | 177.18 (14)  |
| Cl1—C21—C22—C23 | 179.78 (13)  | C12—C13—N5—C9   | −0.46 (16)   |
| Cl1—C21—C26—C25 | 179.76 (15)  | C12—C11—C10—C9  | 0.33 (16)    |
| N3—C7—C8—N4     | 1.92 (13)    | C15—N6—C19—C18  | −1.20 (14)   |
| N3—C7—C6—C5     | 0.10 (16)    | C15—C16—C17—C18 | −1.12 (17)   |
| N3—C3—C4—C5     | 0.10 (18)    | C14—C13—N5—C9   | 175.98 (11)  |
| N4—C20—C19—N6   | −30.10 (12)  | C14—C13—C12—C11 | −174.84 (13) |
| N4—C20—C19—C18  | 151.62 (11)  | C3—N3—C7—C8     | 178.32 (13)  |
| N4—C14—C13—N5   | 28.93 (12)   | C3—N3—C7—C6     | −0.73 (15)   |
| N4—C14—C13—C12  | −154.63 (10) | C3—C4—C5—C6     | −0.73 (18)   |
| N4—C8—C7—C6     | −179.02 (11) | C8—C7—C6—C5     | −178.91 (14) |
| N5—C13—C12—C11  | 1.34 (15)    | C22—C21—C26—C25 | −0.0 (2)     |
| N5—C9—C10—C11   | 0.57 (16)    | C22—C23—C24—C25 | 0.4 (2)      |
| N6—C19—C18—C17  | −0.97 (15)   | C21—C22—C23—C24 | 0.26 (19)    |
| N6—C15—C16—C17  | −1.09 (16)   | C21—C26—C25—C24 | 0.7 (2)      |
| C19—N6—C15—C16  | 2.24 (14)    | C23—C22—C21—C26 | −0.4 (2)     |
| C19—C20—N4—C14  | 157.15 (10)  | C23—C24—C25—C26 | −0.8 (2)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_160K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 14.4380$  (3) Å

$b = 12.6792$  (2) Å

$c = 15.2705$  (3) Å

$\beta = 110.018$  (2)°

$V = 2626.57$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.454$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 48201 reflections

$\theta = 2.0$ – $32.3^\circ$

$\mu = 0.86$  mm<sup>−1</sup>

$T = 160$  K

, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.558$ ,  $T_{\max} = 1.000$

76542 measured reflections

6526 independent reflections

5815 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.033$  $wR(F^2) = 0.092$  $S = 1.04$ 

6526 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 1.2523P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = -0.0002$  $\Delta\rho_{\max} = 1.38 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.88 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.339459 (15) | 0.337242 (16) | 0.681283 (13) | 0.02176 (7)                      |
| S2  | 0.52455 (3)   | 0.04115 (3)   | 0.83149 (3)   | 0.03404 (10)                     |
| S1  | 0.25381 (4)   | 0.30230 (4)   | 0.95334 (3)   | 0.04227 (12)                     |
| Cl1 | 0.35378 (5)   | 0.03588 (5)   | 0.44232 (4)   | 0.06193 (16)                     |
| N3  | 0.38978 (9)   | 0.34374 (10)  | 0.57696 (8)   | 0.0243 (2)                       |
| N4  | 0.24840 (9)   | 0.45255 (9)   | 0.61475 (8)   | 0.0227 (2)                       |
| N5  | 0.22986 (9)   | 0.24355 (10)  | 0.60840 (8)   | 0.0240 (2)                       |
| N1  | 0.28903 (10)  | 0.33595 (10)  | 0.78609 (9)   | 0.0274 (3)                       |
| N6  | 0.42617 (9)   | 0.45728 (10)  | 0.73940 (8)   | 0.0249 (2)                       |
| N2  | 0.42742 (10)  | 0.22384 (11)  | 0.74340 (9)   | 0.0296 (3)                       |
| C19 | 0.37772 (11)  | 0.55016 (12)  | 0.72958 (9)   | 0.0251 (3)                       |
| C13 | 0.14442 (11)  | 0.29618 (12)  | 0.56739 (10)  | 0.0249 (3)                       |
| C7  | 0.34961 (10)  | 0.41987 (12)  | 0.51403 (9)   | 0.0243 (3)                       |
| C20 | 0.26804 (11)  | 0.54239 (11)  | 0.68135 (10)  | 0.0260 (3)                       |
| C2  | 0.46809 (11)  | 0.14823 (12)  | 0.78066 (10)  | 0.0255 (3)                       |
| C12 | 0.05917 (11)  | 0.24613 (12)  | 0.51060 (10)  | 0.0292 (3)                       |
| C1  | 0.27445 (11)  | 0.32288 (11)  | 0.85581 (10)  | 0.0268 (3)                       |
| C14 | 0.14666 (10)  | 0.41090 (12)  | 0.59234 (11)  | 0.0268 (3)                       |
| C15 | 0.52454 (11)  | 0.45625 (13)  | 0.78227 (10)  | 0.0307 (3)                       |
| C9  | 0.23166 (12)  | 0.13896 (12)  | 0.59384 (10)  | 0.0282 (3)                       |
| C3  | 0.46125 (11)  | 0.28275 (13)  | 0.56546 (11)  | 0.0310 (3)                       |
| C8  | 0.26837 (11)  | 0.48340 (12)  | 0.52835 (10)  | 0.0280 (3)                       |
| C18 | 0.42589 (12)  | 0.64410 (13)  | 0.76384 (11)  | 0.0304 (3)                       |
| C11 | 0.06143 (13)  | 0.13789 (13)  | 0.49699 (11)  | 0.0328 (3)                       |
| C10 | 0.14911 (13)  | 0.08351 (13)  | 0.53908 (11)  | 0.0331 (3)                       |
| C6  | 0.38015 (12)  | 0.43803 (14)  | 0.43813 (11)  | 0.0332 (3)                       |
| C16 | 0.57712 (12)  | 0.54686 (15)  | 0.81964 (11)  | 0.0357 (4)                       |
| C17 | 0.52708 (13)  | 0.64243 (14)  | 0.81101 (12)  | 0.0353 (3)                       |
| C4  | 0.49527 (13)  | 0.29612 (15)  | 0.49173 (12)  | 0.0369 (4)                       |
| C5  | 0.45432 (13)  | 0.37569 (16)  | 0.42708 (12)  | 0.0389 (4)                       |
| C22 | 0.24648 (14)  | 0.20419 (15)  | 0.35824 (14)  | 0.0413 (4)                       |
| C21 | 0.30453 (13)  | 0.12158 (15)  | 0.34880 (13)  | 0.0395 (4)                       |
| C23 | 0.20713 (14)  | 0.27323 (17)  | 0.28275 (16)  | 0.0486 (5)                       |
| C26 | 0.32409 (17)  | 0.10525 (17)  | 0.26757 (17)  | 0.0535 (5)                       |
| C24 | 0.22651 (17)  | 0.25750 (19)  | 0.20089 (15)  | 0.0557 (6)                       |
| C25 | 0.2844 (2)    | 0.17340 (19)  | 0.19374 (17)  | 0.0600 (6)                       |

|     |               |               |              |             |
|-----|---------------|---------------|--------------|-------------|
| H23 | 0.37078 (17)  | 0.03866 (17)  | 0.26170 (17) | 0.0643 (6)* |
| H22 | 0.2991 (2)    | 0.16061 (19)  | 0.12824 (17) | 0.0721 (8)* |
| H21 | 0.19583 (17)  | 0.31191 (19)  | 0.14163 (15) | 0.0668 (7)* |
| H20 | 0.16071 (14)  | 0.34009 (17)  | 0.28859 (16) | 0.0583 (6)* |
| H19 | 0.23134 (14)  | 0.21573 (15)  | 0.42366 (14) | 0.0495 (5)* |
| H1  | 0.49382 (11)  | 0.21989 (13)  | 0.61669 (11) | 0.0372 (4)* |
| H2  | 0.55375 (13)  | 0.24470 (15)  | 0.48427 (12) | 0.0443 (4)* |
| H3  | 0.48049 (13)  | 0.38888 (16)  | 0.36804 (12) | 0.0467 (5)* |
| H4  | 0.34588 (12)  | 0.50102 (14)  | 0.38760 (11) | 0.0399 (4)* |
| H13 | 0.30124 (12)  | 0.09566 (12)  | 0.62658 (10) | 0.0338 (4)* |
| H14 | 0.15307 (13)  | −0.00233 (13) | 0.52917 (11) | 0.0397 (4)* |
| H15 | −0.00530 (13) | 0.09578 (13)  | 0.45348 (11) | 0.0394 (4)* |
| H16 | −0.00894 (11) | 0.29156 (12)  | 0.47700 (10) | 0.0350 (4)* |
| H7  | 0.56463 (11)  | 0.38108 (13)  | 0.78789 (10) | 0.0368 (4)* |
| H8  | 0.65757 (12)  | 0.54330 (15)  | 0.85574 (11) | 0.0429 (4)* |
| H9  | 0.56699 (13)  | 0.71536 (14)  | 0.84105 (12) | 0.0423 (4)* |
| H10 | 0.38455 (12)  | 0.71890 (13)  | 0.75378 (11) | 0.0365 (4)* |
| H11 | 0.23916 (11)  | 0.61681 (11)  | 0.64342 (10) | 0.0312 (3)* |
| H12 | 0.23099 (11)  | 0.52889 (11)  | 0.73327 (10) | 0.0312 (3)* |
| H18 | 0.12236 (10)  | 0.42086 (12)  | 0.65346 (11) | 0.0322 (3)* |
| H17 | 0.09526 (10)  | 0.45568 (12)  | 0.53298 (11) | 0.0322 (3)* |
| H5  | 0.20001 (11)  | 0.47286 (12)  | 0.46713 (10) | 0.0336 (4)* |
| H6  | 0.28857 (11)  | 0.56828 (12)  | 0.53269 (10) | 0.0336 (4)* |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.02462 (11) | 0.02383 (11) | 0.01689 (10) | 0.00315 (7)  | 0.00718 (8)  | 0.00087 (7)   |
| S2  | 0.0320 (2)   | 0.0319 (2)   | 0.0388 (2)   | 0.00656 (15) | 0.01289 (16) | 0.01133 (16)  |
| S1  | 0.0674 (3)   | 0.0390 (2)   | 0.0286 (2)   | −0.0145 (2)  | 0.0271 (2)   | −0.00645 (17) |
| Cl1 | 0.0714 (4)   | 0.0479 (3)   | 0.0614 (3)   | 0.0012 (3)   | 0.0163 (3)   | 0.0040 (2)    |
| N3  | 0.0255 (6)   | 0.0286 (6)   | 0.0191 (5)   | 0.0025 (5)   | 0.0081 (5)   | 0.0007 (4)    |
| N4  | 0.0248 (6)   | 0.0216 (6)   | 0.0202 (5)   | 0.0034 (4)   | 0.0060 (4)   | −0.0003 (4)   |
| N5  | 0.0292 (6)   | 0.0237 (6)   | 0.0197 (5)   | 0.0018 (5)   | 0.0091 (5)   | 0.0004 (4)    |
| N1  | 0.0334 (6)   | 0.0289 (6)   | 0.0221 (6)   | −0.0003 (5)  | 0.0124 (5)   | −0.0004 (5)   |
| N6  | 0.0263 (6)   | 0.0282 (6)   | 0.0184 (5)   | 0.0011 (5)   | 0.0055 (4)   | 0.0011 (4)    |
| N2  | 0.0326 (6)   | 0.0315 (7)   | 0.0232 (6)   | 0.0071 (5)   | 0.0077 (5)   | 0.0051 (5)    |
| C19 | 0.0286 (7)   | 0.0267 (7)   | 0.0195 (6)   | −0.0001 (5)  | 0.0077 (5)   | −0.0012 (5)   |
| C13 | 0.0269 (7)   | 0.0253 (7)   | 0.0219 (6)   | −0.0002 (5)  | 0.0076 (5)   | −0.0013 (5)   |
| C7  | 0.0272 (6)   | 0.0275 (7)   | 0.0175 (6)   | −0.0012 (5)  | 0.0068 (5)   | 0.0009 (5)    |
| C20 | 0.0280 (7)   | 0.0239 (7)   | 0.0247 (7)   | 0.0031 (5)   | 0.0072 (6)   | −0.0025 (5)   |
| C2  | 0.0257 (7)   | 0.0291 (7)   | 0.0219 (6)   | 0.0030 (5)   | 0.0083 (5)   | 0.0024 (5)    |
| C12 | 0.0290 (7)   | 0.0303 (8)   | 0.0259 (7)   | −0.0043 (6)  | 0.0065 (6)   | −0.0008 (6)   |
| C1  | 0.0357 (8)   | 0.0249 (7)   | 0.0222 (7)   | −0.0039 (6)  | 0.0129 (6)   | −0.0033 (5)   |
| C14 | 0.0242 (6)   | 0.0263 (7)   | 0.0282 (7)   | 0.0029 (5)   | 0.0068 (5)   | −0.0019 (6)   |
| C15 | 0.0279 (7)   | 0.0374 (8)   | 0.0233 (7)   | 0.0010 (6)   | 0.0043 (6)   | 0.0033 (6)    |
| C9  | 0.0381 (8)   | 0.0244 (7)   | 0.0236 (7)   | 0.0028 (6)   | 0.0127 (6)   | 0.0009 (5)    |
| C3  | 0.0302 (7)   | 0.0386 (8)   | 0.0258 (7)   | 0.0073 (6)   | 0.0116 (6)   | 0.0015 (6)    |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C8  | 0.0325 (7)  | 0.0286 (7)  | 0.0217 (6)  | 0.0050 (6)   | 0.0077 (6)  | 0.0049 (5)   |
| C18 | 0.0334 (8)  | 0.0293 (7)  | 0.0273 (7)  | −0.0041 (6)  | 0.0089 (6)  | −0.0047 (6)  |
| C11 | 0.0387 (8)  | 0.0301 (8)  | 0.0283 (7)  | −0.0080 (6)  | 0.0097 (6)  | −0.0031 (6)  |
| C10 | 0.0470 (9)  | 0.0248 (7)  | 0.0285 (7)  | −0.0033 (7)  | 0.0141 (7)  | −0.0024 (6)  |
| C6  | 0.0372 (8)  | 0.0413 (9)  | 0.0220 (7)  | −0.0022 (7)  | 0.0113 (6)  | 0.0044 (6)   |
| C16 | 0.0279 (8)  | 0.0460 (10) | 0.0282 (7)  | −0.0046 (7)  | 0.0031 (6)  | 0.0011 (7)   |
| C17 | 0.0350 (8)  | 0.0390 (9)  | 0.0299 (8)  | −0.0098 (7)  | 0.0085 (6)  | −0.0063 (7)  |
| C4  | 0.0331 (8)  | 0.0520 (10) | 0.0302 (8)  | 0.0049 (7)   | 0.0164 (7)  | −0.0018 (7)  |
| C5  | 0.0405 (9)  | 0.0552 (11) | 0.0267 (8)  | −0.0024 (8)  | 0.0190 (7)  | 0.0007 (7)   |
| C22 | 0.0429 (9)  | 0.0450 (10) | 0.0428 (10) | −0.0067 (8)  | 0.0235 (8)  | −0.0123 (8)  |
| C21 | 0.0408 (9)  | 0.0364 (9)  | 0.0441 (10) | −0.0070 (7)  | 0.0183 (8)  | −0.0085 (7)  |
| C23 | 0.0346 (9)  | 0.0446 (10) | 0.0655 (13) | −0.0013 (8)  | 0.0156 (9)  | −0.0036 (9)  |
| C26 | 0.0653 (13) | 0.0455 (11) | 0.0657 (14) | −0.0087 (10) | 0.0430 (11) | −0.0190 (10) |
| C24 | 0.0551 (12) | 0.0597 (13) | 0.0432 (11) | −0.0224 (10) | 0.0052 (9)  | 0.0030 (10)  |
| C25 | 0.0855 (17) | 0.0605 (14) | 0.0450 (12) | −0.0266 (12) | 0.0364 (12) | −0.0138 (10) |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N3  | 1.9660 (12) | C15—C16 | 1.388 (2) |
| Fe1—N4  | 1.9959 (12) | C15—H7  | 1.1030    |
| Fe1—N5  | 1.9869 (13) | C9—C10  | 1.390 (2) |
| Fe1—N1  | 1.9722 (13) | C9—H13  | 1.1030    |
| Fe1—N6  | 1.9776 (13) | C3—C4   | 1.384 (2) |
| Fe1—N2  | 1.9378 (13) | C3—H1   | 1.1030    |
| S2—C2   | 1.6362 (15) | C8—H5   | 1.1110    |
| S1—C1   | 1.6368 (15) | C8—H6   | 1.1110    |
| C11—C21 | 1.741 (2)   | C18—C17 | 1.390 (2) |
| N3—C7   | 1.3454 (18) | C18—H10 | 1.1030    |
| N3—C3   | 1.3481 (19) | C11—C10 | 1.391 (2) |
| N4—C20  | 1.4882 (17) | C11—H15 | 1.1030    |
| N4—C14  | 1.4864 (18) | C10—H14 | 1.1030    |
| N4—C8   | 1.4957 (18) | C6—C5   | 1.387 (2) |
| N5—C13  | 1.3529 (18) | C6—H4   | 1.1030    |
| N5—C9   | 1.3464 (19) | C16—C17 | 1.394 (3) |
| N1—C1   | 1.166 (2)   | C16—H8  | 1.1030    |
| N6—C19  | 1.3515 (19) | C17—H9  | 1.1030    |
| N6—C15  | 1.3451 (19) | C4—C5   | 1.394 (3) |
| N2—C2   | 1.166 (2)   | C4—H2   | 1.1030    |
| C19—C20 | 1.504 (2)   | C5—H3   | 1.1030    |
| C19—C18 | 1.388 (2)   | C22—C21 | 1.380 (3) |
| C13—C12 | 1.393 (2)   | C22—C23 | 1.403 (3) |
| C13—C14 | 1.501 (2)   | C22—H19 | 1.1030    |
| C7—C8   | 1.499 (2)   | C21—C26 | 1.379 (3) |
| C7—C6   | 1.393 (2)   | C23—C24 | 1.386 (3) |
| C20—H11 | 1.1110      | C23—H20 | 1.1030    |
| C20—H12 | 1.1110      | C26—C25 | 1.380 (4) |
| C12—C11 | 1.390 (2)   | C26—H23 | 1.1030    |
| C12—H16 | 1.1030      | C24—C25 | 1.382 (4) |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C14—H18     | 1.1110      | C24—H21     | 1.1030      |
| C14—H17     | 1.1110      | C25—H22     | 1.1030      |
| N4—Fe1—N3   | 85.33 (5)   | C16—C15—N6  | 122.01 (15) |
| N5—Fe1—N3   | 90.89 (5)   | H7—C15—N6   | 119.00 (9)  |
| N5—Fe1—N4   | 83.87 (5)   | H7—C15—C16  | 119.00 (10) |
| N1—Fe1—N3   | 178.07 (5)  | C10—C9—N5   | 122.45 (15) |
| N1—Fe1—N4   | 93.29 (5)   | H13—C9—N5   | 118.78 (9)  |
| N1—Fe1—N5   | 90.29 (5)   | H13—C9—C10  | 118.78 (9)  |
| N6—Fe1—N3   | 88.77 (5)   | C4—C3—N3    | 122.43 (15) |
| N6—Fe1—N4   | 82.54 (5)   | H1—C3—N3    | 118.79 (9)  |
| N6—Fe1—N5   | 166.39 (5)  | H1—C3—C4    | 118.79 (10) |
| N6—Fe1—N1   | 89.72 (5)   | C7—C8—N4    | 112.63 (11) |
| N2—Fe1—N3   | 93.76 (5)   | H5—C8—N4    | 109.07 (7)  |
| N2—Fe1—N4   | 178.80 (5)  | H5—C8—C7    | 109.07 (8)  |
| N2—Fe1—N5   | 95.36 (5)   | H6—C8—N4    | 109.07 (8)  |
| N2—Fe1—N1   | 87.64 (5)   | H6—C8—C7    | 109.07 (8)  |
| N2—Fe1—N6   | 98.24 (6)   | H6—C8—H5    | 107.8       |
| C7—N3—Fe1   | 114.85 (10) | C17—C18—C19 | 118.96 (15) |
| C3—N3—Fe1   | 126.38 (10) | H10—C18—C19 | 120.52 (9)  |
| C3—N3—C7    | 118.72 (13) | H10—C18—C17 | 120.52 (10) |
| C20—N4—Fe1  | 105.93 (8)  | C10—C11—C12 | 118.99 (15) |
| C14—N4—Fe1  | 106.49 (9)  | H15—C11—C12 | 120.50 (10) |
| C14—N4—C20  | 111.80 (11) | H15—C11—C10 | 120.50 (9)  |
| C8—N4—Fe1   | 110.38 (9)  | C11—C10—C9  | 118.99 (15) |
| C8—N4—C20   | 110.70 (11) | H14—C10—C9  | 120.50 (9)  |
| C8—N4—C14   | 111.32 (11) | H14—C10—C11 | 120.50 (9)  |
| C13—N5—Fe1  | 112.93 (10) | C5—C6—C7    | 118.98 (15) |
| C9—N5—Fe1   | 128.66 (11) | H4—C6—C7    | 120.51 (9)  |
| C9—N5—C13   | 118.39 (13) | H4—C6—C5    | 120.51 (10) |
| C1—N1—Fe1   | 167.07 (13) | C17—C16—C15 | 119.21 (15) |
| C19—N6—Fe1  | 113.37 (10) | H8—C16—C15  | 120.39 (10) |
| C15—N6—Fe1  | 127.71 (11) | H8—C16—C17  | 120.39 (9)  |
| C15—N6—C19  | 118.86 (13) | C16—C17—C18 | 118.75 (15) |
| C2—N2—Fe1   | 170.17 (13) | H9—C17—C18  | 120.63 (10) |
| C20—C19—N6  | 114.55 (13) | H9—C17—C16  | 120.63 (9)  |
| C18—C19—N6  | 122.16 (14) | C5—C4—C3    | 118.82 (16) |
| C18—C19—C20 | 123.26 (13) | H2—C4—C3    | 120.59 (10) |
| C12—C13—N5  | 122.35 (14) | H2—C4—C5    | 120.59 (10) |
| C14—C13—N5  | 115.05 (12) | C4—C5—C6    | 118.97 (15) |
| C14—C13—C12 | 122.50 (13) | H3—C5—C6    | 120.51 (10) |
| C8—C7—N3    | 116.67 (12) | H3—C5—C4    | 120.51 (10) |
| C6—C7—N3    | 122.07 (14) | C23—C22—C21 | 118.51 (18) |
| C6—C7—C8    | 121.25 (13) | H19—C22—C21 | 120.75 (12) |
| C19—C20—N4  | 107.89 (11) | H19—C22—C23 | 120.75 (12) |
| H11—C20—N4  | 110.13 (7)  | C22—C21—C11 | 118.70 (15) |
| H11—C20—C19 | 110.13 (8)  | C26—C21—C11 | 119.50 (16) |
| H12—C20—N4  | 110.13 (8)  | C26—C21—C22 | 121.8 (2)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| H12—C20—C19     | 110.13 (8)   | C24—C23—C22     | 120.1 (2)    |
| H12—C20—H11     | 108.4        | H20—C23—C22     | 119.96 (12)  |
| N2—C2—S2        | 179.14 (14)  | H20—C23—C24     | 119.96 (14)  |
| C11—C12—C13     | 118.82 (15)  | C25—C26—C21     | 119.0 (2)    |
| H16—C12—C13     | 120.59 (9)   | H23—C26—C21     | 120.48 (13)  |
| H16—C12—C11     | 120.59 (10)  | H23—C26—C25     | 120.48 (13)  |
| N1—C1—S1        | 179.00 (14)  | C25—C24—C23     | 119.8 (2)    |
| C13—C14—N4      | 109.66 (11)  | H21—C24—C23     | 120.11 (14)  |
| H18—C14—N4      | 109.74 (7)   | H21—C24—C25     | 120.11 (14)  |
| H18—C14—C13     | 109.74 (8)   | C24—C25—C26     | 120.8 (2)    |
| H17—C14—N4      | 109.74 (7)   | H22—C25—C26     | 119.61 (13)  |
| H17—C14—C13     | 109.74 (8)   | H22—C25—C24     | 119.61 (14)  |
| H17—C14—H18     | 108.2        |                 |              |
| Fe1—N3—C7—C8    | −4.02 (11)   | C19—C20—N4—C8   | −78.18 (12)  |
| Fe1—N3—C7—C6    | 177.17 (10)  | C19—C18—C17—C16 | 2.04 (18)    |
| Fe1—N3—C3—C4    | −176.96 (13) | C13—N5—C9—C10   | −0.51 (15)   |
| Fe1—N4—C20—C19  | 41.49 (9)    | C13—C12—C11—C10 | −1.17 (17)   |
| Fe1—N4—C14—C13  | −36.83 (9)   | C13—C14—N4—C20  | −152.09 (11) |
| Fe1—N4—C8—C7    | 0.58 (9)     | C13—C14—N4—C8   | 83.53 (13)   |
| Fe1—N5—C13—C12  | 177.88 (9)   | C7—N3—C3—C4     | 0.50 (17)    |
| Fe1—N5—C13—C14  | −5.71 (10)   | C7—C8—N4—C20    | 117.55 (13)  |
| Fe1—N5—C9—C10   | −178.50 (12) | C7—C8—N4—C14    | −117.45 (12) |
| Fe1—N6—C19—C20  | 3.03 (10)    | C7—C6—C5—C4     | 0.65 (19)    |
| Fe1—N6—C19—C18  | −178.63 (10) | C20—C19—N6—C15  | −179.58 (12) |
| Fe1—N6—C15—C16  | 179.38 (13)  | C20—C19—C18—C17 | 177.21 (15)  |
| Cl1—C21—C22—C23 | 179.85 (14)  | C12—C13—N5—C9   | −0.41 (17)   |
| Cl1—C21—C26—C25 | 179.77 (17)  | C12—C11—C10—C9  | 0.31 (18)    |
| N3—C7—C8—N4     | 2.21 (14)    | C14—C13—N5—C9   | 175.99 (12)  |
| N3—C7—C6—C5     | −0.00 (17)   | C14—C13—C12—C11 | −174.88 (15) |
| N3—C3—C4—C5     | 0.1 (2)      | C15—N6—C19—C18  | −1.24 (15)   |
| N4—C20—C19—N6   | −30.16 (13)  | C15—C16—C17—C18 | −0.95 (18)   |
| N4—C20—C19—C18  | 151.52 (11)  | C3—N3—C7—C8     | 178.24 (14)  |
| N4—C14—C13—N5   | 28.97 (13)   | C3—N3—C7—C6     | −0.57 (17)   |
| N4—C14—C13—C12  | −154.63 (11) | C3—C4—C5—C6     | −0.7 (2)     |
| N4—C8—C7—C6     | −178.97 (12) | C8—C7—C6—C5     | −178.76 (15) |
| N5—C13—C12—C11  | 1.26 (16)    | C22—C21—C26—C25 | −0.1 (2)     |
| N5—C9—C10—C11   | 0.56 (17)    | C22—C23—C24—C25 | 0.2 (2)      |
| N6—C19—C18—C17  | −0.98 (17)   | C21—C22—C23—C24 | 0.2 (2)      |
| N6—C15—C16—C17  | −1.32 (18)   | C21—C26—C25—C24 | 0.5 (2)      |
| C19—N6—C15—C16  | 2.41 (15)    | C23—C22—C21—C26 | −0.3 (2)     |
| C19—C20—N4—C14  | 157.09 (11)  | C23—C24—C25—C26 | −0.6 (2)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_170K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.2878$  (2) Å

$b = 11.9522$  (1) Å

$c = 15.4638$  (2) Å

$\beta = 110.537$  (2)°

$V = 2646.01$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.443$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 67144 reflections

$\theta = 2.1\text{--}32.2^\circ$

$\mu = 0.86$  mm<sup>-1</sup>

$T = 170$  K

Block, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.536$ ,  $T_{\max} = 1.000$

78174 measured reflections

6554 independent reflections

6169 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -15 \rightarrow 15$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.076$

$S = 1.04$

6554 reflections

387 parameters

39 restraints

55 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 1.5565P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.27$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.53$  e Å<sup>-3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Fe1 | 0.347223 (13) | 0.328292 (15) | 0.699107 (13) | 0.02461 (6)                      |           |
| S2  | 0.53189 (3)   | 0.00221 (3)   | 0.83600 (3)   | 0.03793 (9)                      |           |
| S1  | 0.23069 (10)  | 0.34438 (11)  | 0.95417 (8)   | 0.0688 (2)                       |           |
| Cl1 | 0.40028 (8)   | 0.00085 (8)   | 0.39914 (6)   | 0.0746 (3)                       | 0.722 (2) |
| N3  | 0.39306 (8)   | 0.33743 (9)   | 0.57981 (8)   | 0.0267 (2)                       |           |
| N4  | 0.26204 (7)   | 0.47166 (9)   | 0.61931 (7)   | 0.0237 (2)                       |           |
| N6  | 0.43802 (8)   | 0.47465 (9)   | 0.74754 (7)   | 0.0263 (2)                       |           |
| N5  | 0.21569 (8)   | 0.25030 (9)   | 0.61117 (7)   | 0.0259 (2)                       |           |
| N1  | 0.29460 (9)   | 0.35071 (11)  | 0.80737 (9)   | 0.0361 (3)                       |           |
| N2  | 0.42686 (10)  | 0.19003 (12)  | 0.75487 (9)   | 0.0404 (3)                       |           |
| C19 | 0.39459 (9)   | 0.57396 (11)  | 0.72884 (9)   | 0.0253 (2)                       |           |
| C13 | 0.14438 (9)   | 0.32200 (11)  | 0.57472 (9)   | 0.0260 (3)                       |           |
| C7  | 0.35511 (9)   | 0.41897 (11)  | 0.51880 (9)   | 0.0258 (3)                       |           |
| C18 | 0.44218 (10)  | 0.67468 (12)  | 0.75683 (10)  | 0.0318 (3)                       |           |



|      |               |              |              |             |           |
|------|---------------|--------------|--------------|-------------|-----------|
| C12  | 0.05663 (10)  | 0.28686 (12) | 0.51715 (10) | 0.0318 (3)  |           |
| C20  | 0.29050 (9)   | 0.57027 (11) | 0.68037 (10) | 0.0302 (3)  |           |
| C8   | 0.27782 (10)  | 0.48844 (12) | 0.53106 (9)  | 0.0307 (3)  |           |
| C14  | 0.16378 (9)   | 0.44261 (11) | 0.60324 (10) | 0.0287 (3)  |           |
| C9   | 0.20094 (10)  | 0.14125 (11) | 0.59073 (9)  | 0.0297 (3)  |           |
| C10  | 0.11511 (11)  | 0.09945 (12) | 0.53410 (10) | 0.0360 (3)  |           |
| C17  | 0.53720 (11)  | 0.67163 (13) | 0.80669 (11) | 0.0375 (3)  |           |
| C2   | 0.47073 (9)   | 0.11128 (12) | 0.78827 (9)  | 0.0295 (3)  |           |
| C3   | 0.45966 (10)  | 0.27295 (14) | 0.56749 (10) | 0.0354 (3)  |           |
| C6   | 0.38245 (11)  | 0.43823 (14) | 0.44321 (10) | 0.0361 (3)  |           |
| C11  | 0.04166 (11)  | 0.17367 (13) | 0.49641 (11) | 0.0365 (3)  |           |
| C15  | 0.53020 (10)  | 0.47246 (13) | 0.79311 (10) | 0.0334 (3)  |           |
| C1   | 0.26792 (11)  | 0.34910 (13) | 0.86907 (10) | 0.0345 (3)  |           |
| C16  | 0.58264 (11)  | 0.56886 (14) | 0.82468 (11) | 0.0390 (3)  |           |
| C4   | 0.49049 (11)  | 0.28684 (16) | 0.49410 (12) | 0.0434 (4)  |           |
| C5   | 0.45096 (12)  | 0.37117 (17) | 0.43081 (11) | 0.0441 (4)  |           |
| C26  | 0.3017 (2)    | 0.1322 (4)   | 0.2602 (2)   | 0.0458 (10) | 0.722 (2) |
| C21  | 0.3187 (3)    | 0.1007 (4)   | 0.3503 (3)   | 0.0438 (9)  | 0.722 (2) |
| C23  | 0.2094 (2)    | 0.2341 (3)   | 0.3637 (2)   | 0.0462 (8)  | 0.722 (2) |
| C24  | 0.1922 (3)    | 0.2698 (4)   | 0.2731 (3)   | 0.0484 (9)  | 0.722 (2) |
| C22  | 0.27173 (19)  | 0.1497 (3)   | 0.4014 (2)   | 0.0453 (6)  | 0.722 (2) |
| C25  | 0.2378 (2)    | 0.2190 (3)   | 0.2191 (2)   | 0.0493 (6)  | 0.722 (2) |
| H1   | 0.49111 (10)  | 0.20610 (14) | 0.61802 (10) | 0.0425 (4)* |           |
| H2   | 0.54525 (11)  | 0.23212 (16) | 0.48609 (12) | 0.0520 (4)* |           |
| H3   | 0.47379 (12)  | 0.38462 (17) | 0.37140 (11) | 0.0530 (5)* |           |
| H4   | 0.35002 (11)  | 0.50593 (14) | 0.39391 (10) | 0.0433 (4)* |           |
| H7   | 0.56615 (10)  | 0.39084 (13) | 0.80621 (10) | 0.0401 (4)* |           |
| H8   | 0.65846 (11)  | 0.56391 (14) | 0.86308 (11) | 0.0468 (4)* |           |
| H9   | 0.57629 (11)  | 0.74974 (13) | 0.83178 (11) | 0.0450 (4)* |           |
| H10  | 0.40489 (10)  | 0.75525 (12) | 0.73961 (10) | 0.0382 (3)* |           |
| H13  | 0.25938 (10)  | 0.08228 (11) | 0.62010 (9)  | 0.0357 (3)* |           |
| H14  | 0.10553 (11)  | 0.00908 (12) | 0.51938 (10) | 0.0432 (4)* |           |
| H15  | −0.02728 (11) | 0.14341 (13) | 0.45088 (11) | 0.0437 (4)* |           |
| H16  | −0.00034 (10) | 0.34787 (12) | 0.48830 (10) | 0.0382 (3)* |           |
| H11  | 0.26732 (9)   | 0.64762 (11) | 0.63876 (10) | 0.0362 (3)* |           |
| H12  | 0.25574 (9)   | 0.56748 (11) | 0.73251 (10) | 0.0362 (3)* |           |
| H5   | 0.21182 (10)  | 0.46925 (12) | 0.47340 (9)  | 0.0369 (3)* |           |
| H6   | 0.29394 (10)  | 0.57819 (12) | 0.52539 (9)  | 0.0369 (3)* |           |
| H18  | 0.14851 (9)   | 0.45741 (11) | 0.66745 (10) | 0.0345 (3)* |           |
| H17  | 0.11711 (9)   | 0.49741 (11) | 0.54831 (10) | 0.0345 (3)* |           |
| C23a | 0.1945 (6)    | 0.2876 (7)   | 0.2911 (7)   | 0.074 (5)   | 0.278 (2) |
| Cl1a | 0.3440 (3)    | 0.0382 (3)   | 0.4357 (2)   | 0.1155 (19) | 0.278 (2) |
| C21a | 0.2940 (7)    | 0.1267 (7)   | 0.3429 (7)   | 0.053 (4)   | 0.278 (2) |
| C26a | 0.3030 (6)    | 0.1187 (7)   | 0.2568 (8)   | 0.099 (8)   | 0.278 (2) |
| C22a | 0.2397 (7)    | 0.2112 (9)   | 0.3601 (6)   | 0.070 (4)   | 0.278 (2) |
| C25a | 0.2578 (6)    | 0.1951 (7)   | 0.1878 (6)   | 0.113 (5)   | 0.278 (2) |
| C24a | 0.2035 (6)    | 0.2795 (6)   | 0.2050 (6)   | 0.091 (3)   | 0.278 (2) |
| H19  | 0.28406 (19)  | 0.1212 (3)   | 0.4725 (2)   | 0.0544 (7)* | 0.722 (2) |

|     |            |            |            |              |           |
|-----|------------|------------|------------|--------------|-----------|
| H21 | 0.1725 (2) | 0.2743 (3) | 0.4051 (2) | 0.0555 (10)* | 0.722 (2) |
| H23 | 0.1424 (3) | 0.3385 (4) | 0.2440 (3) | 0.0580 (10)* | 0.722 (2) |
| H25 | 0.2244 (2) | 0.2457 (3) | 0.1473 (2) | 0.0592 (8)*  | 0.722 (2) |
| H27 | 0.3383 (2) | 0.0892 (4) | 0.2197 (2) | 0.0550 (12)* | 0.722 (2) |
| H22 | 0.1515 (6) | 0.3546 (7) | 0.3048 (7) | 0.089 (6)*   | 0.278 (2) |
| H24 | 0.1676 (6) | 0.3402 (6) | 0.1502 (6) | 0.109 (4)*   | 0.278 (2) |
| H26 | 0.2649 (6) | 0.1887 (7) | 0.1194 (6) | 0.136 (5)*   | 0.278 (2) |
| H28 | 0.3460 (6) | 0.0516 (7) | 0.2431 (8) | 0.118 (9)*   | 0.278 (2) |
| H20 | 0.2326 (7) | 0.2176 (9) | 0.4285 (6) | 0.084 (5)*   | 0.278 (2) |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Fe1 | 0.02623 (10) | 0.02240 (10) | 0.02447 (10) | 0.00052 (7)  | 0.00798 (7)  | −0.00143 (7) |
| S2  | 0.03330 (18) | 0.03494 (19) | 0.0494 (2)   | 0.00939 (14) | 0.01931 (16) | 0.00848 (16) |
| S1  | 0.0929 (4)   | 0.0811 (4)   | 0.0485 (3)   | −0.0508 (4)  | 0.0451 (3)   | −0.0302 (3)  |
| Cl1 | 0.0949 (7)   | 0.0634 (5)   | 0.0655 (5)   | 0.0276 (4)   | 0.0281 (4)   | 0.0102 (4)   |
| N3  | 0.0261 (5)   | 0.0290 (6)   | 0.0250 (5)   | 0.0035 (4)   | 0.0088 (4)   | −0.0015 (4)  |
| N4  | 0.0235 (5)   | 0.0201 (5)   | 0.0268 (5)   | 0.0027 (4)   | 0.0082 (4)   | −0.0014 (4)  |
| N6  | 0.0274 (5)   | 0.0236 (5)   | 0.0259 (5)   | 0.0011 (4)   | 0.0067 (4)   | 0.0010 (4)   |
| N5  | 0.0277 (5)   | 0.0224 (5)   | 0.0253 (5)   | 0.0013 (4)   | 0.0065 (4)   | 0.0000 (4)   |
| N1  | 0.0446 (7)   | 0.0332 (6)   | 0.0344 (6)   | −0.0058 (5)  | 0.0186 (6)   | −0.0040 (5)  |
| N2  | 0.0416 (7)   | 0.0371 (7)   | 0.0388 (7)   | 0.0074 (6)   | 0.0096 (6)   | 0.0064 (6)   |
| C19 | 0.0280 (6)   | 0.0216 (6)   | 0.0257 (6)   | −0.0006 (5)  | 0.0088 (5)   | −0.0027 (5)  |
| C13 | 0.0245 (6)   | 0.0237 (6)   | 0.0282 (6)   | −0.0006 (5)  | 0.0070 (5)   | 0.0003 (5)   |
| C7  | 0.0254 (6)   | 0.0291 (6)   | 0.0225 (6)   | −0.0015 (5)  | 0.0077 (5)   | −0.0018 (5)  |
| C18 | 0.0346 (7)   | 0.0248 (7)   | 0.0346 (7)   | −0.0034 (5)  | 0.0104 (6)   | −0.0050 (5)  |
| C12 | 0.0259 (6)   | 0.0308 (7)   | 0.0347 (7)   | −0.0031 (5)  | 0.0056 (5)   | 0.0025 (6)   |
| C20 | 0.0278 (6)   | 0.0230 (6)   | 0.0377 (7)   | 0.0031 (5)   | 0.0090 (5)   | −0.0065 (5)  |
| C8  | 0.0331 (7)   | 0.0306 (7)   | 0.0289 (6)   | 0.0075 (5)   | 0.0114 (5)   | 0.0060 (5)   |
| C14 | 0.0236 (6)   | 0.0245 (6)   | 0.0374 (7)   | 0.0027 (5)   | 0.0099 (5)   | −0.0025 (5)  |
| C9  | 0.0372 (7)   | 0.0214 (6)   | 0.0278 (6)   | 0.0001 (5)   | 0.0078 (5)   | 0.0012 (5)   |
| C10 | 0.0450 (8)   | 0.0248 (7)   | 0.0340 (7)   | −0.0077 (6)  | 0.0085 (6)   | −0.0005 (5)  |
| C17 | 0.0357 (8)   | 0.0339 (8)   | 0.0394 (8)   | −0.0092 (6)  | 0.0088 (6)   | −0.0068 (6)  |
| C2  | 0.0279 (6)   | 0.0292 (7)   | 0.0318 (6)   | 0.0048 (5)   | 0.0109 (5)   | 0.0046 (5)   |
| C3  | 0.0306 (7)   | 0.0410 (8)   | 0.0353 (7)   | 0.0090 (6)   | 0.0124 (6)   | −0.0044 (6)  |
| C6  | 0.0374 (7)   | 0.0448 (8)   | 0.0286 (7)   | −0.0026 (6)  | 0.0147 (6)   | 0.0023 (6)   |
| C11 | 0.0346 (7)   | 0.0328 (8)   | 0.0361 (7)   | −0.0100 (6)  | 0.0050 (6)   | 0.0002 (6)   |
| C15 | 0.0294 (7)   | 0.0321 (7)   | 0.0323 (7)   | 0.0024 (6)   | 0.0029 (5)   | 0.0033 (6)   |
| C1  | 0.0408 (8)   | 0.0346 (7)   | 0.0303 (7)   | −0.0128 (6)  | 0.0153 (6)   | −0.0074 (6)  |
| C16 | 0.0292 (7)   | 0.0418 (9)   | 0.0382 (8)   | −0.0041 (6)  | 0.0021 (6)   | −0.0015 (6)  |
| C4  | 0.0353 (8)   | 0.0564 (10)  | 0.0438 (8)   | 0.0054 (7)   | 0.0206 (7)   | −0.0111 (8)  |
| C5  | 0.0418 (8)   | 0.0622 (11)  | 0.0364 (8)   | −0.0034 (8)  | 0.0237 (7)   | −0.0065 (8)  |
| C26 | 0.054 (2)    | 0.0560 (19)  | 0.0368 (15)  | 0.0107 (17)  | 0.0273 (14)  | 0.0007 (14)  |
| C21 | 0.049 (2)    | 0.0452 (19)  | 0.0406 (18)  | 0.0002 (15)  | 0.0197 (15)  | −0.0070 (14) |
| C23 | 0.0369 (14)  | 0.0531 (19)  | 0.0580 (17)  | −0.0131 (13) | 0.0283 (13)  | −0.0286 (14) |
| C24 | 0.0330 (17)  | 0.0464 (17)  | 0.069 (2)    | −0.0056 (12) | 0.0226 (14)  | −0.0192 (16) |
| C22 | 0.0464 (14)  | 0.0546 (17)  | 0.0433 (14)  | −0.0152 (12) | 0.0263 (12)  | −0.0176 (12) |

|      |             |             |             |             |             |              |
|------|-------------|-------------|-------------|-------------|-------------|--------------|
| C25  | 0.0419 (14) | 0.0553 (18) | 0.0572 (16) | 0.0043 (12) | 0.0253 (12) | −0.0057 (13) |
| C23a | 0.039 (5)   | 0.048 (6)   | 0.131 (10)  | 0.000 (4)   | 0.024 (5)   | −0.035 (5)   |
| Cl1a | 0.132 (3)   | 0.0732 (19) | 0.105 (2)   | −0.032 (2)  | −0.003 (2)  | 0.0305 (17)  |
| C21a | 0.049 (6)   | 0.041 (6)   | 0.055 (5)   | −0.021 (5)  | 0.000 (5)   | 0.016 (5)    |
| C26a | 0.081 (10)  | 0.076 (9)   | 0.176 (17)  | −0.029 (7)  | 0.093 (10)  | −0.086 (10)  |
| C22a | 0.086 (9)   | 0.068 (8)   | 0.084 (8)   | −0.033 (7)  | 0.065 (8)   | −0.043 (6)   |
| C25a | 0.081 (6)   | 0.061 (5)   | 0.235 (13)  | −0.044 (4)  | 0.103 (6)   | −0.084 (6)   |
| C24a | 0.064 (6)   | 0.074 (6)   | 0.133 (9)   | −0.020 (4)  | 0.033 (5)   | −0.050 (5)   |

*Geometric parameters (Å, °)*

|         |             |           |           |
|---------|-------------|-----------|-----------|
| Fe1—N3  | 2.1940 (11) | C10—C11   | 1.389 (2) |
| Fe1—N4  | 2.2423 (11) | C10—H14   | 1.1030    |
| Fe1—N6  | 2.1951 (11) | C17—C16   | 1.390 (2) |
| Fe1—N5  | 2.2007 (11) | C17—H9    | 1.1030    |
| Fe1—N1  | 2.1136 (13) | C3—C4     | 1.383 (2) |
| Fe1—N2  | 2.0543 (14) | C3—H1     | 1.1030    |
| S2—C2   | 1.6230 (14) | C6—C5     | 1.385 (2) |
| S1—C1   | 1.6077 (18) | C6—H4     | 1.1030    |
| Cl1—C21 | 1.700 (4)   | C11—H15   | 1.1030    |
| N3—C7   | 1.3388 (17) | C15—C16   | 1.391 (2) |
| N3—C3   | 1.3427 (17) | C15—H7    | 1.1030    |
| N4—C20  | 1.4770 (16) | C16—H8    | 1.1030    |
| N4—C8   | 1.4804 (17) | C4—C5     | 1.387 (3) |
| N4—C14  | 1.4755 (16) | C4—H2     | 1.1030    |
| N6—C19  | 1.3412 (17) | C5—H3     | 1.1030    |
| N6—C15  | 1.3366 (17) | C26—C21   | 1.376 (4) |
| N5—C13  | 1.3452 (16) | C26—C25   | 1.414 (5) |
| N5—C9   | 1.3414 (17) | C26—H27   | 1.1030    |
| N1—C1   | 1.162 (2)   | C21—C22   | 1.372 (5) |
| N2—C2   | 1.1660 (19) | C23—C24   | 1.398 (5) |
| C19—C18 | 1.3946 (18) | C23—C22   | 1.369 (5) |
| C19—C20 | 1.5030 (18) | C23—H21   | 1.1030    |
| C13—C12 | 1.3895 (18) | C24—C25   | 1.400 (5) |
| C13—C14 | 1.5062 (18) | C24—H23   | 1.1030    |
| C7—C8   | 1.5095 (18) | C22—H19   | 1.1030    |
| C7—C6   | 1.3917 (18) | C25—H25   | 1.1030    |
| C18—C17 | 1.386 (2)   | C23a—C22a | 1.3900    |
| C18—H10 | 1.1030      | C23a—C24a | 1.3900    |
| C12—C11 | 1.391 (2)   | C23a—H22  | 1.1030    |
| C12—H16 | 1.1030      | Cl1a—C21a | 1.730 (9) |
| C20—H11 | 1.1110      | C21a—C26a | 1.3900    |
| C20—H12 | 1.1110      | C21a—C22a | 1.3900    |
| C8—H5   | 1.1110      | C26a—C25a | 1.3900    |
| C8—H6   | 1.1110      | C26a—H28  | 1.1030    |
| C14—H18 | 1.1110      | C22a—H20  | 1.1030    |
| C14—H17 | 1.1110      | C25a—C24a | 1.3900    |
| C9—C10  | 1.390 (2)   | C25a—H26  | 1.1030    |

| C9—H13      | 1.1030      | C24a—H24    | 1.1030      |
|-------------|-------------|-------------|-------------|
| N4—Fe1—N3   | 78.13 (4)   | H13—C9—C10  | 118.62 (9)  |
| N6—Fe1—N3   | 84.08 (4)   | C11—C10—C9  | 118.71 (13) |
| N6—Fe1—N4   | 75.78 (4)   | H14—C10—C9  | 120.64 (9)  |
| N5—Fe1—N3   | 89.64 (4)   | H14—C10—C11 | 120.64 (9)  |
| N5—Fe1—N4   | 75.83 (4)   | C16—C17—C18 | 119.16 (14) |
| N5—Fe1—N6   | 151.61 (4)  | H9—C17—C18  | 120.42 (9)  |
| N1—Fe1—N3   | 169.23 (5)  | H9—C17—C16  | 120.42 (9)  |
| N1—Fe1—N4   | 91.72 (5)   | N2—C2—S2    | 179.30 (14) |
| N1—Fe1—N6   | 89.98 (5)   | C4—C3—N3    | 122.77 (15) |
| N1—Fe1—N5   | 91.37 (5)   | H1—C3—N3    | 118.62 (9)  |
| N2—Fe1—N3   | 94.35 (5)   | H1—C3—C4    | 118.62 (10) |
| N2—Fe1—N4   | 171.91 (5)  | C5—C6—C7    | 119.03 (15) |
| N2—Fe1—N6   | 106.69 (5)  | H4—C6—C7    | 120.49 (9)  |
| N2—Fe1—N5   | 101.37 (5)  | H4—C6—C5    | 120.49 (10) |
| N2—Fe1—N1   | 95.95 (5)   | C10—C11—C12 | 118.77 (13) |
| C7—N3—Fe1   | 116.19 (9)  | H15—C11—C12 | 120.62 (9)  |
| C3—N3—Fe1   | 124.93 (10) | H15—C11—C10 | 120.62 (9)  |
| C3—N3—C7    | 118.73 (12) | C16—C15—N6  | 122.70 (14) |
| C20—N4—Fe1  | 105.90 (8)  | H7—C15—N6   | 118.65 (8)  |
| C8—N4—Fe1   | 111.09 (8)  | H7—C15—C16  | 118.65 (9)  |
| C8—N4—C20   | 111.99 (11) | N1—C1—S1    | 178.93 (15) |
| C14—N4—Fe1  | 105.86 (8)  | C15—C16—C17 | 118.40 (13) |
| C14—N4—C20  | 110.47 (10) | H8—C16—C17  | 120.80 (9)  |
| C14—N4—C8   | 111.24 (10) | H8—C16—C15  | 120.80 (9)  |
| C19—N6—Fe1  | 115.20 (8)  | C5—C4—C3    | 118.46 (15) |
| C15—N6—Fe1  | 126.00 (10) | H2—C4—C3    | 120.77 (10) |
| C15—N6—C19  | 118.80 (12) | H2—C4—C5    | 120.77 (9)  |
| C13—N5—Fe1  | 114.81 (9)  | C4—C5—C6    | 119.11 (14) |
| C9—N5—Fe1   | 126.78 (9)  | H3—C5—C6    | 120.45 (10) |
| C9—N5—C13   | 118.41 (12) | H3—C5—C4    | 120.45 (9)  |
| C1—N1—Fe1   | 171.55 (13) | C25—C26—C21 | 120.7 (4)   |
| C2—N2—Fe1   | 178.07 (13) | H27—C26—C21 | 119.6 (3)   |
| C18—C19—N6  | 122.15 (12) | H27—C26—C25 | 119.64 (18) |
| C20—C19—N6  | 116.04 (11) | C26—C21—C11 | 119.4 (3)   |
| C20—C19—C18 | 121.73 (12) | C22—C21—C11 | 119.6 (3)   |
| C12—C13—N5  | 122.29 (12) | C22—C21—C26 | 121.0 (4)   |
| C14—C13—N5  | 116.08 (11) | C22—C23—C24 | 120.6 (3)   |
| C14—C13—C12 | 121.57 (12) | H21—C23—C24 | 119.7 (2)   |
| C8—C7—N3    | 118.47 (11) | H21—C23—C22 | 119.69 (16) |
| C6—C7—N3    | 121.91 (13) | C25—C24—C23 | 120.5 (4)   |
| C6—C7—C8    | 119.57 (12) | H23—C24—C23 | 119.7 (2)   |
| C17—C18—C19 | 118.75 (13) | H23—C24—C25 | 119.7 (2)   |
| H10—C18—C19 | 120.63 (8)  | C23—C22—C21 | 119.7 (3)   |
| H10—C18—C17 | 120.63 (9)  | H19—C22—C21 | 120.1 (2)   |
| C11—C12—C13 | 119.05 (13) | H19—C22—C23 | 120.14 (16) |
| H16—C12—C13 | 120.47 (8)  | C24—C25—C26 | 117.4 (3)   |

|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| H16—C12—C11     | 120.47 (9)   | H25—C25—C26         | 121.32 (18)  |
| C19—C20—N4      | 111.42 (10)  | H25—C25—C24         | 121.3 (2)    |
| H11—C20—N4      | 109.34 (7)   | C24a—C23a—C22a      | 120.0        |
| H11—C20—C19     | 109.34 (7)   | H22—C23a—C22a       | 120.0        |
| H12—C20—N4      | 109.34 (7)   | H22—C23a—C24a       | 120.0        |
| H12—C20—C19     | 109.34 (8)   | C26a—C21a—C11a      | 126.1 (7)    |
| H12—C20—H11     | 108.0        | C22a—C21a—C11a      | 113.9 (7)    |
| C7—C8—N4        | 115.41 (11)  | C22a—C21a—C26a      | 120.0        |
| H5—C8—N4        | 108.42 (7)   | C25a—C26a—C21a      | 120.0        |
| H5—C8—C7        | 108.42 (7)   | H28—C26a—C21a       | 120.0 (3)    |
| H6—C8—N4        | 108.42 (7)   | H28—C26a—C25a       | 120.0 (3)    |
| H6—C8—C7        | 108.42 (8)   | C21a—C22a—C23a      | 120.0        |
| H6—C8—H5        | 107.5        | H20—C22a—C23a       | 120.0 (3)    |
| C13—C14—N4      | 111.02 (10)  | H20—C22a—C21a       | 120.0 (3)    |
| H18—C14—N4      | 109.43 (7)   | C24a—C25a—C26a      | 120.0        |
| H18—C14—C13     | 109.43 (7)   | H26—C25a—C26a       | 120.0 (3)    |
| H17—C14—N4      | 109.43 (7)   | H26—C25a—C24a       | 120.0 (3)    |
| H17—C14—C13     | 109.43 (7)   | C25a—C24a—C23a      | 120.0        |
| H17—C14—H18     | 108.0        | H24—C24a—C23a       | 120.0 (3)    |
| C10—C9—N5       | 122.76 (13)  | H24—C24a—C25a       | 120.0 (3)    |
| H13—C9—N5       | 118.62 (8)   |                     |              |
| Fe1—N3—C7—C8    | −6.51 (10)   | C13—C14—N4—C20      | −157.68 (11) |
| Fe1—N3—C7—C6    | 175.95 (10)  | C13—C14—N4—C8       | 77.30 (12)   |
| Fe1—N3—C3—C4    | −175.60 (12) | C7—N3—C3—C4         | −0.31 (15)   |
| Fe1—N4—C20—C19  | 42.41 (8)    | C7—C8—N4—C20        | 110.29 (12)  |
| Fe1—N4—C8—C7    | −7.90 (8)    | C7—C8—N4—C14        | −125.55 (12) |
| Fe1—N4—C14—C13  | −43.48 (8)   | C7—C6—C5—C4         | −0.10 (17)   |
| Fe1—N6—C19—C18  | 178.54 (9)   | C18—C19—N6—C15      | −1.17 (16)   |
| Fe1—N6—C19—C20  | 1.75 (10)    | C18—C17—C16—C15     | −1.14 (18)   |
| Fe1—N6—C15—C16  | −177.52 (12) | C12—C13—N5—C9       | 0.09 (16)    |
| Fe1—N5—C13—C12  | 179.59 (9)   | C12—C11—C10—C9      | −0.34 (18)   |
| Fe1—N5—C13—C14  | −3.01 (10)   | C20—C19—N6—C15      | −177.96 (12) |
| Fe1—N5—C9—C10   | −179.85 (11) | C20—C19—C18—C17     | 175.68 (14)  |
| Cl1—C21—C26—C25 | −176.7 (3)   | C8—C7—N3—C3         | 177.80 (13)  |
| Cl1—C21—C22—C23 | 176.7 (3)    | C8—C7—C6—C5         | −177.57 (14) |
| N3—C7—C8—N4     | 9.87 (13)    | C14—C13—N5—C9       | 177.49 (12)  |
| N3—C7—C6—C5     | −0.06 (16)   | C14—C13—C12—C11     | −177.16 (14) |
| N3—C3—C4—C5     | 0.16 (18)    | C3—N3—C7—C6         | 0.26 (15)    |
| N4—C20—C19—N6   | −31.10 (13)  | C3—C4—C5—C6         | 0.05 (19)    |
| N4—C20—C19—C18  | 152.10 (11)  | C26—C21—C22—C23     | −2.5 (4)     |
| N4—C8—C7—C6     | −172.53 (12) | C26—C25—C24—C23     | −0.7 (4)     |
| N4—C14—C13—N5   | 32.65 (13)   | C21—C26—C25—C24     | −0.9 (5)     |
| N4—C14—C13—C12  | −149.93 (11) | C21—C22—C23—C24     | 0.9 (4)      |
| N6—C19—C18—C17  | −0.92 (16)   | C22—C21—C26—C25     | 2.5 (5)      |
| N6—C15—C16—C17  | −1.01 (18)   | C22—C23—C24—C25     | 0.6 (4)      |
| N5—C13—C12—C11  | 0.10 (16)    | C23a—C22a—C21a—C11a | −179.3 (2)   |
| N5—C9—C10—C11   | 0.55 (17)    | C23a—C22a—C21a—C26a | −0.0 (6)     |

|                 |             |                     |           |
|-----------------|-------------|---------------------|-----------|
| C19—N6—C15—C16  | 2.16 (15)   | C23a—C24a—C25a—C26a | 0.0 (7)   |
| C19—C18—C17—C16 | 2.04 (17)   | C11a—C21a—C26a—C25a | 179.2 (2) |
| C19—C20—N4—C8   | −78.82 (12) | C21a—C26a—C25a—C24a | −0.0 (8)  |
| C19—C20—N4—C14  | 156.59 (11) | C21a—C22a—C23a—C24a | 0.0 (6)   |
| C13—N5—C9—C10   | −0.42 (14)  | C22a—C23a—C24a—C25a | −0.0 (8)  |
| C13—C12—C11—C10 | 0.03 (16)   | C22a—C21a—C26a—C25a | 0.0 (7)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_180K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.2799$  (3) Å

$b = 12.0091$  (2) Å

$c = 15.4602$  (2) Å

$\beta = 110.422$  (2)°

$V = 2658.61$  (8) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.436$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 65939 reflections

$\theta = 2.1$ – $32.2^\circ$

$\mu = 0.85$  mm<sup>−1</sup>

$T = 180$  K

Block, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.537$ ,  $T_{\max} = 1.000$

79036 measured reflections

6587 independent reflections

6155 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.068$

$S = 1.05$

6587 reflections

375 parameters

4 restraints

56 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2 + 1.1135P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.0004$

$\Delta\rho_{\max} = 0.83$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.54$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Fe1 | 0.347972 (12) | 0.324898 (14) | 0.198641 (11) | 0.02686 (5)                      |           |
| S2  | 0.53211 (2)   | 0.00115 (3)   | 0.33735 (3)   | 0.04174 (9)                      |           |
| S1  | 0.23648 (9)   | 0.33512 (10)  | 0.45715 (7)   | 0.0785 (2)                       |           |
| N4  | 0.26189 (6)   | 0.46718 (8)   | 0.11970 (7)   | 0.02587 (19)                     |           |
| N3  | 0.39358 (7)   | 0.33557 (8)   | 0.07925 (7)   | 0.0290 (2)                       |           |
| N5  | 0.21702 (7)   | 0.24629 (8)   | 0.11048 (7)   | 0.0283 (2)                       |           |
| N6  | 0.43798 (7)   | 0.47177 (8)   | 0.24692 (7)   | 0.0288 (2)                       |           |

|      |               |              |               |              |           |
|------|---------------|--------------|---------------|--------------|-----------|
| N1   | 0.29589 (9)   | 0.34592 (10) | 0.30743 (8)   | 0.0393 (3)   |           |
| N2   | 0.42812 (9)   | 0.18766 (11) | 0.25403 (9)   | 0.0438 (3)   |           |
| C7   | 0.35560 (8)   | 0.41728 (10) | 0.01903 (8)   | 0.0278 (2)   |           |
| C19  | 0.39353 (8)   | 0.56997 (10) | 0.22924 (8)   | 0.0279 (2)   |           |
| C13  | 0.14548 (8)   | 0.31715 (10) | 0.07408 (8)   | 0.0287 (2)   |           |
| C12  | 0.05813 (9)   | 0.28148 (11) | 0.01606 (9)   | 0.0355 (3)   |           |
| H16  | 0.00087 (9)   | 0.34174 (11) | −0.01297 (9)  | 0.0426 (3)*  |           |
| C14  | 0.16396 (8)   | 0.43704 (10) | 0.10327 (9)   | 0.0317 (2)   |           |
| H18  | 0.14827 (8)   | 0.45070 (10) | 0.16743 (9)   | 0.0380 (3)*  |           |
| H17  | 0.11709 (8)   | 0.49162 (10) | 0.04861 (9)   | 0.0380 (3)*  |           |
| C18  | 0.43997 (9)   | 0.67063 (10) | 0.25716 (9)   | 0.0350 (3)   |           |
| H10  | 0.40177 (9)   | 0.75028 (10) | 0.24072 (9)   | 0.0420 (3)*  |           |
| C9   | 0.20316 (9)   | 0.13770 (10) | 0.08968 (8)   | 0.0330 (3)   |           |
| H13  | 0.26194 (9)   | 0.07948 (10) | 0.11897 (8)   | 0.0397 (3)*  |           |
| C8   | 0.27815 (9)   | 0.48567 (11) | 0.03189 (8)   | 0.0329 (3)   |           |
| H5   | 0.21235 (9)   | 0.46727 (11) | −0.02609 (8)  | 0.0394 (3)*  |           |
| H6   | 0.29434 (9)   | 0.57520 (11) | 0.02733 (8)   | 0.0394 (3)*  |           |
| C20  | 0.28939 (9)   | 0.56473 (10) | 0.18152 (9)   | 0.0330 (3)   |           |
| H11  | 0.26501 (9)   | 0.64201 (10) | 0.14069 (9)   | 0.0396 (3)*  |           |
| H12  | 0.25512 (9)   | 0.55991 (10) | 0.23406 (9)   | 0.0396 (3)*  |           |
| C10  | 0.11782 (10)  | 0.09570 (11) | 0.03279 (9)   | 0.0398 (3)   |           |
| H14  | 0.10885 (10)  | 0.00578 (11) | 0.01776 (9)   | 0.0478 (4)*  |           |
| C1   | 0.27105 (10)  | 0.34217 (11) | 0.37023 (9)   | 0.0382 (3)   |           |
| C2   | 0.47149 (9)   | 0.10934 (11) | 0.28835 (9)   | 0.0325 (2)   |           |
| C3   | 0.46029 (9)   | 0.27213 (12) | 0.06633 (10)  | 0.0384 (3)   |           |
| H1   | 0.49166 (9)   | 0.20502 (12) | 0.11623 (10)  | 0.0461 (3)*  |           |
| C11  | 0.04397 (10)  | 0.16901 (12) | −0.00484 (10) | 0.0408 (3)   |           |
| H15  | −0.02467 (10) | 0.13842 (12) | −0.05049 (10) | 0.0490 (4)*  |           |
| C15  | 0.53041 (9)   | 0.47097 (12) | 0.29153 (9)   | 0.0364 (3)   |           |
| H7   | 0.56732 (9)   | 0.39035 (12) | 0.30382 (9)   | 0.0436 (3)*  |           |
| C6   | 0.38312 (10)  | 0.43785 (13) | −0.05633 (9)  | 0.0394 (3)   |           |
| H4   | 0.35066 (10)  | 0.50569 (13) | −0.10506 (9)  | 0.0473 (4)*  |           |
| C17  | 0.53525 (10)  | 0.66875 (12) | 0.30606 (10)  | 0.0413 (3)   |           |
| H9   | 0.57363 (10)  | 0.74689 (12) | 0.33120 (10)  | 0.0496 (4)*  |           |
| C16  | 0.58172 (10)  | 0.56730 (13) | 0.32297 (10)  | 0.0425 (3)   |           |
| H8   | 0.65770 (10)  | 0.56340 (13) | 0.36053 (10)  | 0.0510 (4)*  |           |
| Cl1a | 0.34303 (14)  | 0.03781 (13) | −0.06168 (12) | 0.0903 (8)   | 0.387 (2) |
| C4   | 0.49142 (10)  | 0.28719 (15) | −0.00675 (11) | 0.0471 (3)   |           |
| H2   | 0.54631 (10)  | 0.23325 (15) | −0.01519 (11) | 0.0565 (4)*  |           |
| C5   | 0.45188 (11)  | 0.37183 (15) | −0.06918 (10) | 0.0479 (4)   |           |
| H3   | 0.47491 (11)  | 0.38639 (15) | −0.12837 (10) | 0.0575 (4)*  |           |
| C22  | 0.27176 (17)  | 0.1528 (3)   | −0.10182 (15) | 0.0538 (7)   | 0.613 (2) |
| H19  | 0.28479 (17)  | 0.1262 (3)   | −0.03010 (15) | 0.0645 (9)*  | 0.613 (2) |
| C21  | 0.3191 (2)    | 0.1017 (2)   | −0.1532 (2)   | 0.0529 (10)  | 0.613 (2) |
| C26  | 0.3026 (2)    | 0.1351 (3)   | −0.2436 (2)   | 0.0569 (13)  | 0.613 (2) |
| H27  | 0.3402 (2)    | 0.0946 (3)   | −0.2844 (2)   | 0.0683 (15)* | 0.613 (2) |
| C25  | 0.23892 (18)  | 0.2196 (3)   | −0.28259 (17) | 0.0568 (8)   | 0.613 (2) |
| H25  | 0.22589 (18)  | 0.2462 (3)   | −0.35431 (17) | 0.0681 (10)* | 0.613 (2) |



|      |              |             |              |              |           |
|------|--------------|-------------|--------------|--------------|-----------|
| C24  | 0.19162 (17) | 0.2707 (2)  | −0.2312 (2)  | 0.0594 (13)  | 0.613 (2) |
| H23  | 0.14105 (17) | 0.3378 (2)  | −0.2621 (2)  | 0.0712 (15)* | 0.613 (2) |
| C23  | 0.20804 (17) | 0.2373 (3)  | −0.1408 (2)  | 0.0567 (11)  | 0.613 (2) |
| H21  | 0.17050 (17) | 0.2778 (3)  | −0.1000 (2)  | 0.0680 (13)* | 0.613 (2) |
| C21a | 0.2939 (3)   | 0.1263 (3)  | −0.1499 (3)  | 0.0388 (12)  | 0.387 (2) |
| C26a | 0.3061 (3)   | 0.1137 (3)  | −0.2343 (3)  | 0.0486 (18)  | 0.387 (2) |
| H28  | 0.3498 (3)   | 0.0454 (3)  | −0.2444 (3)  | 0.058 (2)*   | 0.387 (2) |
| C25a | 0.2632 (3)   | 0.1872 (4)  | −0.3061 (2)  | 0.0596 (14)  | 0.387 (2) |
| H26  | 0.2729 (3)   | 0.1772 (4)  | −0.3731 (2)  | 0.0715 (16)* | 0.387 (2) |
| C24a | 0.2081 (3)   | 0.2733 (3)  | −0.2934 (3)  | 0.0625 (14)  | 0.387 (2) |
| H24  | 0.1741 (3)   | 0.3317 (3)  | −0.3503 (3)  | 0.0750 (17)* | 0.387 (2) |
| C23a | 0.1959 (3)   | 0.2860 (3)  | −0.2089 (4)  | 0.0556 (18)  | 0.387 (2) |
| H22  | 0.1522 (3)   | 0.3543 (3)  | −0.1989 (4)  | 0.067 (2)*   | 0.387 (2) |
| C22a | 0.2388 (3)   | 0.2124 (4)  | −0.1372 (3)  | 0.0491 (13)  | 0.387 (2) |
| H20  | 0.2291 (3)   | 0.2224 (4)  | −0.0702 (3)  | 0.0589 (16)* | 0.387 (2) |
| Cl1  | 0.40109 (9)  | 0.00461 (9) | −0.10025 (7) | 0.0897 (4)   | 0.613 (2) |

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

|      | $U^{11}$     | $U^{22}$     | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------|--------------|--------------|-------------|--------------|--------------|--------------|
| Fe1  | 0.02846 (9)  | 0.02558 (9)  | 0.02568 (9) | 0.00034 (6)  | 0.00837 (7)  | −0.00138 (6) |
| S2   | 0.03576 (17) | 0.03853 (18) | 0.0551 (2)  | 0.00995 (13) | 0.02108 (15) | 0.00987 (15) |
| S1   | 0.1088 (4)   | 0.0941 (4)   | 0.0508 (2)  | −0.0604 (3)  | 0.0507 (3)   | −0.0333 (3)  |
| N4   | 0.0253 (4)   | 0.0233 (4)   | 0.0289 (5)  | 0.0026 (4)   | 0.0093 (4)   | −0.0019 (4)  |
| N3   | 0.0277 (5)   | 0.0326 (5)   | 0.0270 (5)  | 0.0035 (4)   | 0.0096 (4)   | −0.0016 (4)  |
| N5   | 0.0302 (5)   | 0.0260 (5)   | 0.0265 (5)  | 0.0009 (4)   | 0.0071 (4)   | −0.0001 (4)  |
| N6   | 0.0297 (5)   | 0.0271 (5)   | 0.0272 (5)  | 0.0005 (4)   | 0.0067 (4)   | 0.0005 (4)   |
| N1   | 0.0485 (6)   | 0.0375 (6)   | 0.0362 (6)  | −0.0072 (5)  | 0.0201 (5)   | −0.0038 (5)  |
| N2   | 0.0444 (7)   | 0.0406 (7)   | 0.0421 (6)  | 0.0082 (5)   | 0.0099 (5)   | 0.0073 (5)   |
| C7   | 0.0277 (5)   | 0.0313 (6)   | 0.0245 (5)  | −0.0021 (4)  | 0.0090 (4)   | −0.0021 (4)  |
| C19  | 0.0305 (6)   | 0.0251 (5)   | 0.0273 (5)  | −0.0008 (4)  | 0.0092 (4)   | −0.0027 (4)  |
| C13  | 0.0267 (5)   | 0.0275 (6)   | 0.0303 (6)  | −0.0008 (4)  | 0.0077 (4)   | 0.0011 (4)   |
| C12  | 0.0285 (6)   | 0.0350 (6)   | 0.0381 (6)  | −0.0042 (5)  | 0.0054 (5)   | 0.0027 (5)   |
| C14  | 0.0259 (5)   | 0.0283 (6)   | 0.0398 (6)  | 0.0027 (4)   | 0.0103 (5)   | −0.0026 (5)  |
| C18  | 0.0385 (7)   | 0.0280 (6)   | 0.0372 (6)  | −0.0043 (5)  | 0.0115 (5)   | −0.0061 (5)  |
| C9   | 0.0416 (7)   | 0.0256 (6)   | 0.0290 (6)  | −0.0006 (5)  | 0.0087 (5)   | 0.0016 (4)   |
| C8   | 0.0351 (6)   | 0.0329 (6)   | 0.0304 (6)  | 0.0071 (5)   | 0.0112 (5)   | 0.0050 (5)   |
| C20  | 0.0307 (6)   | 0.0270 (6)   | 0.0396 (6)  | 0.0028 (5)   | 0.0103 (5)   | −0.0075 (5)  |
| C10  | 0.0497 (8)   | 0.0288 (6)   | 0.0365 (6)  | −0.0092 (6)  | 0.0094 (6)   | −0.0006 (5)  |
| C1   | 0.0460 (7)   | 0.0396 (7)   | 0.0315 (6)  | −0.0155 (6)  | 0.0165 (6)   | −0.0078 (5)  |
| C2   | 0.0303 (6)   | 0.0330 (6)   | 0.0348 (6)  | 0.0045 (5)   | 0.0119 (5)   | 0.0049 (5)   |
| C3   | 0.0324 (6)   | 0.0444 (7)   | 0.0390 (7)  | 0.0084 (5)   | 0.0132 (5)   | −0.0046 (6)  |
| C11  | 0.0380 (7)   | 0.0382 (7)   | 0.0393 (7)  | −0.0120 (6)  | 0.0048 (6)   | 0.0004 (5)   |
| C15  | 0.0319 (6)   | 0.0363 (7)   | 0.0340 (6)  | 0.0018 (5)   | 0.0030 (5)   | 0.0029 (5)   |
| C6   | 0.0409 (7)   | 0.0498 (8)   | 0.0304 (6)  | −0.0041 (6)  | 0.0161 (5)   | 0.0015 (6)   |
| C17  | 0.0396 (7)   | 0.0387 (7)   | 0.0420 (7)  | −0.0112 (6)  | 0.0097 (6)   | −0.0074 (6)  |
| C16  | 0.0322 (6)   | 0.0466 (8)   | 0.0407 (7)  | −0.0056 (6)  | 0.0024 (5)   | −0.0019 (6)  |
| Cl1a | 0.1122 (14)  | 0.0588 (8)   | 0.0799 (10) | −0.0200 (8)  | 0.0085 (9)   | 0.0202 (7)   |

|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| C4   | 0.0378 (7)  | 0.0618 (9)  | 0.0478 (8)  | 0.0054 (7)   | 0.0226 (6)  | −0.0120 (7)  |
| C5   | 0.0451 (8)  | 0.0682 (10) | 0.0393 (7)  | −0.0035 (7)  | 0.0260 (6)  | −0.0059 (7)  |
| C22  | 0.0530 (16) | 0.066 (2)   | 0.0525 (17) | −0.0198 (15) | 0.0307 (14) | −0.0249 (16) |
| C21  | 0.061 (2)   | 0.0505 (19) | 0.058 (2)   | −0.0023 (15) | 0.0344 (16) | −0.0144 (15) |
| C26  | 0.064 (3)   | 0.061 (2)   | 0.056 (3)   | 0.0126 (19)  | 0.033 (2)   | −0.001 (2)   |
| C25  | 0.0508 (18) | 0.063 (2)   | 0.064 (2)   | 0.0056 (15)  | 0.0295 (16) | −0.0016 (18) |
| C24  | 0.0373 (19) | 0.054 (2)   | 0.090 (3)   | −0.0063 (15) | 0.0264 (18) | −0.027 (2)   |
| C23  | 0.0368 (17) | 0.067 (2)   | 0.074 (2)   | −0.0126 (16) | 0.0290 (15) | −0.038 (2)   |
| C21a | 0.053 (3)   | 0.030 (2)   | 0.039 (2)   | −0.0123 (19) | 0.024 (2)   | −0.0087 (17) |
| C26a | 0.065 (4)   | 0.043 (3)   | 0.058 (4)   | −0.005 (2)   | 0.046 (3)   | −0.012 (2)   |
| C25a | 0.078 (4)   | 0.053 (3)   | 0.058 (3)   | −0.013 (3)   | 0.037 (3)   | −0.009 (2)   |
| C24a | 0.045 (3)   | 0.049 (3)   | 0.078 (4)   | −0.006 (2)   | 0.003 (3)   | −0.004 (3)   |
| C23a | 0.038 (3)   | 0.053 (3)   | 0.075 (4)   | −0.004 (2)   | 0.018 (2)   | −0.017 (3)   |
| C22a | 0.051 (3)   | 0.042 (3)   | 0.065 (3)   | −0.008 (3)   | 0.034 (3)   | −0.017 (2)   |
| Cl1  | 0.1185 (9)  | 0.0707 (6)  | 0.0750 (6)  | 0.0298 (6)   | 0.0276 (6)  | 0.0115 (5)   |

*Geometric parameters (Å, °)*

|         |             |           |             |
|---------|-------------|-----------|-------------|
| Fe1—N4  | 2.2403 (10) | C10—C11   | 1.389 (2)   |
| Fe1—N3  | 2.1925 (10) | C3—H1     | 1.1030      |
| Fe1—N5  | 2.2021 (10) | C3—C4     | 1.382 (2)   |
| Fe1—N6  | 2.2007 (10) | C11—H15   | 1.1030      |
| Fe1—N1  | 2.1119 (12) | C15—H7    | 1.1030      |
| Fe1—N2  | 2.0536 (12) | C15—C16   | 1.3868 (19) |
| S2—C2   | 1.6217 (13) | C6—H4     | 1.1030      |
| S1—C1   | 1.6085 (17) | C6—C5     | 1.385 (2)   |
| N4—C14  | 1.4730 (15) | C17—H9    | 1.1030      |
| N4—C8   | 1.4796 (15) | C17—C16   | 1.388 (2)   |
| N4—C20  | 1.4772 (14) | C16—H8    | 1.1030      |
| N3—C7   | 1.3377 (15) | Cl1a—C21a | 1.685 (3)   |
| N3—C3   | 1.3422 (16) | C4—H2     | 1.1030      |
| N5—C13  | 1.3440 (15) | C4—C5     | 1.387 (2)   |
| N5—C9   | 1.3421 (16) | C5—H3     | 1.1030      |
| N6—C19  | 1.3406 (15) | C22—H19   | 1.1030      |
| N6—C15  | 1.3382 (16) | C22—C21   | 1.3900      |
| N1—C1   | 1.1601 (18) | C22—C23   | 1.3900      |
| N2—C2   | 1.1663 (17) | C21—C26   | 1.3900      |
| C7—C8   | 1.5096 (16) | C21—Cl1   | 1.698 (2)   |
| C7—C6   | 1.3917 (16) | C26—H27   | 1.1030      |
| C19—C18 | 1.3919 (16) | C26—C25   | 1.3900      |
| C19—C20 | 1.5037 (17) | C25—H25   | 1.1030      |
| C13—C12 | 1.3907 (17) | C25—C24   | 1.3900      |
| C13—C14 | 1.5057 (16) | C24—H23   | 1.1030      |
| C12—H16 | 1.1030      | C24—C23   | 1.3900      |
| C12—C11 | 1.3880 (19) | C23—H21   | 1.1030      |
| C14—H18 | 1.1110      | C21a—C26a | 1.3900      |
| C14—H17 | 1.1110      | C21a—C22a | 1.3900      |
| C18—H10 | 1.1030      | C26a—H28  | 1.1030      |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C18—C17     | 1.3869 (19) | C26a—C25a   | 1.3900      |
| C9—H13      | 1.1030      | C25a—H26    | 1.1030      |
| C9—C10      | 1.3886 (18) | C25a—C24a   | 1.3900      |
| C8—H5       | 1.1110      | C24a—H24    | 1.1030      |
| C8—H6       | 1.1110      | C24a—C23a   | 1.3900      |
| C20—H11     | 1.1110      | C23a—H22    | 1.1030      |
| C20—H12     | 1.1110      | C23a—C22a   | 1.3900      |
| C10—H14     | 1.1030      | C22a—H20    | 1.1030      |
|             |             |             |             |
| N3—Fe1—N4   | 78.20 (4)   | H12—C20—H11 | 108.0       |
| N5—Fe1—N4   | 75.87 (4)   | H14—C10—C9  | 120.62 (8)  |
| N5—Fe1—N3   | 89.49 (4)   | C11—C10—C9  | 118.77 (12) |
| N6—Fe1—N4   | 75.65 (4)   | C11—C10—H14 | 120.62 (8)  |
| N6—Fe1—N3   | 83.92 (4)   | N1—C1—S1    | 179.21 (14) |
| N6—Fe1—N5   | 151.50 (4)  | N2—C2—S2    | 179.27 (13) |
| N1—Fe1—N4   | 91.71 (4)   | H1—C3—N3    | 118.55 (8)  |
| N1—Fe1—N3   | 169.21 (4)  | C4—C3—N3    | 122.90 (14) |
| N1—Fe1—N5   | 91.76 (4)   | C4—C3—H1    | 118.55 (9)  |
| N1—Fe1—N6   | 89.92 (4)   | C10—C11—C12 | 118.78 (12) |
| N2—Fe1—N4   | 172.13 (4)  | H15—C11—C12 | 120.61 (8)  |
| N2—Fe1—N3   | 94.56 (5)   | H15—C11—C10 | 120.61 (8)  |
| N2—Fe1—N5   | 101.24 (4)  | H7—C15—N6   | 118.64 (7)  |
| N2—Fe1—N6   | 106.89 (5)  | C16—C15—N6  | 122.72 (13) |
| N2—Fe1—N1   | 95.71 (5)   | C16—C15—H7  | 118.64 (8)  |
| C14—N4—Fe1  | 105.93 (7)  | H4—C6—C7    | 120.51 (8)  |
| C8—N4—Fe1   | 111.08 (7)  | C5—C6—C7    | 118.98 (13) |
| C8—N4—C14   | 111.37 (9)  | C5—C6—H4    | 120.51 (9)  |
| C20—N4—Fe1  | 105.78 (7)  | H9—C17—C18  | 120.41 (8)  |
| C20—N4—C14  | 110.43 (9)  | C16—C17—C18 | 119.19 (12) |
| C20—N4—C8   | 111.94 (10) | C16—C17—H9  | 120.41 (8)  |
| C7—N3—Fe1   | 116.15 (8)  | C17—C16—C15 | 118.46 (12) |
| C3—N3—Fe1   | 124.97 (9)  | H8—C16—C15  | 120.77 (8)  |
| C3—N3—C7    | 118.71 (11) | H8—C16—C17  | 120.77 (8)  |
| C13—N5—Fe1  | 114.73 (8)  | H2—C4—C3    | 120.85 (9)  |
| C9—N5—Fe1   | 126.73 (8)  | C5—C4—C3    | 118.31 (13) |
| C9—N5—C13   | 118.53 (11) | C5—C4—H2    | 120.85 (8)  |
| C19—N6—Fe1  | 115.07 (8)  | C4—C5—C6    | 119.20 (13) |
| C15—N6—Fe1  | 126.25 (9)  | H3—C5—C6    | 120.40 (9)  |
| C15—N6—C19  | 118.68 (11) | H3—C5—C4    | 120.40 (8)  |
| C1—N1—Fe1   | 170.43 (12) | C21—C22—H19 | 120.0       |
| C2—N2—Fe1   | 176.86 (12) | C23—C22—H19 | 120.0       |
| C8—C7—N3    | 118.49 (10) | C23—C22—C21 | 120.0       |
| C6—C7—N3    | 121.90 (12) | C26—C21—C22 | 120.0       |
| C6—C7—C8    | 119.57 (11) | Cl1—C21—C22 | 118.1 (2)   |
| C18—C19—N6  | 122.25 (11) | Cl1—C21—C26 | 121.8 (2)   |
| C20—C19—N6  | 115.93 (10) | H27—C26—C21 | 120.00 (10) |
| C20—C19—C18 | 121.74 (11) | C25—C26—C21 | 120.0       |
| C12—C13—N5  | 122.16 (11) | C25—C26—H27 | 120.00 (10) |

|                |              |                     |              |
|----------------|--------------|---------------------|--------------|
| C14—C13—N5     | 116.10 (10)  | H25—C25—C26         | 120.00 (10)  |
| C14—C13—C12    | 121.68 (11)  | C24—C25—C26         | 120.0        |
| H16—C12—C13    | 120.45 (8)   | C24—C25—H25         | 120.00 (10)  |
| C11—C12—C13    | 119.11 (12)  | H23—C24—C25         | 120.00 (10)  |
| C11—C12—H16    | 120.45 (8)   | C23—C24—C25         | 120.0        |
| C13—C14—N4     | 111.08 (9)   | C23—C24—H23         | 120.00 (10)  |
| H18—C14—N4     | 109.42 (6)   | C24—C23—C22         | 120.0        |
| H18—C14—C13    | 109.42 (7)   | H21—C23—C22         | 120.00 (10)  |
| H17—C14—N4     | 109.42 (6)   | H21—C23—C24         | 120.00 (10)  |
| H17—C14—C13    | 109.42 (7)   | C26a—C21a—C11a      | 121.6        |
| H17—C14—H18    | 108.0        | C22a—C21a—C11a      | 118.4        |
| H10—C18—C19    | 120.68 (7)   | C22a—C21a—C26a      | 120.0        |
| C17—C18—C19    | 118.64 (12)  | H28—C26a—C21a       | 120.00 (16)  |
| C17—C18—H10    | 120.68 (8)   | C25a—C26a—C21a      | 120.0        |
| H13—C9—N5      | 118.68 (7)   | C25a—C26a—H28       | 120.00 (16)  |
| C10—C9—N5      | 122.65 (12)  | H26—C25a—C26a       | 120.00 (16)  |
| C10—C9—H13     | 118.68 (8)   | C24a—C25a—C26a      | 120.0        |
| C7—C8—N4       | 115.44 (10)  | C24a—C25a—H26       | 120.00 (16)  |
| H5—C8—N4       | 108.42 (6)   | H24—C24a—C25a       | 120.00 (16)  |
| H5—C8—C7       | 108.42 (6)   | C23a—C24a—C25a      | 120.0        |
| H6—C8—N4       | 108.42 (6)   | C23a—C24a—H24       | 120.00 (16)  |
| H6—C8—C7       | 108.42 (7)   | H22—C23a—C24a       | 120.00 (16)  |
| H6—C8—H5       | 107.5        | C22a—C23a—C24a      | 120.0        |
| C19—C20—N4     | 111.35 (9)   | C22a—C23a—H22       | 120.00 (16)  |
| H11—C20—N4     | 109.36 (6)   | C23a—C22a—C21a      | 120.0        |
| H11—C20—C19    | 109.36 (7)   | H20—C22a—C21a       | 120.00 (16)  |
| H12—C20—N4     | 109.36 (6)   | H20—C22a—C23a       | 120.00 (16)  |
| H12—C20—C19    | 109.36 (7)   |                     |              |
| Fe1—N4—C14—C13 | −43.42 (7)   | C13—N5—C9—C10       | −0.53 (13)   |
| Fe1—N4—C8—C7   | −7.23 (8)    | C13—C12—C11—C10     | −0.20 (15)   |
| Fe1—N4—C20—C19 | 43.14 (7)    | C13—C14—N4—C8       | 77.46 (11)   |
| Fe1—N3—C7—C8   | −6.76 (9)    | C13—C14—N4—C20      | −157.51 (10) |
| Fe1—N3—C7—C6   | 175.63 (9)   | C12—C13—N5—C9       | 0.10 (15)    |
| Fe1—N3—C3—C4   | −175.22 (11) | C12—C11—C10—C9      | −0.20 (16)   |
| Fe1—N5—C13—C12 | 179.32 (8)   | C14—C13—N5—C9       | 177.35 (11)  |
| Fe1—N5—C13—C14 | −3.44 (9)    | C14—C13—C12—C11     | −176.83 (13) |
| Fe1—N5—C9—C10  | −179.64 (10) | C18—C19—N6—C15      | −1.19 (14)   |
| Fe1—N6—C19—C18 | 178.43 (8)   | C18—C17—C16—C15     | −1.15 (17)   |
| Fe1—N6—C19—C20 | 1.42 (9)     | C8—C7—N3—C3         | 177.80 (11)  |
| Fe1—N6—C15—C16 | −177.37 (11) | C8—C7—C6—C5         | −177.65 (13) |
| N4—C14—C13—N5  | 32.89 (12)   | C20—C19—N6—C15      | −178.20 (11) |
| N4—C14—C13—C12 | −149.85 (10) | C20—C19—C18—C17     | 175.90 (13)  |
| N4—C8—C7—N3    | 9.57 (13)    | C3—N3—C7—C6         | 0.19 (14)    |
| N4—C8—C7—C6    | −172.77 (11) | C3—C4—C5—C6         | −0.00 (17)   |
| N4—C20—C19—N6  | −31.33 (12)  | C11a—C21a—C26a—C25a | 178.92 (12)  |
| N4—C20—C19—C18 | 151.64 (10)  | C11a—C21a—C22a—C23a | −178.96 (12) |
| N3—C7—C6—C5    | −0.07 (14)   | C22—C21—C26—C25     | 0.0 (3)      |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N3—C3—C4—C5     | 0.13 (17)    | C22—C23—C24—C25     | 0.0 (3)     |
| N5—C13—C12—C11  | 0.26 (14)    | C21—C22—C23—C24     | 0.00 (19)   |
| N5—C9—C10—C11   | 0.59 (15)    | C21—C26—C25—C24     | −0.0 (3)    |
| N6—C19—C18—C17  | −0.94 (14)   | C26—C21—C22—C23     | −0.0 (3)    |
| N6—C15—C16—C17  | −1.03 (16)   | C26—C25—C24—C23     | −0.00 (19)  |
| C7—N3—C3—C4     | −0.22 (14)   | C25—C26—C21—C11     | −176.06 (9) |
| C7—C8—N4—C14    | −125.05 (11) | C23—C22—C21—C11     | 176.21 (9)  |
| C7—C8—N4—C20    | 110.77 (11)  | C21a—C26a—C25a—C24a | −0.0 (4)    |
| C7—C6—C5—C4     | −0.02 (16)   | C21a—C22a—C23a—C24a | 0.0 (3)     |
| C19—N6—C15—C16  | 2.20 (13)    | C26a—C21a—C22a—C23a | 0.0 (3)     |
| C19—C18—C17—C16 | 2.08 (15)    | C26a—C25a—C24a—C23a | 0.0 (3)     |
| C19—C20—N4—C14  | 157.32 (10)  | C25a—C26a—C21a—C22a | −0.0 (4)    |
| C19—C20—N4—C8   | −77.98 (11)  | C25a—C24a—C23a—C22a | −0.0 (3)    |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_190K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.2759$  (2) Å

$b = 12.0449$  (1) Å

$c = 15.4697$  (2) Å

$\beta = 110.312$  (2)°

$V = 2669.38$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.431$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 65948 reflections

$\theta = 2.1$ – $32.1^\circ$

$\mu = 0.85$  mm<sup>−1</sup>

$T = 190$  K

Block, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.535$ ,  $T_{\max} = 1.000$

78961 measured reflections

6617 independent reflections

6189 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.064$

$S = 1.04$

6617 reflections

375 parameters

2 restraints

56 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 1.0037P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.0001$

$\Delta\rho_{\max} = 0.70$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.57$  e Å<sup>−3</sup>

*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  | 0.348425 (11) | 0.323116 (13) | 0.198558 (11) | 0.02818 (5)                      |             |
| S2   | 0.53236 (2)   | 0.00090 (3)   | 0.33812 (3)   | 0.04428 (8)                      |             |
| S1   | 0.23968 (9)   | 0.33030 (10)  | 0.45870 (7)   | 0.08252 (19)                     |             |
| N4   | 0.26184 (6)   | 0.46485 (7)   | 0.11998 (6)   | 0.02739 (18)                     |             |
| N3   | 0.39385 (7)   | 0.33455 (8)   | 0.07904 (6)   | 0.03059 (19)                     |             |
| N6   | 0.43797 (7)   | 0.47037 (8)   | 0.24666 (6)   | 0.03044 (19)                     |             |
| N5   | 0.21773 (7)   | 0.24425 (8)   | 0.11027 (6)   | 0.02977 (19)                     |             |
| Cl1  | 0.40134 (11)  | 0.00670 (10)  | −0.09983 (8)  | 0.0993 (5)                       | 0.5501 (19) |
| N1   | 0.29647 (8)   | 0.34354 (9)   | 0.30754 (8)   | 0.0413 (2)                       |             |
| N2   | 0.42883 (8)   | 0.18649 (10)  | 0.25369 (8)   | 0.0460 (3)                       |             |
| C7   | 0.35580 (7)   | 0.41633 (10)  | 0.01923 (7)   | 0.0297 (2)                       |             |
| C19  | 0.39294 (8)   | 0.56792 (9)   | 0.22930 (7)   | 0.0295 (2)                       |             |
| C13  | 0.14606 (8)   | 0.31464 (9)   | 0.07396 (8)   | 0.0305 (2)                       |             |
| C12  | 0.05899 (8)   | 0.27886 (11)  | 0.01577 (9)   | 0.0380 (3)                       |             |
| C14  | 0.16393 (8)   | 0.43413 (10)  | 0.10335 (9)   | 0.0335 (2)                       |             |
| C8   | 0.27831 (8)   | 0.48402 (10)  | 0.03241 (8)   | 0.0346 (2)                       |             |
| C20  | 0.28879 (8)   | 0.56190 (10)  | 0.18212 (9)   | 0.0345 (2)                       |             |
| C9   | 0.20423 (9)   | 0.13599 (10)  | 0.08936 (8)   | 0.0347 (2)                       |             |
| C18  | 0.43878 (9)   | 0.66857 (10)  | 0.25711 (9)   | 0.0370 (3)                       |             |
| C3   | 0.46076 (9)   | 0.27164 (12)  | 0.06593 (9)   | 0.0405 (3)                       |             |
| C1   | 0.27286 (10)  | 0.33850 (11)  | 0.37104 (9)   | 0.0400 (3)                       |             |
| C2   | 0.47199 (8)   | 0.10854 (10)  | 0.28845 (8)   | 0.0343 (2)                       |             |
| C10  | 0.11918 (10)  | 0.09390 (11)  | 0.03225 (9)   | 0.0419 (3)                       |             |
| C15  | 0.53039 (9)   | 0.47032 (11)  | 0.29075 (8)   | 0.0382 (3)                       |             |
| C11  | 0.04522 (10)  | 0.16670 (11)  | −0.00527 (9)  | 0.0431 (3)                       |             |
| C17  | 0.53403 (10)  | 0.66747 (11)  | 0.30550 (10)  | 0.0437 (3)                       |             |
| C6   | 0.38348 (9)   | 0.43737 (12)  | −0.05593 (9)  | 0.0418 (3)                       |             |
| C16  | 0.58112 (9)   | 0.56679 (12)  | 0.32203 (9)   | 0.0448 (3)                       |             |
| C4   | 0.49185 (10)  | 0.28748 (14)  | −0.00714 (10) | 0.0499 (3)                       |             |
| C5   | 0.45230 (10)  | 0.37206 (15)  | −0.06904 (10) | 0.0511 (4)                       |             |
| C26  | 0.3033 (2)    | 0.1354 (3)    | −0.2441 (2)   | 0.0629 (15)                      | 0.5501 (19) |
| C24  | 0.19164 (19)  | 0.2704 (3)    | −0.2328 (3)   | 0.0649 (15)                      | 0.5501 (19) |
| C21  | 0.3194 (2)    | 0.1028 (3)    | −0.1537 (3)   | 0.0577 (11)                      | 0.5501 (19) |
| C23  | 0.20772 (19)  | 0.2378 (3)    | −0.1424 (2)   | 0.0660 (14)                      | 0.5501 (19) |
| C25  | 0.2394 (2)    | 0.2192 (3)    | −0.2837 (2)   | 0.0606 (9)                       | 0.5501 (19) |
| C22  | 0.2716 (2)    | 0.1540 (3)    | −0.10287 (18) | 0.0600 (9)                       | 0.5501 (19) |
| C22a | 0.2388 (3)    | 0.2124 (3)    | −0.1375 (2)   | 0.0513 (11)                      | 0.4499 (19) |
| Cl1a | 0.34280 (11)  | 0.03728 (10)  | −0.06108 (9)  | 0.0877 (6)                       | 0.4499 (19) |
| C21a | 0.2937 (3)    | 0.1261 (2)    | −0.1497 (2)   | 0.0408 (10)                      | 0.4499 (19) |
| C26a | 0.3064 (3)    | 0.1130 (3)    | −0.2339 (3)   | 0.0508 (14)                      | 0.4499 (19) |
| C23a | 0.1966 (3)    | 0.2857 (3)    | −0.2093 (3)   | 0.0573 (14)                      | 0.4499 (19) |
| C25a | 0.2642 (3)    | 0.1863 (3)    | −0.3057 (2)   | 0.0600 (11)                      | 0.4499 (19) |
| C24a | 0.2093 (2)    | 0.2726 (3)    | −0.2934 (2)   | 0.0622 (11)                      | 0.4499 (19) |
| H1   | 0.49223 (9)   | 0.20454 (12)  | 0.11551 (9)   | 0.0486 (3)*                      |             |
| H2   | 0.54681 (10)  | 0.23404 (14)  | −0.01592 (10) | 0.0599 (4)*                      |             |

|     |               |              |               |              |             |
|-----|---------------|--------------|---------------|--------------|-------------|
| H3  | 0.47542 (10)  | 0.38718 (15) | −0.12810 (10) | 0.0613 (4)*  |             |
| H4  | 0.35100 (9)   | 0.50516 (12) | −0.10438 (9)  | 0.0501 (3)*  |             |
| H7  | 0.56779 (9)   | 0.39028 (11) | 0.30274 (8)   | 0.0459 (3)*  |             |
| H8  | 0.65713 (9)   | 0.56354 (12) | 0.35917 (9)   | 0.0538 (4)*  |             |
| H9  | 0.57193 (10)  | 0.74566 (11) | 0.33056 (10)  | 0.0524 (4)*  |             |
| H10 | 0.40009 (9)   | 0.74766 (10) | 0.24098 (9)   | 0.0444 (3)*  |             |
| H13 | 0.26306 (9)   | 0.07811 (10) | 0.11873 (8)   | 0.0416 (3)*  |             |
| H14 | 0.11057 (10)  | 0.00429 (11) | 0.01697 (9)   | 0.0503 (3)*  |             |
| H15 | −0.02324 (10) | 0.13601 (11) | −0.05095 (9)  | 0.0517 (4)*  |             |
| H16 | 0.00165 (8)   | 0.33877 (11) | −0.01327 (9)  | 0.0456 (3)*  |             |
| H18 | 0.14793 (8)   | 0.44727 (10) | 0.16739 (9)   | 0.0403 (3)*  |             |
| H17 | 0.11701 (8)   | 0.48850 (10) | 0.04882 (9)   | 0.0403 (3)*  |             |
| H5  | 0.21263 (8)   | 0.46588 (10) | −0.02558 (8)  | 0.0415 (3)*  |             |
| H6  | 0.29444 (8)   | 0.57338 (10) | 0.02826 (8)   | 0.0415 (3)*  |             |
| H11 | 0.26379 (8)   | 0.63905 (10) | 0.14172 (9)   | 0.0414 (3)*  |             |
| H12 | 0.25481 (8)   | 0.55605 (10) | 0.23486 (9)   | 0.0414 (3)*  |             |
| H19 | 0.2844 (2)    | 0.1281 (3)   | −0.03114 (18) | 0.0720 (11)* | 0.5501 (19) |
| H21 | 0.16978 (19)  | 0.2785 (3)   | −0.1021 (2)   | 0.0792 (17)* | 0.5501 (19) |
| H23 | 0.14094 (19)  | 0.3370 (3)   | −0.2642 (3)   | 0.0779 (18)* | 0.5501 (19) |
| H25 | 0.2267 (2)    | 0.2451 (3)   | −0.3554 (2)   | 0.0728 (11)* | 0.5501 (19) |
| H27 | 0.3413 (2)    | 0.0947 (3)   | −0.2845 (2)   | 0.0755 (18)* | 0.5501 (19) |
| H20 | 0.2287 (3)    | 0.2228 (3)   | −0.0707 (2)   | 0.0615 (13)* | 0.4499 (19) |
| H22 | 0.1530 (3)    | 0.3542 (3)   | −0.1996 (3)   | 0.0688 (17)* | 0.4499 (19) |
| H24 | 0.1758 (2)    | 0.3307 (3)   | −0.3504 (2)   | 0.0747 (14)* | 0.4499 (19) |
| H26 | 0.2743 (3)    | 0.1759 (3)   | −0.3725 (2)   | 0.0721 (13)* | 0.4499 (19) |
| H28 | 0.3500 (3)    | 0.0445 (3)   | −0.2436 (3)   | 0.0610 (17)* | 0.4499 (19) |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|-------------|--------------|--------------|--------------|
| Fe1 | 0.03008 (9)  | 0.02696 (8)  | 0.02648 (8) | 0.00061 (6)  | 0.00856 (6)  | −0.00093 (6) |
| S2  | 0.03772 (16) | 0.04049 (17) | 0.0589 (2)  | 0.01068 (13) | 0.02218 (15) | 0.01134 (15) |
| S1  | 0.1172 (4)   | 0.0972 (4)   | 0.0524 (2)  | −0.0628 (3)  | 0.0538 (3)   | −0.0339 (2)  |
| N4  | 0.0269 (4)   | 0.0247 (4)   | 0.0302 (4)  | 0.0028 (3)   | 0.0094 (4)   | −0.0016 (3)  |
| N3  | 0.0290 (4)   | 0.0345 (5)   | 0.0283 (4)  | 0.0037 (4)   | 0.0100 (4)   | −0.0016 (4)  |
| N6  | 0.0316 (5)   | 0.0285 (5)   | 0.0283 (4)  | −0.0000 (4)  | 0.0068 (4)   | 0.0004 (4)   |
| N5  | 0.0320 (5)   | 0.0276 (5)   | 0.0277 (4)  | 0.0003 (4)   | 0.0079 (4)   | −0.0004 (4)  |
| Cl1 | 0.1329 (11)  | 0.0732 (7)   | 0.0824 (7)  | 0.0296 (7)   | 0.0255 (7)   | 0.0116 (5)   |
| N1  | 0.0522 (6)   | 0.0393 (6)   | 0.0367 (5)  | −0.0076 (5)  | 0.0210 (5)   | −0.0037 (4)  |
| N2  | 0.0470 (6)   | 0.0421 (6)   | 0.0447 (6)  | 0.0100 (5)   | 0.0105 (5)   | 0.0086 (5)   |
| C7  | 0.0294 (5)   | 0.0341 (6)   | 0.0255 (5)  | −0.0026 (4)  | 0.0094 (4)   | −0.0024 (4)  |
| C19 | 0.0327 (5)   | 0.0267 (5)   | 0.0282 (5)  | −0.0010 (4)  | 0.0096 (4)   | −0.0029 (4)  |
| C13 | 0.0286 (5)   | 0.0297 (5)   | 0.0316 (5)  | −0.0010 (4)  | 0.0084 (4)   | 0.0006 (4)   |
| C12 | 0.0303 (6)   | 0.0381 (6)   | 0.0405 (6)  | −0.0047 (5)  | 0.0057 (5)   | 0.0031 (5)   |
| C14 | 0.0273 (5)   | 0.0304 (6)   | 0.0421 (6)  | 0.0028 (4)   | 0.0109 (5)   | −0.0030 (5)  |
| C8  | 0.0373 (6)   | 0.0346 (6)   | 0.0316 (5)  | 0.0072 (5)   | 0.0116 (5)   | 0.0053 (5)   |
| C20 | 0.0331 (6)   | 0.0282 (5)   | 0.0408 (6)  | 0.0028 (4)   | 0.0111 (5)   | −0.0077 (5)  |
| C9  | 0.0437 (6)   | 0.0273 (5)   | 0.0305 (5)  | −0.0009 (5)  | 0.0096 (5)   | 0.0016 (4)   |



|      |             |             |            |              |             |              |
|------|-------------|-------------|------------|--------------|-------------|--------------|
| C18  | 0.0410 (7)  | 0.0295 (6)  | 0.0390 (6) | −0.0045 (5)  | 0.0119 (5)  | −0.0065 (5)  |
| C3   | 0.0343 (6)  | 0.0469 (7)  | 0.0413 (6) | 0.0085 (5)   | 0.0145 (5)  | −0.0050 (5)  |
| C1   | 0.0492 (7)  | 0.0410 (7)  | 0.0322 (6) | −0.0160 (5)  | 0.0172 (5)  | −0.0079 (5)  |
| C2   | 0.0315 (5)  | 0.0349 (6)  | 0.0366 (6) | 0.0045 (5)   | 0.0120 (5)  | 0.0050 (5)   |
| C10  | 0.0520 (8)  | 0.0310 (6)  | 0.0381 (6) | −0.0102 (5)  | 0.0099 (6)  | −0.0011 (5)  |
| C15  | 0.0333 (6)  | 0.0385 (6)  | 0.0358 (6) | 0.0019 (5)   | 0.0030 (5)  | 0.0028 (5)   |
| C11  | 0.0407 (7)  | 0.0402 (7)  | 0.0410 (7) | −0.0127 (5)  | 0.0050 (5)  | 0.0003 (5)   |
| C17  | 0.0421 (7)  | 0.0403 (7)  | 0.0447 (7) | −0.0120 (6)  | 0.0100 (6)  | −0.0084 (5)  |
| C6   | 0.0434 (7)  | 0.0527 (8)  | 0.0326 (6) | −0.0042 (6)  | 0.0174 (5)  | 0.0021 (5)   |
| C16  | 0.0348 (6)  | 0.0493 (8)  | 0.0420 (7) | −0.0066 (6)  | 0.0026 (5)  | −0.0020 (6)  |
| C4   | 0.0396 (7)  | 0.0660 (9)  | 0.0507 (8) | 0.0053 (6)   | 0.0240 (6)  | −0.0127 (7)  |
| C5   | 0.0481 (7)  | 0.0726 (10) | 0.0419 (7) | −0.0042 (7)  | 0.0274 (6)  | −0.0064 (7)  |
| C26  | 0.068 (3)   | 0.068 (2)   | 0.065 (3)  | 0.014 (2)    | 0.038 (2)   | −0.001 (2)   |
| C24  | 0.039 (2)   | 0.057 (2)   | 0.102 (4)  | −0.0064 (16) | 0.028 (2)   | −0.030 (2)   |
| C21  | 0.070 (3)   | 0.054 (2)   | 0.062 (2)  | −0.0038 (17) | 0.0385 (19) | −0.0147 (16) |
| C23  | 0.0401 (19) | 0.081 (3)   | 0.085 (3)  | −0.0173 (19) | 0.0321 (18) | −0.046 (3)   |
| C25  | 0.055 (2)   | 0.066 (3)   | 0.069 (2)  | 0.0056 (17)  | 0.0312 (18) | −0.000 (2)   |
| C22  | 0.0584 (19) | 0.075 (2)   | 0.058 (2)  | −0.0245 (18) | 0.0349 (17) | −0.0293 (19) |
| C22a | 0.055 (3)   | 0.047 (2)   | 0.063 (3)  | −0.012 (2)   | 0.035 (2)   | −0.018 (2)   |
| Cl1a | 0.1131 (11) | 0.0586 (7)  | 0.0766 (8) | −0.0172 (7)  | 0.0141 (7)  | 0.0167 (6)   |
| C21a | 0.052 (2)   | 0.0326 (17) | 0.044 (2)  | −0.0133 (16) | 0.0247 (17) | −0.0100 (15) |
| C26a | 0.069 (4)   | 0.044 (2)   | 0.057 (3)  | −0.0065 (19) | 0.045 (3)   | −0.0121 (18) |
| C23a | 0.041 (2)   | 0.051 (3)   | 0.080 (3)  | −0.006 (2)   | 0.021 (2)   | −0.017 (3)   |
| C25a | 0.077 (3)   | 0.057 (3)   | 0.055 (2)  | −0.012 (2)   | 0.035 (2)   | −0.0078 (19) |
| C24a | 0.046 (2)   | 0.052 (2)   | 0.076 (3)  | −0.0061 (17) | 0.004 (2)   | −0.001 (2)   |

*Geometric parameters (Å, °)*

|         |             |         |             |
|---------|-------------|---------|-------------|
| Fe1—N4  | 2.2417 (9)  | C3—C4   | 1.3829 (19) |
| Fe1—N3  | 2.1930 (10) | C3—H1   | 1.1030      |
| Fe1—N6  | 2.2054 (10) | C10—C11 | 1.388 (2)   |
| Fe1—N5  | 2.2047 (10) | C10—H14 | 1.1030      |
| Fe1—N1  | 2.1123 (11) | C15—C16 | 1.3873 (18) |
| Fe1—N2  | 2.0535 (11) | C15—H7  | 1.1030      |
| S2—C2   | 1.6216 (12) | C11—H15 | 1.1030      |
| S1—C1   | 1.6072 (16) | C17—C16 | 1.388 (2)   |
| N4—C14  | 1.4742 (14) | C17—H9  | 1.1030      |
| N4—C8   | 1.4793 (14) | C6—C5   | 1.383 (2)   |
| N4—C20  | 1.4782 (14) | C6—H4   | 1.1030      |
| N3—C7   | 1.3382 (15) | C16—H8  | 1.1030      |
| N3—C3   | 1.3432 (15) | C4—C5   | 1.385 (2)   |
| N6—C19  | 1.3407 (14) | C4—H2   | 1.1030      |
| N6—C15  | 1.3379 (15) | C5—H3   | 1.1030      |
| N5—C13  | 1.3439 (14) | C26—C21 | 1.3900      |
| N5—C9   | 1.3421 (15) | C26—C25 | 1.3900      |
| Cl1—C21 | 1.696 (3)   | C26—H27 | 1.1030      |
| N1—C1   | 1.1596 (17) | C24—C23 | 1.3900      |
| N2—C2   | 1.1659 (16) | C24—C25 | 1.3900      |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| C7—C8      | 1.5082 (16) | C24—H23     | 1.1030      |
| C7—C6      | 1.3916 (16) | C21—C22     | 1.3900      |
| C19—C20    | 1.5043 (16) | C23—C22     | 1.3900      |
| C19—C18    | 1.3914 (16) | C23—H21     | 1.1030      |
| C13—C12    | 1.3906 (16) | C25—H25     | 1.1030      |
| C13—C14    | 1.5055 (16) | C22—H19     | 1.1030      |
| C12—C11    | 1.3883 (18) | C22a—C21a   | 1.3900      |
| C12—H16    | 1.1030      | C22a—C23a   | 1.3900      |
| C14—H18    | 1.1110      | C22a—H20    | 1.1030      |
| C14—H17    | 1.1110      | Cl1a—C21a   | 1.695 (3)   |
| C8—H5      | 1.1110      | C21a—C26a   | 1.3900      |
| C8—H6      | 1.1110      | C26a—C25a   | 1.3900      |
| C20—H11    | 1.1110      | C26a—H28    | 1.1030      |
| C20—H12    | 1.1110      | C23a—C24a   | 1.3900      |
| C9—C10     | 1.3887 (17) | C23a—H22    | 1.1030      |
| C9—H13     | 1.1030      | C25a—C24a   | 1.3900      |
| C18—C17    | 1.3862 (18) | C25a—H26    | 1.1030      |
| C18—H10    | 1.1030      | C24a—H24    | 1.1030      |
| N3—Fe1—N4  | 78.16 (3)   | H10—C18—C17 | 120.66 (8)  |
| N6—Fe1—N4  | 75.56 (3)   | C4—C3—N3    | 122.75 (13) |
| N6—Fe1—N3  | 83.87 (4)   | H1—C3—N3    | 118.62 (7)  |
| N5—Fe1—N4  | 75.83 (3)   | H1—C3—C4    | 118.62 (9)  |
| N5—Fe1—N3  | 89.33 (3)   | N1—C1—S1    | 179.42 (12) |
| N5—Fe1—N6  | 151.37 (4)  | N2—C2—S2    | 179.24 (12) |
| N1—Fe1—N4  | 91.70 (4)   | C11—C10—C9  | 118.77 (12) |
| N1—Fe1—N3  | 169.14 (4)  | H14—C10—C9  | 120.62 (8)  |
| N1—Fe1—N6  | 89.88 (4)   | H14—C10—C11 | 120.62 (7)  |
| N1—Fe1—N5  | 91.96 (4)   | C16—C15—N6  | 122.70 (12) |
| N2—Fe1—N4  | 172.21 (4)  | H7—C15—N6   | 118.65 (7)  |
| N2—Fe1—N3  | 94.69 (4)   | H7—C15—C16  | 118.65 (8)  |
| N2—Fe1—N6  | 107.03 (4)  | C10—C11—C12 | 118.78 (12) |
| N2—Fe1—N5  | 101.22 (4)  | H15—C11—C12 | 120.61 (8)  |
| N2—Fe1—N1  | 95.63 (5)   | H15—C11—C10 | 120.61 (7)  |
| C14—N4—Fe1 | 105.98 (7)  | C16—C17—C18 | 119.19 (12) |
| C8—N4—Fe1  | 111.07 (6)  | H9—C17—C18  | 120.41 (8)  |
| C8—N4—C14  | 111.36 (9)  | H9—C17—C16  | 120.41 (8)  |
| C20—N4—Fe1 | 105.71 (7)  | C5—C6—C7    | 119.07 (13) |
| C20—N4—C14 | 110.40 (9)  | H4—C6—C7    | 120.47 (8)  |
| C20—N4—C8  | 112.00 (9)  | H4—C6—C5    | 120.47 (9)  |
| C7—N3—Fe1  | 116.14 (7)  | C17—C16—C15 | 118.46 (12) |
| C3—N3—Fe1  | 124.89 (9)  | H8—C16—C15  | 120.77 (8)  |
| C3—N3—C7   | 118.78 (11) | H8—C16—C17  | 120.77 (8)  |
| C19—N6—Fe1 | 114.98 (7)  | C5—C4—C3    | 118.38 (13) |
| C15—N6—Fe1 | 126.35 (8)  | H2—C4—C3    | 120.81 (9)  |
| C15—N6—C19 | 118.67 (10) | H2—C4—C5    | 120.81 (8)  |
| C13—N5—Fe1 | 114.72 (7)  | C4—C5—C6    | 119.23 (13) |
| C9—N5—Fe1  | 126.75 (8)  | H3—C5—C6    | 120.38 (9)  |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C9—N5—C13      | 118.52 (10)  | H3—C5—C4        | 120.38 (8)   |
| C1—N1—Fe1      | 169.59 (11)  | C25—C26—C21     | 120.0        |
| C2—N2—Fe1      | 176.28 (11)  | H27—C26—C21     | 120.00 (12)  |
| C8—C7—N3       | 118.51 (10)  | H27—C26—C25     | 120.00 (12)  |
| C6—C7—N3       | 121.78 (11)  | C25—C24—C23     | 120.0        |
| C6—C7—C8       | 119.66 (11)  | H23—C24—C23     | 120.00 (12)  |
| C20—C19—N6     | 115.95 (10)  | H23—C24—C25     | 120.00 (12)  |
| C18—C19—N6     | 122.24 (10)  | C26—C21—Cl1     | 122.0 (2)    |
| C18—C19—C20    | 121.74 (10)  | C22—C21—Cl1     | 117.9 (2)    |
| C12—C13—N5     | 122.16 (11)  | C22—C21—C26     | 120.0        |
| C14—C13—N5     | 116.16 (10)  | C22—C23—C24     | 120.0        |
| C14—C13—C12    | 121.62 (11)  | H21—C23—C24     | 120.00 (12)  |
| C11—C12—C13    | 119.11 (12)  | H21—C23—C22     | 120.00 (12)  |
| H16—C12—C13    | 120.45 (7)   | C24—C25—C26     | 120.0        |
| H16—C12—C11    | 120.45 (8)   | H25—C25—C26     | 120.00 (12)  |
| C13—C14—N4     | 111.05 (9)   | H25—C25—C24     | 120.00 (12)  |
| H18—C14—N4     | 109.43 (6)   | C23—C22—C21     | 120.0        |
| H18—C14—C13    | 109.43 (6)   | H19—C22—C21     | 120.0        |
| H17—C14—N4     | 109.43 (6)   | H19—C22—C23     | 120.0        |
| H17—C14—C13    | 109.43 (6)   | C23a—C22a—C21a  | 120.0        |
| H17—C14—H18    | 108.0        | H20—C22a—C21a   | 120.0        |
| C7—C8—N4       | 115.49 (9)   | H20—C22a—C23a   | 120.0        |
| H5—C8—N4       | 108.41 (6)   | Cl1a—C21a—C22a  | 118.7 (3)    |
| H5—C8—C7       | 108.41 (6)   | C26a—C21a—C22a  | 120.0        |
| H6—C8—N4       | 108.41 (6)   | C26a—C21a—Cl1a  | 121.3 (3)    |
| H6—C8—C7       | 108.41 (6)   | C25a—C26a—C21a  | 120.0        |
| H6—C8—H5       | 107.5        | H28—C26a—C21a   | 120.00 (14)  |
| C19—C20—N4     | 111.26 (9)   | H28—C26a—C25a   | 120.00 (14)  |
| H11—C20—N4     | 109.38 (6)   | C24a—C23a—C22a  | 120.0        |
| H11—C20—C19    | 109.38 (6)   | H22—C23a—C22a   | 120.00 (14)  |
| H12—C20—N4     | 109.38 (6)   | H22—C23a—C24a   | 120.00 (14)  |
| H12—C20—C19    | 109.38 (6)   | C24a—C25a—C26a  | 120.0        |
| H12—C20—H11    | 108.0        | H26—C25a—C26a   | 120.00 (14)  |
| C10—C9—N5      | 122.65 (12)  | H26—C25a—C24a   | 120.00 (14)  |
| H13—C9—N5      | 118.67 (7)   | C25a—C24a—C23a  | 120.0        |
| H13—C9—C10     | 118.67 (8)   | H24—C24a—C23a   | 120.00 (14)  |
| C17—C18—C19    | 118.68 (12)  | H24—C24a—C25a   | 120.00 (14)  |
| H10—C18—C19    | 120.66 (7)   |                 |              |
| Fe1—N4—C14—C13 | −43.37 (7)   | C19—C20—N4—C8   | −77.54 (11)  |
| Fe1—N4—C8—C7   | −6.97 (7)    | C19—C18—C17—C16 | 2.10 (14)    |
| Fe1—N4—C20—C19 | 43.56 (7)    | C13—N5—C9—C10   | −0.37 (12)   |
| Fe1—N3—C7—C8   | −6.93 (9)    | C13—C12—C11—C10 | −0.31 (14)   |
| Fe1—N3—C7—C6   | 175.45 (8)   | C13—C14—N4—C8   | 77.53 (11)   |
| Fe1—N3—C3—C4   | −175.04 (11) | C13—C14—N4—C20  | −157.39 (10) |
| Fe1—N6—C19—C20 | 1.42 (8)     | C12—C13—N5—C9   | −0.03 (14)   |
| Fe1—N6—C19—C18 | 178.37 (8)   | C12—C11—C10—C9  | −0.05 (15)   |
| Fe1—N6—C15—C16 | −177.34 (10) | C14—C13—N5—C9   | 177.21 (10)  |

|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| Fe1—N5—C13—C12  | 179.17 (8)   | C14—C13—C12—C11     | −176.73 (12) |
| Fe1—N5—C13—C14  | −3.59 (8)    | C8—C7—N3—C3         | 177.85 (11)  |
| Fe1—N5—C9—C10   | −179.46 (10) | C8—C7—C6—C5         | −177.70 (12) |
| N4—C14—C13—N5   | 32.94 (11)   | C20—C19—N6—C15      | −178.21 (10) |
| N4—C14—C13—C12  | −149.81 (10) | C20—C19—C18—C17     | 175.88 (12)  |
| N4—C8—C7—N3     | 9.50 (12)    | C18—C19—N6—C15      | −1.25 (14)   |
| N4—C8—C7—C6     | −172.83 (10) | C18—C17—C16—C15     | −1.19 (16)   |
| N4—C20—C19—N6   | −31.61 (11)  | C3—N3—C7—C6         | 0.23 (13)    |
| N4—C20—C19—C18  | 151.42 (10)  | C3—C4—C5—C6         | −0.06 (17)   |
| N3—C7—C6—C5     | −0.11 (14)   | C26—C21—C22—C23     | 0.0 (3)      |
| N3—C3—C4—C5     | 0.19 (16)    | C26—C25—C24—C23     | −0.0 (2)     |
| N6—C19—C18—C17  | −0.90 (13)   | C24—C23—C22—C21     | −0.0 (3)     |
| N6—C15—C16—C17  | −1.02 (15)   | C24—C25—C26—C21     | −0.0 (2)     |
| N5—C13—C12—C11  | 0.36 (14)    | C25—C26—C21—C22     | 0.0 (3)      |
| N5—C9—C10—C11   | 0.41 (14)    | C25—C24—C23—C22     | 0.0 (3)      |
| C11—C21—C26—C25 | −176.10 (10) | C22a—C21a—C26a—C25a | 0.0 (3)      |
| C11—C21—C22—C23 | 176.26 (10)  | C22a—C23a—C24a—C25a | −0.0 (4)     |
| C7—N3—C3—C4     | −0.28 (13)   | C11a—C21a—C22a—C23a | −179.15 (10) |
| C7—C8—N4—C14    | −124.84 (10) | C11a—C21a—C26a—C25a | 179.13 (10)  |
| C7—C8—N4—C20    | 110.98 (10)  | C21a—C22a—C23a—C24a | 0.0 (3)      |
| C7—C6—C5—C4     | 0.02 (15)    | C21a—C26a—C25a—C24a | 0.0 (4)      |
| C19—N6—C15—C16  | 2.24 (13)    | C26a—C21a—C22a—C23a | −0.0 (3)     |
| C19—C20—N4—C14  | 157.75 (10)  | C26a—C25a—C24a—C23a | −0.0 (3)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_210K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.2919$  (2) Å

$b = 12.0800$  (1) Å

$c = 15.4930$  (2) Å

$\beta = 110.248$  (2)°

$V = 2685.10$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.422$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 65480 reflections

$\theta = 2.1$ – $32.0^\circ$

$\mu = 0.84$  mm<sup>−1</sup>

$T = 210$  K

Block, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.535$ ,  $T_{\max} = 1.000$

78410 measured reflections

6660 independent reflections

6186 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.026$  $wR(F^2) = 0.066$  $S = 1.05$ 

6660 reflections

375 parameters

2 restraints

56 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.7849P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.78 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$ *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  | 0.348634 (11) | 0.321786 (13) | 0.198671 (10) | 0.03036 (5)                      |           |
| S2   | 0.53252 (2)   | 0.00118 (3)   | 0.33855 (3)   | 0.04835 (9)                      |           |
| S1   | 0.24231 (9)   | 0.32693 (10)  | 0.45983 (7)   | 0.0890 (2)                       |           |
| N4   | 0.26173 (6)   | 0.46312 (7)   | 0.12020 (6)   | 0.02990 (18)                     |           |
| N3   | 0.39394 (6)   | 0.33375 (8)   | 0.07901 (6)   | 0.03348 (19)                     |           |
| N5   | 0.43769 (6)   | 0.46947 (8)   | 0.24647 (6)   | 0.03314 (19)                     |           |
| N6   | 0.21800 (6)   | 0.24284 (8)   | 0.11035 (6)   | 0.03237 (19)                     |           |
| N1   | 0.29683 (8)   | 0.34171 (9)   | 0.30778 (8)   | 0.0447 (2)                       |           |
| Cl1a | 0.34214 (11)  | 0.03685 (10)  | −0.06085 (9)  | 0.0928 (6)                       | 0.491 (2) |
| N2   | 0.42929 (9)   | 0.18584 (10)  | 0.25354 (8)   | 0.0499 (3)                       |           |
| C13  | 0.14640 (8)   | 0.31294 (9)   | 0.07411 (8)   | 0.0333 (2)                       |           |
| C7   | 0.35583 (7)   | 0.41530 (9)   | 0.01966 (7)   | 0.0324 (2)                       |           |
| C19  | 0.39230 (8)   | 0.56640 (9)   | 0.22907 (7)   | 0.0322 (2)                       |           |
| C9   | 0.20471 (9)   | 0.13492 (10)  | 0.08926 (8)   | 0.0375 (2)                       |           |
| C20  | 0.28838 (8)   | 0.55972 (9)   | 0.18226 (8)   | 0.0375 (2)                       |           |
| C12  | 0.05960 (8)   | 0.27728 (11)  | 0.01588 (9)   | 0.0417 (3)                       |           |
| C18  | 0.43765 (9)   | 0.66710 (10)  | 0.25656 (9)   | 0.0407 (3)                       |           |
| C8   | 0.27823 (8)   | 0.48245 (10)  | 0.03290 (8)   | 0.0379 (2)                       |           |
| C15  | 0.53001 (9)   | 0.47012 (11)  | 0.29037 (8)   | 0.0421 (3)                       |           |
| C14  | 0.16410 (7)   | 0.43211 (9)   | 0.10358 (9)   | 0.0366 (2)                       |           |
| C2   | 0.47235 (8)   | 0.10822 (10)  | 0.28855 (8)   | 0.0370 (2)                       |           |
| C11  | 0.04610 (10)  | 0.16536 (11)  | −0.00515 (9)  | 0.0469 (3)                       |           |
| C1   | 0.27416 (10)  | 0.33592 (10)  | 0.37161 (9)   | 0.0428 (3)                       |           |
| C10  | 0.11996 (10)  | 0.09281 (11)  | 0.03223 (9)   | 0.0453 (3)                       |           |
| C17  | 0.53280 (10)  | 0.66668 (11)  | 0.30455 (10)  | 0.0481 (3)                       |           |
| C3   | 0.46092 (9)   | 0.27147 (12)  | 0.06577 (9)   | 0.0445 (3)                       |           |
| C6   | 0.38353 (9)   | 0.43686 (12)  | −0.05539 (9)  | 0.0460 (3)                       |           |
| C16  | 0.58025 (9)   | 0.56655 (12)  | 0.32125 (9)   | 0.0494 (3)                       |           |
| C4   | 0.49198 (10)  | 0.28778 (14)  | −0.00712 (11) | 0.0553 (4)                       |           |
| C26a | 0.3068 (3)    | 0.1122 (3)    | −0.2330 (3)   | 0.0551 (13)                      | 0.491 (2) |
| C5   | 0.45238 (10)  | 0.37207 (15)  | −0.06863 (10) | 0.0566 (4)                       |           |
| C23a | 0.1968 (2)    | 0.2848 (2)    | −0.2097 (3)   | 0.0618 (13)                      | 0.491 (2) |
| C21a | 0.2934 (2)    | 0.1257 (2)    | −0.1494 (2)   | 0.0453 (10)                      | 0.491 (2) |
| C22a | 0.2383 (3)    | 0.2120 (3)    | −0.1377 (2)   | 0.0571 (11)                      | 0.491 (2) |
| C25a | 0.2653 (3)    | 0.1851 (3)    | −0.30496 (19) | 0.0640 (10)                      | 0.491 (2) |
| C24a | 0.2103 (2)    | 0.2714 (2)    | −0.2933 (2)   | 0.0659 (11)                      | 0.491 (2) |

|     |               |              |               |              |           |
|-----|---------------|--------------|---------------|--------------|-----------|
| H1  | 0.49219 (9)   | 0.20510 (12) | 0.11467 (9)   | 0.0534 (3)*  |           |
| H2  | 0.54652 (10)  | 0.23531 (14) | −0.01593 (11) | 0.0664 (4)*  |           |
| H3  | 0.47524 (10)  | 0.38731 (15) | −0.12706 (10) | 0.0679 (4)*  |           |
| H4  | 0.35134 (9)   | 0.50399 (12) | −0.10311 (9)  | 0.0553 (3)*  |           |
| H7  | 0.56738 (9)   | 0.39128 (11) | 0.30239 (8)   | 0.0505 (3)*  |           |
| H8  | 0.65548 (9)   | 0.56380 (12) | 0.35788 (9)   | 0.0592 (4)*  |           |
| H9  | 0.57000 (10)  | 0.74421 (11) | 0.32894 (10)  | 0.0578 (4)*  |           |
| H10 | 0.39898 (9)   | 0.74498 (10) | 0.24061 (9)   | 0.0488 (3)*  |           |
| H13 | 0.26302 (9)   | 0.07785 (10) | 0.11819 (8)   | 0.0450 (3)*  |           |
| H14 | 0.11155 (10)  | 0.00428 (11) | 0.01705 (9)   | 0.0543 (3)*  |           |
| H15 | −0.02156 (10) | 0.13495 (11) | −0.05038 (9)  | 0.0563 (4)*  |           |
| H16 | 0.00283 (8)   | 0.33640 (11) | −0.01291 (9)  | 0.0500 (3)*  |           |
| H11 | 0.26322 (8)   | 0.63593 (9)  | 0.14242 (8)   | 0.0451 (3)*  |           |
| H12 | 0.25492 (8)   | 0.55349 (9)  | 0.23460 (8)   | 0.0451 (3)*  |           |
| H5  | 0.21331 (8)   | 0.46438 (10) | −0.02446 (8)  | 0.0454 (3)*  |           |
| H6  | 0.29397 (8)   | 0.57082 (10) | 0.02883 (8)   | 0.0454 (3)*  |           |
| H18 | 0.14816 (7)   | 0.44488 (9)  | 0.16693 (9)   | 0.0440 (3)*  |           |
| H17 | 0.11761 (7)   | 0.48581 (9)  | 0.04970 (9)   | 0.0440 (3)*  |           |
| C22 | 0.2711 (2)    | 0.1551 (4)   | −0.1042 (2)   | 0.0692 (11)  | 0.509 (2) |
| Cl1 | 0.40092 (13)  | 0.00834 (11) | −0.09938 (10) | 0.1124 (6)   | 0.509 (2) |
| C21 | 0.3193 (3)    | 0.1036 (3)   | −0.1544 (3)   | 0.0649 (13)  | 0.509 (2) |
| C26 | 0.3038 (3)    | 0.1355 (3)   | −0.2447 (3)   | 0.0702 (16)  | 0.509 (2) |
| C25 | 0.2401 (2)    | 0.2190 (3)   | −0.2848 (2)   | 0.0685 (11)  | 0.509 (2) |
| C23 | 0.2074 (2)    | 0.2386 (4)   | −0.1443 (3)   | 0.0773 (18)  | 0.509 (2) |
| C24 | 0.1919 (2)    | 0.2705 (3)   | −0.2345 (3)   | 0.0767 (18)  | 0.509 (2) |
| H20 | 0.2277 (3)    | 0.2225 (3)   | −0.0720 (2)   | 0.0685 (13)* | 0.491 (2) |
| H22 | 0.1535 (2)    | 0.3527 (2)   | −0.2006 (3)   | 0.0742 (15)* | 0.491 (2) |
| H24 | 0.1776 (2)    | 0.3287 (2)   | −0.3500 (2)   | 0.0791 (13)* | 0.491 (2) |
| H26 | 0.2759 (3)    | 0.1746 (3)   | −0.37071 (19) | 0.0768 (12)* | 0.491 (2) |
| H28 | 0.3501 (3)    | 0.0444 (3)   | −0.2421 (3)   | 0.0662 (16)* | 0.491 (2) |
| H27 | 0.3417 (3)    | 0.0950 (3)   | −0.2842 (3)   | 0.084 (2)*   | 0.509 (2) |
| H25 | 0.2279 (2)    | 0.2441 (3)   | −0.3557 (2)   | 0.0822 (13)* | 0.509 (2) |
| H23 | 0.1418 (2)    | 0.3361 (3)   | −0.2661 (3)   | 0.092 (2)*   | 0.509 (2) |
| H21 | 0.1695 (2)    | 0.2791 (4)   | −0.1048 (3)   | 0.093 (2)*   | 0.509 (2) |
| H19 | 0.2833 (2)    | 0.1300 (4)   | −0.0332 (2)   | 0.0830 (14)* | 0.509 (2) |

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

|      | $U^{11}$     | $U^{22}$     | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|------|--------------|--------------|-------------|--------------|--------------|--------------|
| Fe1  | 0.03269 (9)  | 0.02877 (8)  | 0.02828 (8) | 0.00137 (6)  | 0.00884 (6)  | −0.00003 (5) |
| S2   | 0.04136 (16) | 0.04305 (17) | 0.0650 (2)  | 0.01262 (13) | 0.02396 (15) | 0.01390 (15) |
| S1   | 0.1303 (5)   | 0.1017 (4)   | 0.0565 (2)  | −0.0663 (3)  | 0.0596 (3)   | −0.0355 (2)  |
| N4   | 0.0294 (4)   | 0.0269 (4)   | 0.0327 (4)  | 0.0033 (3)   | 0.0099 (3)   | −0.0019 (3)  |
| N3   | 0.0317 (4)   | 0.0380 (5)   | 0.0312 (4)  | 0.0044 (4)   | 0.0115 (4)   | −0.0017 (4)  |
| N5   | 0.0340 (5)   | 0.0310 (4)   | 0.0310 (4)  | −0.0002 (4)  | 0.0069 (4)   | 0.0007 (4)   |
| N6   | 0.0346 (5)   | 0.0302 (4)   | 0.0304 (4)  | 0.0002 (4)   | 0.0088 (4)   | −0.0006 (3)  |
| N1   | 0.0575 (6)   | 0.0437 (6)   | 0.0378 (5)  | −0.0083 (5)  | 0.0227 (5)   | −0.0039 (4)  |
| Cl1a | 0.1233 (11)  | 0.0630 (6)   | 0.0800 (7)  | −0.0165 (6)  | 0.0198 (7)   | 0.0151 (5)   |

|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| N2   | 0.0518 (6)  | 0.0440 (6)  | 0.0489 (6)  | 0.0125 (5)   | 0.0110 (5)  | 0.0105 (5)   |
| C13  | 0.0310 (5)  | 0.0324 (5)  | 0.0347 (5)  | −0.0014 (4)  | 0.0092 (4)  | 0.0001 (4)   |
| C7   | 0.0318 (5)  | 0.0368 (5)  | 0.0282 (5)  | −0.0029 (4)  | 0.0101 (4)  | −0.0021 (4)  |
| C19  | 0.0357 (5)  | 0.0289 (5)  | 0.0310 (5)  | −0.0012 (4)  | 0.0103 (4)  | −0.0029 (4)  |
| C9   | 0.0473 (6)  | 0.0297 (5)  | 0.0331 (5)  | −0.0015 (5)  | 0.0109 (5)  | 0.0011 (4)   |
| C20  | 0.0363 (5)  | 0.0300 (5)  | 0.0447 (6)  | 0.0035 (4)   | 0.0119 (5)  | −0.0080 (5)  |
| C12  | 0.0326 (6)  | 0.0419 (6)  | 0.0445 (6)  | −0.0050 (5)  | 0.0057 (5)  | 0.0027 (5)   |
| C18  | 0.0451 (7)  | 0.0323 (6)  | 0.0428 (6)  | −0.0052 (5)  | 0.0130 (5)  | −0.0068 (5)  |
| C8   | 0.0406 (6)  | 0.0379 (6)  | 0.0343 (5)  | 0.0080 (5)   | 0.0120 (5)  | 0.0061 (4)   |
| C15  | 0.0365 (6)  | 0.0425 (6)  | 0.0391 (6)  | 0.0017 (5)   | 0.0026 (5)  | 0.0028 (5)   |
| C14  | 0.0298 (5)  | 0.0327 (5)  | 0.0462 (6)  | 0.0034 (4)   | 0.0115 (5)  | −0.0035 (5)  |
| C2   | 0.0345 (5)  | 0.0367 (6)  | 0.0401 (6)  | 0.0055 (5)   | 0.0134 (5)  | 0.0063 (5)   |
| C11  | 0.0441 (7)  | 0.0445 (7)  | 0.0446 (7)  | −0.0137 (5)  | 0.0059 (5)  | −0.0001 (5)  |
| C1   | 0.0538 (7)  | 0.0429 (6)  | 0.0345 (6)  | −0.0165 (5)  | 0.0188 (5)  | −0.0086 (5)  |
| C10  | 0.0555 (8)  | 0.0338 (6)  | 0.0416 (6)  | −0.0109 (5)  | 0.0107 (6)  | −0.0016 (5)  |
| C17  | 0.0464 (7)  | 0.0442 (7)  | 0.0493 (7)  | −0.0134 (6)  | 0.0108 (6)  | −0.0090 (6)  |
| C3   | 0.0373 (6)  | 0.0519 (7)  | 0.0456 (7)  | 0.0097 (5)   | 0.0161 (5)  | −0.0053 (6)  |
| C6   | 0.0481 (7)  | 0.0580 (8)  | 0.0358 (6)  | −0.0049 (6)  | 0.0193 (5)  | 0.0026 (5)   |
| C16  | 0.0379 (6)  | 0.0544 (8)  | 0.0460 (7)  | −0.0083 (6)  | 0.0022 (5)  | −0.0024 (6)  |
| C4   | 0.0440 (7)  | 0.0736 (10) | 0.0561 (8)  | 0.0055 (7)   | 0.0270 (6)  | −0.0141 (7)  |
| C26a | 0.076 (3)   | 0.0484 (19) | 0.060 (3)   | −0.0061 (17) | 0.048 (2)   | −0.0134 (17) |
| C5   | 0.0537 (8)  | 0.0808 (11) | 0.0461 (7)  | −0.0043 (7)  | 0.0309 (6)  | −0.0061 (7)  |
| C23a | 0.047 (2)   | 0.055 (2)   | 0.084 (3)   | −0.0032 (19) | 0.024 (2)   | −0.015 (2)   |
| C21a | 0.057 (2)   | 0.0378 (16) | 0.0483 (19) | −0.0136 (15) | 0.0278 (16) | −0.0102 (14) |
| C22a | 0.061 (3)   | 0.050 (2)   | 0.073 (3)   | −0.013 (2)   | 0.040 (2)   | −0.0202 (19) |
| C25a | 0.082 (3)   | 0.059 (2)   | 0.062 (2)   | −0.011 (2)   | 0.038 (2)   | −0.0070 (18) |
| C24a | 0.046 (2)   | 0.057 (2)   | 0.083 (3)   | −0.0057 (16) | 0.0081 (19) | −0.003 (2)   |
| C22  | 0.068 (2)   | 0.088 (3)   | 0.066 (2)   | −0.031 (2)   | 0.042 (2)   | −0.037 (2)   |
| C11  | 0.1497 (14) | 0.0784 (8)  | 0.0956 (9)  | 0.0285 (8)   | 0.0254 (9)  | 0.0127 (6)   |
| C21  | 0.077 (3)   | 0.060 (2)   | 0.071 (3)   | −0.0074 (19) | 0.044 (2)   | −0.0180 (19) |
| C26  | 0.074 (3)   | 0.076 (3)   | 0.075 (4)   | 0.014 (2)    | 0.043 (3)   | 0.001 (3)    |
| C25  | 0.061 (2)   | 0.074 (3)   | 0.078 (3)   | 0.009 (2)    | 0.034 (2)   | 0.000 (2)    |
| C23  | 0.046 (2)   | 0.096 (4)   | 0.100 (4)   | −0.022 (2)   | 0.038 (2)   | −0.057 (3)   |
| C24  | 0.042 (2)   | 0.068 (3)   | 0.124 (5)   | −0.0101 (19) | 0.034 (3)   | −0.040 (3)   |

*Geometric parameters (Å, °)*

|        |             |         |             |
|--------|-------------|---------|-------------|
| Fe1—N4 | 2.2451 (9)  | C14—H18 | 1.1010      |
| Fe1—N3 | 2.1971 (10) | C14—H17 | 1.1010      |
| Fe1—N5 | 2.2108 (9)  | C11—C10 | 1.388 (2)   |
| Fe1—N6 | 2.2091 (9)  | C11—H15 | 1.0930      |
| Fe1—N1 | 2.1143 (11) | C10—H14 | 1.0930      |
| Fe1—N2 | 2.0530 (11) | C17—C16 | 1.388 (2)   |
| S2—C2  | 1.6203 (12) | C17—H9  | 1.0930      |
| S1—C1  | 1.6056 (15) | C3—C4   | 1.3825 (19) |
| N4—C20 | 1.4769 (13) | C3—H1   | 1.0930      |
| N4—C8  | 1.4777 (14) | C6—C5   | 1.383 (2)   |
| N4—C14 | 1.4729 (14) | C6—H4   | 1.0930      |



|            |             |             |             |
|------------|-------------|-------------|-------------|
| N3—C7      | 1.3360 (14) | C16—H8      | 1.0930      |
| N3—C3      | 1.3425 (15) | C4—C5       | 1.383 (2)   |
| N5—C19     | 1.3402 (14) | C4—H2       | 1.0930      |
| N5—C15     | 1.3384 (15) | C26a—C21a   | 1.3900      |
| N6—C13     | 1.3438 (14) | C26a—C25a   | 1.3900      |
| N6—C9      | 1.3423 (15) | C26a—H28    | 1.0930      |
| N1—C1      | 1.1577 (16) | C5—H3       | 1.0930      |
| Cl1a—C21a  | 1.697 (3)   | C23a—C22a   | 1.3900      |
| N2—C2      | 1.1661 (16) | C23a—C24a   | 1.3900      |
| C13—C12    | 1.3900 (16) | C23a—H22    | 1.0930      |
| C13—C14    | 1.5062 (15) | C21a—C22a   | 1.3900      |
| C7—C8      | 1.5089 (15) | C22a—H20    | 1.0930      |
| C7—C6      | 1.3930 (16) | C25a—C24a   | 1.3900      |
| C19—C20    | 1.5033 (16) | C25a—H26    | 1.0930      |
| C19—C18    | 1.3919 (15) | C24a—H24    | 1.0930      |
| C9—C10     | 1.3880 (17) | C22—C21     | 1.3900      |
| C9—H13     | 1.0930      | C22—C23     | 1.3900      |
| C20—H11    | 1.1010      | C22—H19     | 1.0930      |
| C20—H12    | 1.1010      | Cl1—C21     | 1.695 (3)   |
| C12—C11    | 1.3892 (18) | C21—C26     | 1.3900      |
| C12—H16    | 1.0930      | C26—C25     | 1.3900      |
| C18—C17    | 1.3862 (19) | C26—H27     | 1.0930      |
| C18—H10    | 1.0930      | C25—C24     | 1.3900      |
| C8—H5      | 1.1010      | C25—H25     | 1.0930      |
| C8—H6      | 1.1010      | C23—C24     | 1.3900      |
| C15—C16    | 1.3862 (18) | C23—H21     | 1.0930      |
| C15—H7     | 1.0930      | C24—H23     | 1.0930      |
|            |             |             |             |
| N3—Fe1—N4  | 78.08 (3)   | H18—C14—C13 | 109.40 (6)  |
| N5—Fe1—N4  | 75.42 (3)   | H17—C14—N4  | 109.40 (6)  |
| N5—Fe1—N3  | 83.80 (3)   | H17—C14—C13 | 109.40 (6)  |
| N6—Fe1—N4  | 75.76 (3)   | H17—C14—H18 | 108.0       |
| N6—Fe1—N3  | 89.28 (3)   | N2—C2—S2    | 179.21 (12) |
| N6—Fe1—N5  | 151.16 (3)  | C10—C11—C12 | 118.90 (12) |
| N1—Fe1—N4  | 91.78 (4)   | H15—C11—C12 | 120.55 (8)  |
| N1—Fe1—N3  | 169.12 (4)  | H15—C11—C10 | 120.55 (7)  |
| N1—Fe1—N5  | 89.89 (4)   | N1—C1—S1    | 179.53 (12) |
| N1—Fe1—N6  | 92.04 (4)   | C11—C10—C9  | 118.68 (12) |
| N2—Fe1—N4  | 172.17 (4)  | H14—C10—C9  | 120.66 (8)  |
| N2—Fe1—N3  | 94.72 (4)   | H14—C10—C11 | 120.66 (7)  |
| N2—Fe1—N5  | 107.14 (4)  | C16—C17—C18 | 119.16 (12) |
| N2—Fe1—N6  | 101.31 (4)  | H9—C17—C18  | 120.42 (8)  |
| N2—Fe1—N1  | 95.60 (5)   | H9—C17—C16  | 120.42 (8)  |
| C20—N4—Fe1 | 105.67 (7)  | C4—C3—N3    | 122.74 (13) |
| C8—N4—Fe1  | 111.05 (6)  | H1—C3—N3    | 118.63 (7)  |
| C8—N4—C20  | 112.02 (9)  | H1—C3—C4    | 118.63 (9)  |
| C14—N4—Fe1 | 105.99 (7)  | C5—C6—C7    | 119.01 (13) |
| C14—N4—C20 | 110.45 (8)  | H4—C6—C7    | 120.49 (8)  |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| C14—N4—C8   | 111.34 (9)  | H4—C6—C5       | 120.49 (9)  |
| C7—N3—Fe1   | 116.07 (7)  | C17—C16—C15    | 118.45 (12) |
| C3—N3—Fe1   | 124.89 (9)  | H8—C16—C15     | 120.77 (8)  |
| C3—N3—C7    | 118.84 (10) | H8—C16—C17     | 120.77 (8)  |
| C19—N5—Fe1  | 114.94 (7)  | C5—C4—C3       | 118.39 (13) |
| C15—N5—Fe1  | 126.45 (8)  | H2—C4—C3       | 120.80 (9)  |
| C15—N5—C19  | 118.61 (10) | H2—C4—C5       | 120.80 (8)  |
| C13—N6—Fe1  | 114.72 (7)  | C25a—C26a—C21a | 120.0       |
| C9—N6—Fe1   | 126.75 (8)  | H28—C26a—C21a  | 120.00 (14) |
| C9—N6—C13   | 118.52 (10) | H28—C26a—C25a  | 120.00 (14) |
| C1—N1—Fe1   | 169.09 (11) | C4—C5—C6       | 119.27 (13) |
| C2—N2—Fe1   | 175.87 (11) | H3—C5—C6       | 120.37 (9)  |
| C12—C13—N6  | 122.20 (11) | H3—C5—C4       | 120.37 (8)  |
| C14—C13—N6  | 116.14 (10) | C24a—C23a—C22a | 120.0       |
| C14—C13—C12 | 121.60 (10) | H22—C23a—C22a  | 120.00 (14) |
| C8—C7—N3    | 118.63 (9)  | H22—C23a—C24a  | 120.00 (14) |
| C6—C7—N3    | 121.74 (11) | C26a—C21a—C11a | 120.9       |
| C6—C7—C8    | 119.58 (11) | C22a—C21a—C11a | 119.1       |
| C20—C19—N5  | 115.94 (9)  | C22a—C21a—C26a | 120.0       |
| C18—C19—N5  | 122.25 (10) | C21a—C22a—C23a | 120.0       |
| C18—C19—C20 | 121.73 (10) | H20—C22a—C23a  | 120.00 (14) |
| C10—C9—N6   | 122.70 (12) | H20—C22a—C21a  | 120.00 (14) |
| H13—C9—N6   | 118.65 (7)  | C24a—C25a—C26a | 120.0       |
| H13—C9—C10  | 118.65 (8)  | H26—C25a—C26a  | 120.00 (14) |
| C19—C20—N4  | 111.34 (9)  | H26—C25a—C24a  | 120.00 (14) |
| H11—C20—N4  | 109.36 (6)  | C25a—C24a—C23a | 120.0       |
| H11—C20—C19 | 109.36 (6)  | H24—C24a—C23a  | 120.00 (14) |
| H12—C20—N4  | 109.36 (6)  | H24—C24a—C25a  | 120.00 (14) |
| H12—C20—C19 | 109.36 (6)  | C23—C22—C21    | 120.0       |
| H12—C20—H11 | 108.0       | H19—C22—C21    | 120.00 (15) |
| C11—C12—C13 | 119.00 (12) | H19—C22—C23    | 120.00 (15) |
| H16—C12—C13 | 120.50 (7)  | C11—C21—C22    | 117.6       |
| H16—C12—C11 | 120.50 (8)  | C26—C21—C22    | 120.0       |
| C17—C18—C19 | 118.70 (12) | C26—C21—C11    | 122.3       |
| H10—C18—C19 | 120.65 (7)  | C25—C26—C21    | 120.0       |
| H10—C18—C17 | 120.65 (8)  | H27—C26—C21    | 120.00 (15) |
| C7—C8—N4    | 115.53 (9)  | H27—C26—C25    | 120.00 (15) |
| H5—C8—N4    | 108.40 (6)  | C24—C25—C26    | 120.0       |
| H5—C8—C7    | 108.40 (6)  | H25—C25—C26    | 120.00 (15) |
| H6—C8—N4    | 108.40 (6)  | H25—C25—C24    | 120.00 (15) |
| H6—C8—C7    | 108.40 (6)  | C24—C23—C22    | 120.0       |
| H6—C8—H5    | 107.5       | H21—C23—C22    | 120.00 (15) |
| C16—C15—N5  | 122.79 (12) | H21—C23—C24    | 120.00 (15) |
| H7—C15—N5   | 118.61 (7)  | C23—C24—C25    | 120.0       |
| H7—C15—C16  | 118.61 (8)  | H23—C24—C25    | 120.00 (15) |
| C13—C14—N4  | 111.17 (9)  | H23—C24—C23    | 120.00 (15) |
| H18—C14—N4  | 109.40 (6)  |                |             |

|                     |              |                     |              |
|---------------------|--------------|---------------------|--------------|
| Fe1—N4—C20—C19      | 43.78 (7)    | C7—C8—N4—C14        | −124.82 (10) |
| Fe1—N4—C8—C7        | −6.96 (7)    | C7—C6—C5—C4         | −0.07 (15)   |
| Fe1—N4—C14—C13      | −43.38 (7)   | C19—N5—C15—C16      | 2.15 (13)    |
| Fe1—N3—C7—C8        | −7.09 (9)    | C19—C20—N4—C8       | −77.27 (11)  |
| Fe1—N3—C7—C6        | 175.29 (8)   | C19—C20—N4—C14      | 157.99 (10)  |
| Fe1—N3—C3—C4        | −174.89 (11) | C19—C18—C17—C16     | 1.92 (14)    |
| Fe1—N5—C19—C20      | 1.48 (8)     | C9—N6—C13—C12       | 0.03 (12)    |
| Fe1—N5—C19—C18      | 178.42 (8)   | C9—N6—C13—C14       | 177.33 (10)  |
| Fe1—N5—C15—C16      | −177.42 (10) | C9—C10—C11—C12      | −0.06 (14)   |
| Fe1—N6—C13—C12      | 179.03 (8)   | C20—C19—N5—C15      | −178.14 (10) |
| Fe1—N6—C13—C14      | −3.66 (8)    | C20—C19—C18—C17     | 175.95 (12)  |
| Fe1—N6—C9—C10       | −179.39 (10) | C18—C19—N5—C15      | −1.20 (14)   |
| N4—C20—C19—N5       | −31.82 (11)  | C18—C17—C16—C15     | −1.05 (16)   |
| N4—C20—C19—C18      | 151.22 (10)  | C8—C7—N3—C3         | 177.86 (11)  |
| N4—C8—C7—N3         | 9.61 (12)    | C8—C7—C6—C5         | −177.65 (12) |
| N4—C8—C7—C6         | −172.72 (10) | C14—C13—C12—C11     | −176.72 (12) |
| N4—C14—C13—N6       | 33.02 (11)   | C3—N3—C7—C6         | 0.24 (13)    |
| N4—C14—C13—C12      | −149.66 (10) | C3—C4—C5—C6         | 0.00 (17)    |
| N3—C7—C6—C5         | −0.05 (14)   | C26a—C21a—C22a—C23a | 0.0 (3)      |
| N3—C3—C4—C5         | 0.19 (16)    | C26a—C25a—C24a—C23a | −0.0 (3)     |
| N5—C19—C18—C17      | −0.82 (13)   | C21a—C26a—C25a—C24a | 0.0 (4)      |
| N5—C15—C16—C17      | −1.03 (15)   | C21a—C22a—C23a—C24a | −0.0 (3)     |
| N6—C13—C12—C11      | 0.43 (14)    | C22a—C23a—C24a—C25a | 0.0 (4)      |
| N6—C9—C10—C11       | 0.53 (14)    | C22a—C21a—C26a—C25a | −0.0 (3)     |
| Cl1a—C21a—C26a—C25a | 179.20 (9)   | C22—C21—C26—C25     | −0.0 (4)     |
| Cl1a—C21a—C22a—C23a | −179.22 (9)  | C22—C23—C24—C25     | 0.0 (4)      |
| C13—N6—C9—C10       | −0.52 (12)   | Cl1—C21—C22—C23     | 176.16 (11)  |
| C13—C12—C11—C10     | −0.40 (14)   | Cl1—C21—C26—C25     | −175.98 (12) |
| C13—C14—N4—C20      | −157.38 (10) | C21—C22—C23—C24     | −0.0 (2)     |
| C13—C14—N4—C8       | 77.49 (10)   | C21—C26—C25—C24     | 0.0 (4)      |
| C7—N3—C3—C4         | −0.31 (13)   | C26—C21—C22—C23     | 0.0 (4)      |
| C7—C8—N4—C20        | 110.93 (10)  | C26—C25—C24—C23     | −0.0 (2)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_240K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.3247$  (3) Å

$b = 12.1065$  (2) Å

$c = 15.5211$  (2) Å

$\beta = 110.261$  (2)°

$V = 2701.43$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.414$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 61862 reflections

$\theta = 2.1$ – $31.8^\circ$

$\mu = 0.84$  mm<sup>−1</sup>

$T = 240$  K

Block, yellow

$0.36 \times 0.2 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector  
Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source  
Mirror monochromator  
Detector resolution: 10.0000 pixels mm<sup>-1</sup>  
 $\omega$  scans  
Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.533$ ,  $T_{\max} = 1.000$   
82537 measured reflections  
6713 independent reflections  
6179 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.012$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -20 \rightarrow 20$   
 $k = -16 \rightarrow 16$   
 $l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.075$   
 $S = 1.03$   
6713 reflections  
375 parameters  
2 restraints

56 constraints  
Primary atom site location: dual  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.8372P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.0003$   
 $\Delta\rho_{\max} = 0.94 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.70 \text{ e } \text{\AA}^{-3}$

*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  | 0.651741 (12) | 0.321378 (14) | 0.801298 (11) | 0.03473 (6)                      |           |
| S2   | 0.46769 (3)   | 0.00236 (3)   | 0.66186 (3)   | 0.05609 (10)                     |           |
| S1   | 0.75584 (11)  | 0.32580 (11)  | 0.53983 (8)   | 0.1005 (2)                       |           |
| N3   | 0.60647 (7)   | 0.33353 (9)   | 0.92091 (7)   | 0.0387 (2)                       |           |
| N4   | 0.73863 (7)   | 0.46231 (8)   | 0.87984 (7)   | 0.0346 (2)                       |           |
| N6   | 0.56299 (7)   | 0.46921 (9)   | 0.75365 (7)   | 0.0382 (2)                       |           |
| N5   | 0.78227 (7)   | 0.24256 (8)   | 0.88953 (7)   | 0.0371 (2)                       |           |
| N1   | 0.70327 (9)   | 0.34097 (10)  | 0.69217 (8)   | 0.0509 (3)                       |           |
| C13  | 0.85369 (9)   | 0.31242 (10)  | 0.92565 (9)   | 0.0383 (2)                       |           |
| Cl1a | 0.65943 (14)  | 0.03731 (12)  | 1.06121 (10)  | 0.1065 (7)                       | 0.506 (3) |
| C7   | 0.64467 (8)   | 0.41477 (10)  | 0.98008 (8)   | 0.0374 (2)                       |           |
| C19  | 0.60845 (9)   | 0.56562 (10)  | 0.77150 (8)   | 0.0371 (2)                       |           |
| N2   | 0.57098 (10)  | 0.18608 (11)  | 0.74666 (9)   | 0.0569 (3)                       |           |
| C18  | 0.56336 (10)  | 0.66627 (11)  | 0.74459 (10)  | 0.0469 (3)                       |           |
| C12  | 0.94017 (9)   | 0.27685 (12)  | 0.98353 (10)  | 0.0475 (3)                       |           |
| C20  | 0.71205 (9)   | 0.55888 (10)  | 0.81814 (10)  | 0.0434 (3)                       |           |
| C8   | 0.72231 (9)   | 0.48120 (11)  | 0.96704 (9)   | 0.0441 (3)                       |           |
| C9   | 0.79547 (10)  | 0.13492 (11)  | 0.91047 (9)   | 0.0430 (3)                       |           |
| C2   | 0.52797 (9)   | 0.10874 (11)  | 0.71185 (9)   | 0.0422 (3)                       |           |
| C14  | 0.83599 (8)   | 0.43132 (11)  | 0.89633 (10)  | 0.0425 (3)                       |           |
| C17  | 0.46851 (11)  | 0.66616 (13)  | 0.69657 (11)  | 0.0557 (4)                       |           |
| C15  | 0.47103 (10)  | 0.47001 (12)  | 0.70971 (9)   | 0.0483 (3)                       |           |
| C11  | 0.95361 (11)  | 0.16528 (13)  | 1.00444 (11)  | 0.0538 (4)                       |           |
| C10  | 0.88002 (11)  | 0.09295 (12)  | 0.96728 (10)  | 0.0518 (3)                       |           |
| C3   | 0.53949 (10)  | 0.27186 (13)  | 0.93397 (10)  | 0.0514 (3)                       |           |
| C6   | 0.61695 (11)  | 0.43650 (14)  | 1.05481 (10)  | 0.0538 (3)                       |           |
| C1   | 0.72511 (11)  | 0.33507 (12)  | 0.62814 (10)  | 0.0485 (3)                       |           |

|      |              |              |              |              |           |
|------|--------------|--------------|--------------|--------------|-----------|
| C16  | 0.42107 (11) | 0.56636 (14) | 0.67936 (11) | 0.0569 (4)   |           |
| C4   | 0.50849 (12) | 0.28853 (17) | 1.00671 (12) | 0.0647 (4)   |           |
| C26a | 0.6926 (3)   | 0.1120 (3)   | 1.2326 (3)   | 0.0642 (16)  | 0.506 (3) |
| C5   | 0.54801 (12) | 0.37237 (18) | 1.06796 (12) | 0.0665 (5)   |           |
| C23a | 0.8032 (3)   | 0.2842 (3)   | 1.2108 (4)   | 0.0724 (15)  | 0.506 (3) |
| C22a | 0.7622 (3)   | 0.2116 (3)   | 1.1386 (3)   | 0.0684 (13)  | 0.506 (3) |
| C21a | 0.7069 (3)   | 0.1256 (3)   | 1.1495 (2)   | 0.0553 (12)  | 0.506 (3) |
| C25a | 0.7336 (3)   | 0.1845 (4)   | 1.3047 (2)   | 0.0751 (13)  | 0.506 (3) |
| C24a | 0.7889 (3)   | 0.2706 (3)   | 1.2938 (3)   | 0.0764 (13)  | 0.506 (3) |
| C22  | 0.7292 (3)   | 0.1545 (5)   | 1.1048 (3)   | 0.0837 (17)  | 0.494 (3) |
| C11  | 0.59989 (18) | 0.00928 (14) | 1.09899 (14) | 0.1332 (9)   | 0.494 (3) |
| C21  | 0.6806 (3)   | 0.1032 (4)   | 1.1544 (4)   | 0.0789 (17)  | 0.494 (3) |
| C26  | 0.6955 (4)   | 0.1349 (4)   | 1.2445 (4)   | 0.084 (2)    | 0.494 (3) |
| C25  | 0.7591 (3)   | 0.2180 (4)   | 1.2850 (3)   | 0.0783 (14)  | 0.494 (3) |
| C23  | 0.7928 (3)   | 0.2376 (5)   | 1.1453 (4)   | 0.095 (3)    | 0.494 (3) |
| C24  | 0.8078 (3)   | 0.2693 (4)   | 1.2354 (4)   | 0.094 (3)    | 0.494 (3) |
| H1   | 0.50808 (10) | 0.20571 (13) | 0.88515 (10) | 0.0617 (4)*  |           |
| H2   | 0.45390 (12) | 0.23644 (17) | 1.01546 (12) | 0.0777 (5)*  |           |
| H3   | 0.52510 (12) | 0.38789 (18) | 1.12617 (12) | 0.0798 (5)*  |           |
| H4   | 0.64926 (11) | 0.50340 (14) | 1.10247 (10) | 0.0645 (4)*  |           |
| H5   | 0.43368 (10) | 0.39137 (12) | 0.69718 (9)  | 0.0579 (4)*  |           |
| H6   | 0.34601 (11) | 0.56383 (14) | 0.64271 (11) | 0.0683 (4)*  |           |
| H7   | 0.43152 (11) | 0.74366 (13) | 0.67254 (11) | 0.0668 (4)*  |           |
| H8   | 0.60204 (10) | 0.74388 (11) | 0.76104 (10) | 0.0562 (4)*  |           |
| H11  | 0.73726 (10) | 0.07799 (11) | 0.88159 (9)  | 0.0516 (3)*  |           |
| H12  | 0.88845 (11) | 0.00462 (12) | 0.98244 (10) | 0.0621 (4)*  |           |
| H13  | 1.02117 (11) | 0.13492 (13) | 1.04951 (11) | 0.0645 (4)*  |           |
| H14  | 0.99686 (9)  | 0.33581 (12) | 1.01223 (10) | 0.0570 (4)*  |           |
| H9   | 0.73719 (9)  | 0.63479 (10) | 0.85810 (10) | 0.0520 (3)*  |           |
| H10  | 0.74540 (9)  | 0.55288 (10) | 0.76586 (10) | 0.0520 (3)*  |           |
| H00c | 0.78703 (9)  | 0.46252 (11) | 1.02418 (9)  | 0.0529 (3)*  |           |
| H00d | 0.70710 (9)  | 0.56946 (11) | 0.97153 (9)  | 0.0529 (3)*  |           |
| H16  | 0.85182 (8)  | 0.44413 (11) | 0.83306 (10) | 0.0510 (3)*  |           |
| H15  | 0.88244 (8)  | 0.48485 (11) | 0.95012 (10) | 0.0510 (3)*  |           |
| H18  | 0.7734 (3)   | 0.2223 (3)   | 1.0733 (3)   | 0.0821 (16)* | 0.506 (3) |
| H20  | 0.8467 (3)   | 0.3518 (3)   | 1.2022 (4)   | 0.0869 (18)* | 0.506 (3) |
| H22  | 0.8212 (3)   | 0.3276 (3)   | 1.3505 (3)   | 0.0917 (16)* | 0.506 (3) |
| H24  | 0.7224 (3)   | 0.1739 (4)   | 1.3700 (2)   | 0.0902 (15)* | 0.506 (3) |
| H26  | 0.6491 (3)   | 0.0444 (3)   | 1.2412 (3)   | 0.0770 (19)* | 0.506 (3) |
| H25  | 0.6573 (4)   | 0.0945 (4)   | 1.2835 (4)   | 0.100 (3)*   | 0.494 (3) |
| H23  | 0.7709 (3)   | 0.2429 (4)   | 1.3558 (3)   | 0.0939 (17)* | 0.494 (3) |
| H21  | 0.8578 (3)   | 0.3347 (4)   | 1.2672 (4)   | 0.113 (3)*   | 0.494 (3) |
| H19  | 0.8311 (3)   | 0.2780 (5)   | 1.1063 (4)   | 0.114 (3)*   | 0.494 (3) |
| H17  | 0.7175 (3)   | 0.1296 (5)   | 1.0340 (3)   | 0.100 (2)*   | 0.494 (3) |

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

|      | $U^{11}$     | $U^{22}$    | $U^{33}$    | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|------|--------------|-------------|-------------|---------------|--------------|---------------|
| Fe1  | 0.03761 (10) | 0.03236 (9) | 0.03234 (9) | −0.00223 (6)  | 0.00973 (7)  | −0.00066 (6)  |
| S2   | 0.04832 (19) | 0.0492 (2)  | 0.0755 (3)  | −0.01562 (16) | 0.02751 (18) | −0.01737 (18) |
| S1   | 0.1503 (6)   | 0.1111 (5)  | 0.0645 (3)  | 0.0722 (4)    | 0.0683 (4)   | 0.0391 (3)    |
| N3   | 0.0366 (5)   | 0.0437 (6)  | 0.0361 (5)  | −0.0051 (4)   | 0.0129 (4)   | 0.0021 (4)    |
| N4   | 0.0340 (5)   | 0.0308 (5)  | 0.0378 (5)  | −0.0036 (4)   | 0.0111 (4)   | 0.0021 (4)    |
| N6   | 0.0388 (5)   | 0.0360 (5)  | 0.0356 (5)  | 0.0005 (4)    | 0.0075 (4)   | −0.0006 (4)   |
| N5   | 0.0398 (5)   | 0.0341 (5)  | 0.0350 (5)  | −0.0001 (4)   | 0.0098 (4)   | 0.0008 (4)    |
| N1   | 0.0664 (8)   | 0.0495 (6)  | 0.0424 (6)  | 0.0090 (6)    | 0.0260 (6)   | 0.0041 (5)    |
| C13  | 0.0358 (6)   | 0.0371 (6)  | 0.0397 (6)  | 0.0012 (5)    | 0.0101 (5)   | 0.0002 (5)    |
| Cl1a | 0.1448 (15)  | 0.0739 (8)  | 0.0917 (9)  | 0.0183 (8)    | 0.0295 (8)   | −0.0147 (6)   |
| C7   | 0.0371 (6)   | 0.0422 (6)  | 0.0323 (5)  | 0.0032 (5)    | 0.0111 (4)   | 0.0019 (5)    |
| C19  | 0.0414 (6)   | 0.0327 (6)  | 0.0358 (5)  | 0.0014 (5)    | 0.0115 (5)   | 0.0028 (4)    |
| N2   | 0.0591 (8)   | 0.0488 (7)  | 0.0563 (7)  | −0.0158 (6)   | 0.0117 (6)   | −0.0131 (6)   |
| C18  | 0.0517 (8)   | 0.0370 (7)  | 0.0496 (7)  | 0.0065 (6)    | 0.0146 (6)   | 0.0071 (5)    |
| C12  | 0.0374 (6)   | 0.0478 (7)  | 0.0507 (7)  | 0.0053 (6)    | 0.0069 (5)   | −0.0024 (6)   |
| C20  | 0.0420 (6)   | 0.0340 (6)  | 0.0517 (7)  | −0.0036 (5)   | 0.0133 (5)   | 0.0088 (5)    |
| C8   | 0.0475 (7)   | 0.0439 (7)  | 0.0397 (6)  | −0.0093 (5)   | 0.0136 (5)   | −0.0074 (5)   |
| C9   | 0.0540 (7)   | 0.0340 (6)  | 0.0384 (6)  | 0.0019 (5)    | 0.0128 (5)   | −0.0008 (5)   |
| C2   | 0.0399 (6)   | 0.0415 (7)  | 0.0459 (6)  | −0.0067 (5)   | 0.0157 (5)   | −0.0073 (5)   |
| C14  | 0.0344 (6)   | 0.0381 (6)  | 0.0531 (7)  | −0.0039 (5)   | 0.0128 (5)   | 0.0043 (5)    |
| C17  | 0.0538 (8)   | 0.0510 (8)  | 0.0574 (8)  | 0.0154 (7)    | 0.0130 (7)   | 0.0102 (7)    |
| C15  | 0.0419 (7)   | 0.0491 (8)  | 0.0448 (7)  | −0.0010 (6)   | 0.0035 (5)   | −0.0033 (6)   |
| C11  | 0.0499 (8)   | 0.0509 (8)  | 0.0522 (8)  | 0.0155 (6)    | 0.0071 (6)   | 0.0010 (6)    |
| C10  | 0.0626 (9)   | 0.0390 (7)  | 0.0484 (7)  | 0.0126 (6)    | 0.0125 (6)   | 0.0024 (6)    |
| C3   | 0.0436 (7)   | 0.0598 (9)  | 0.0529 (8)  | −0.0110 (6)   | 0.0193 (6)   | 0.0060 (7)    |
| C6   | 0.0563 (8)   | 0.0677 (10) | 0.0414 (7)  | 0.0058 (7)    | 0.0221 (6)   | −0.0035 (6)   |
| C1   | 0.0614 (8)   | 0.0474 (7)  | 0.0398 (7)  | 0.0179 (6)    | 0.0215 (6)   | 0.0096 (5)    |
| C16  | 0.0434 (7)   | 0.0624 (9)  | 0.0539 (8)  | 0.0097 (7)    | 0.0028 (6)   | 0.0029 (7)    |
| C4   | 0.0522 (8)   | 0.0858 (12) | 0.0657 (10) | −0.0057 (8)   | 0.0325 (8)   | 0.0161 (9)    |
| C26a | 0.088 (4)    | 0.058 (2)   | 0.071 (3)   | 0.008 (2)     | 0.058 (3)    | 0.016 (2)     |
| C5   | 0.0630 (10)  | 0.0952 (13) | 0.0536 (9)  | 0.0053 (9)    | 0.0358 (8)   | 0.0068 (9)    |
| C23a | 0.054 (3)    | 0.068 (3)   | 0.098 (4)   | 0.006 (2)     | 0.030 (2)    | 0.019 (3)     |
| C22a | 0.070 (3)    | 0.062 (3)   | 0.086 (3)   | 0.016 (2)     | 0.044 (3)    | 0.024 (2)     |
| C21a | 0.070 (3)    | 0.047 (2)   | 0.058 (2)   | 0.0178 (18)   | 0.033 (2)    | 0.0133 (17)   |
| C25a | 0.090 (3)    | 0.066 (3)   | 0.084 (3)   | 0.011 (2)     | 0.048 (3)    | 0.012 (2)     |
| C24a | 0.050 (2)    | 0.065 (3)   | 0.101 (4)   | 0.0039 (19)   | 0.009 (2)    | 0.004 (3)     |
| C22  | 0.080 (3)    | 0.109 (4)   | 0.081 (3)   | 0.040 (3)     | 0.051 (3)    | 0.049 (3)     |
| Cl1  | 0.176 (2)    | 0.0915 (10) | 0.1175 (13) | −0.0294 (11)  | 0.0323 (13)  | −0.0156 (9)   |
| C21  | 0.095 (4)    | 0.071 (3)   | 0.087 (4)   | 0.013 (3)     | 0.053 (3)    | 0.022 (3)     |
| C26  | 0.087 (5)    | 0.087 (4)   | 0.091 (5)   | −0.016 (3)    | 0.048 (4)    | −0.002 (4)    |
| C25  | 0.068 (3)    | 0.082 (4)   | 0.092 (4)   | −0.010 (2)    | 0.037 (3)    | 0.002 (3)     |
| C23  | 0.055 (3)    | 0.117 (5)   | 0.124 (5)   | 0.032 (3)     | 0.046 (3)    | 0.072 (5)     |
| C24  | 0.052 (3)    | 0.083 (4)   | 0.152 (6)   | 0.013 (3)     | 0.042 (4)    | 0.053 (4)     |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N3    | 2.2004 (11) | C17—C16     | 1.387 (2)   |
| Fe1—N4    | 2.2463 (10) | C17—H7      | 1.0930      |
| Fe1—N6    | 2.2144 (11) | C15—C16     | 1.385 (2)   |
| Fe1—N5    | 2.2113 (10) | C15—H5      | 1.0930      |
| Fe1—N1    | 2.1154 (12) | C11—C10     | 1.386 (2)   |
| Fe1—N2    | 2.0511 (12) | C11—H13     | 1.0930      |
| S2—C2     | 1.6179 (13) | C10—H12     | 1.0930      |
| S1—C1     | 1.6000 (17) | C3—C4       | 1.383 (2)   |
| N3—C7     | 1.3346 (16) | C3—H1       | 1.0930      |
| N3—C3     | 1.3403 (16) | C6—C5       | 1.382 (2)   |
| N4—C20    | 1.4763 (15) | C6—H4       | 1.0930      |
| N4—C8     | 1.4769 (16) | C16—H6      | 1.0930      |
| N4—C14    | 1.4724 (15) | C4—C5       | 1.379 (3)   |
| N6—C19    | 1.3382 (16) | C4—H2       | 1.0930      |
| N6—C15    | 1.3362 (17) | C26a—C21a   | 1.3900      |
| N5—C13    | 1.3426 (16) | C26a—C25a   | 1.3900      |
| N5—C9     | 1.3414 (16) | C26a—H26    | 1.0930      |
| N1—C1     | 1.1555 (18) | C5—H3       | 1.0930      |
| C13—C12   | 1.3871 (18) | C23a—C22a   | 1.3900      |
| C13—C14   | 1.5057 (17) | C23a—C24a   | 1.3900      |
| Cl1a—C21a | 1.691 (3)   | C23a—H20    | 1.0930      |
| C7—C8     | 1.5068 (17) | C22a—C21a   | 1.3900      |
| C7—C6     | 1.3915 (18) | C22a—H18    | 1.0930      |
| C19—C18   | 1.3917 (17) | C25a—C24a   | 1.3900      |
| C19—C20   | 1.5017 (18) | C25a—H24    | 1.0930      |
| N2—C2     | 1.1646 (18) | C24a—H22    | 1.0930      |
| C18—C17   | 1.385 (2)   | C22—C21     | 1.3900      |
| C18—H8    | 1.0930      | C22—C23     | 1.3900      |
| C12—C11   | 1.388 (2)   | C22—H17     | 1.0930      |
| C12—H14   | 1.0930      | Cl1—C21     | 1.681 (4)   |
| C20—H9    | 1.1010      | C21—C26     | 1.3900      |
| C20—H10   | 1.1010      | C26—C25     | 1.3900      |
| C8—H00c   | 1.1010      | C26—H25     | 1.0930      |
| C8—H00d   | 1.1010      | C25—C24     | 1.3900      |
| C9—C10    | 1.3868 (19) | C25—H23     | 1.0930      |
| C9—H11    | 1.0930      | C23—C24     | 1.3900      |
| C14—H16   | 1.1010      | C23—H19     | 1.0930      |
| C14—H15   | 1.1010      | C24—H21     | 1.0930      |
| N4—Fe1—N3 | 78.01 (4)   | H15—C14—C13 | 109.39 (7)  |
| N6—Fe1—N3 | 83.75 (4)   | H15—C14—H16 | 108.0       |
| N6—Fe1—N4 | 75.37 (4)   | C16—C17—C18 | 119.13 (13) |
| N5—Fe1—N3 | 89.33 (4)   | H7—C17—C18  | 120.44 (9)  |
| N5—Fe1—N4 | 75.68 (4)   | H7—C17—C16  | 120.44 (9)  |
| N5—Fe1—N6 | 151.03 (4)  | C16—C15—N6  | 122.73 (14) |
| N1—Fe1—N3 | 169.19 (5)  | H5—C15—N6   | 118.64 (8)  |



|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N1—Fe1—N4   | 91.94 (4)   | H5—C15—C16     | 118.64 (9)  |
| N1—Fe1—N6   | 89.91 (4)   | C10—C11—C12    | 118.95 (13) |
| N1—Fe1—N5   | 92.05 (4)   | H13—C11—C12    | 120.52 (9)  |
| N2—Fe1—N3   | 94.64 (5)   | H13—C11—C10    | 120.52 (8)  |
| N2—Fe1—N4   | 172.04 (5)  | C11—C10—C9     | 118.66 (13) |
| N2—Fe1—N6   | 107.13 (5)  | H12—C10—C9     | 120.67 (9)  |
| N2—Fe1—N5   | 101.44 (5)  | H12—C10—C11    | 120.67 (8)  |
| N2—Fe1—N1   | 95.60 (5)   | C4—C3—N3       | 122.67 (15) |
| C7—N3—Fe1   | 116.01 (8)  | H1—C3—N3       | 118.66 (9)  |
| C3—N3—Fe1   | 124.90 (10) | H1—C3—C4       | 118.66 (10) |
| C3—N3—C7    | 118.87 (12) | C5—C6—C7       | 119.11 (15) |
| C20—N4—Fe1  | 105.72 (7)  | H4—C6—C7       | 120.44 (9)  |
| C8—N4—Fe1   | 111.00 (7)  | H4—C6—C5       | 120.44 (10) |
| C8—N4—C20   | 112.05 (10) | N1—C1—S1       | 179.46 (13) |
| C14—N4—Fe1  | 105.98 (7)  | C15—C16—C17    | 118.47 (13) |
| C14—N4—C20  | 110.48 (10) | H6—C16—C17     | 120.76 (9)  |
| C14—N4—C8   | 111.29 (10) | H6—C16—C15     | 120.76 (9)  |
| C19—N6—Fe1  | 114.85 (8)  | C5—C4—C3       | 118.50 (15) |
| C15—N6—Fe1  | 126.41 (9)  | H2—C4—C3       | 120.75 (10) |
| C15—N6—C19  | 118.74 (11) | H2—C4—C5       | 120.75 (9)  |
| C13—N5—Fe1  | 114.74 (8)  | C25a—C26a—C21a | 120.0       |
| C9—N5—Fe1   | 126.69 (9)  | H26—C26a—C21a  | 120.00 (17) |
| C9—N5—C13   | 118.56 (11) | H26—C26a—C25a  | 120.00 (17) |
| C1—N1—Fe1   | 168.91 (13) | C4—C5—C6       | 119.17 (15) |
| C12—C13—N5  | 122.19 (12) | H3—C5—C6       | 120.42 (10) |
| C14—C13—N5  | 116.14 (11) | H3—C5—C4       | 120.42 (9)  |
| C14—C13—C12 | 121.62 (12) | C24a—C23a—C22a | 120.0       |
| C8—C7—N3    | 118.65 (11) | H20—C23a—C22a  | 120.00 (17) |
| C6—C7—N3    | 121.68 (13) | H20—C23a—C24a  | 120.00 (17) |
| C6—C7—C8    | 119.63 (12) | C21a—C22a—C23a | 120.0       |
| C18—C19—N6  | 122.19 (12) | H18—C22a—C23a  | 120.00 (17) |
| C20—C19—N6  | 116.11 (10) | H18—C22a—C21a  | 120.00 (17) |
| C20—C19—C18 | 121.63 (11) | C26a—C21a—C11a | 121.0       |
| C2—N2—Fe1   | 175.84 (13) | C22a—C21a—C11a | 119.0       |
| C17—C18—C19 | 118.70 (13) | C22a—C21a—C26a | 120.0       |
| H8—C18—C19  | 120.65 (8)  | C24a—C25a—C26a | 120.0       |
| H8—C18—C17  | 120.65 (9)  | H24—C25a—C26a  | 120.00 (17) |
| C11—C12—C13 | 119.00 (13) | H24—C25a—C24a  | 120.00 (17) |
| H14—C12—C13 | 120.50 (8)  | C25a—C24a—C23a | 120.0       |
| H14—C12—C11 | 120.50 (9)  | H22—C24a—C23a  | 120.00 (17) |
| C19—C20—N4  | 111.34 (10) | H22—C24a—C25a  | 120.00 (17) |
| H9—C20—N4   | 109.36 (7)  | C23—C22—C21    | 120.0       |
| H9—C20—C19  | 109.36 (7)  | H17—C22—C21    | 120.0 (2)   |
| H10—C20—N4  | 109.36 (7)  | H17—C22—C23    | 120.0 (2)   |
| H10—C20—C19 | 109.36 (7)  | C11—C21—C22    | 117.4       |
| H10—C20—H9  | 108.0       | C26—C21—C22    | 120.0       |
| C7—C8—N4    | 115.64 (10) | C26—C21—C11    | 122.5       |
| H00c—C8—N4  | 108.37 (7)  | C25—C26—C21    | 120.0       |

|                     |              |                     |              |
|---------------------|--------------|---------------------|--------------|
| H00c—C8—C7          | 108.37 (7)   | H25—C26—C21         | 120.0 (2)    |
| H00d—C8—N4          | 108.37 (7)   | H25—C26—C25         | 120.0 (2)    |
| H00d—C8—C7          | 108.37 (7)   | C24—C25—C26         | 120.0        |
| H00d—C8—H00c        | 107.4        | H23—C25—C26         | 120.0 (2)    |
| C10—C9—N5           | 122.64 (13)  | H23—C25—C24         | 120.0 (2)    |
| H11—C9—N5           | 118.68 (8)   | C24—C23—C22         | 120.0        |
| H11—C9—C10          | 118.68 (9)   | H19—C23—C22         | 120.0 (2)    |
| N2—C2—S2            | 179.04 (14)  | H19—C23—C24         | 120.0 (2)    |
| C13—C14—N4          | 111.21 (10)  | C23—C24—C25         | 120.0        |
| H16—C14—N4          | 109.39 (7)   | H21—C24—C25         | 120.0 (2)    |
| H16—C14—C13         | 109.39 (7)   | H21—C24—C23         | 120.0 (2)    |
| H15—C14—N4          | 109.39 (7)   |                     |              |
| Fe1—N3—C7—C8        | −7.25 (10)   | C7—C8—N4—C14        | −125.13 (11) |
| Fe1—N3—C7—C6        | 175.17 (9)   | C7—C6—C5—C4         | −0.00 (18)   |
| Fe1—N3—C3—C4        | −174.77 (13) | C19—N6—C15—C16      | 1.98 (14)    |
| Fe1—N4—C20—C19      | 43.73 (8)    | C19—C18—C17—C16     | 2.00 (16)    |
| Fe1—N4—C8—C7        | −7.34 (8)    | C19—C20—N4—C8       | −77.31 (12)  |
| Fe1—N4—C14—C13      | −43.43 (8)   | C19—C20—N4—C14      | 157.96 (11)  |
| Fe1—N6—C19—C18      | 178.71 (9)   | C18—C19—N6—C15      | −1.01 (15)   |
| Fe1—N6—C19—C20      | 1.74 (9)     | C18—C17—C16—C15     | −1.10 (18)   |
| Fe1—N6—C15—C16      | −177.69 (12) | C12—C13—N5—C9       | 0.05 (16)    |
| Fe1—N5—C13—C12      | 179.12 (9)   | C12—C11—C10—C9      | −0.03 (18)   |
| Fe1—N5—C13—C14      | −3.58 (9)    | C20—C19—N6—C15      | −177.98 (12) |
| Fe1—N5—C9—C10       | −179.45 (11) | C20—C19—C18—C17     | 175.83 (14)  |
| N3—C7—C8—N4         | 10.00 (12)   | C8—C7—N3—C3         | 177.83 (12)  |
| N3—C7—C6—C5         | −0.09 (15)   | C8—C7—C6—C5         | −177.64 (14) |
| N3—C3—C4—C5         | 0.26 (19)    | C9—N5—C13—C14       | 177.34 (12)  |
| N4—C20—C19—N6       | −31.96 (13)  | C14—C13—C12—C11     | −176.75 (14) |
| N4—C20—C19—C18      | 151.05 (11)  | C3—N3—C7—C6         | 0.26 (15)    |
| N4—C8—C7—C6         | −172.37 (12) | C3—C4—C5—C6         | −0.1 (2)     |
| N4—C14—C13—N5       | 33.00 (12)   | C26a—C21a—C22a—C23a | −0.0 (3)     |
| N4—C14—C13—C12      | −149.69 (11) | C26a—C25a—C24a—C23a | −0.0 (3)     |
| N6—C19—C18—C17      | −0.97 (15)   | C22a—C23a—C24a—C25a | 0.0 (4)      |
| N6—C15—C16—C17      | −0.93 (18)   | C22a—C21a—C26a—C25a | 0.0 (3)      |
| N5—C13—C12—C11      | 0.39 (15)    | C21a—C26a—C25a—C24a | −0.0 (4)     |
| N5—C9—C10—C11       | 0.49 (16)    | C21a—C22a—C23a—C24a | −0.0 (3)     |
| C13—N5—C9—C10       | −0.50 (14)   | C22—C21—C26—C25     | 0.0 (4)      |
| C13—C12—C11—C10     | −0.39 (16)   | C22—C23—C24—C25     | 0.0 (5)      |
| C13—C14—N4—C20      | −157.50 (11) | C11—C21—C22—C23     | 176.12 (14)  |
| C13—C14—N4—C8       | 77.34 (12)   | C11—C21—C26—C25     | −175.92 (15) |
| C11a—C21a—C26a—C25a | 178.91 (11)  | C21—C22—C23—C24     | −0.0 (3)     |
| C11a—C21a—C22a—C23a | −178.93 (11) | C21—C26—C25—C24     | 0.0 (5)      |
| C7—N3—C3—C4         | −0.35 (15)   | C26—C21—C22—C23     | 0.0 (4)      |
| C7—C8—N4—C20        | 110.60 (12)  | C26—C25—C24—C23     | −0.0 (3)     |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) chlorobenzene monosolvate (cl\_270K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>Cl

$M_r = 574.94$

Monoclinic,  $P2_1/n$

$a = 15.3728$  (3) Å

$b = 12.1343$  (2) Å

$c = 15.5638$  (3) Å

$\beta = 110.308$  (2)°

$V = 2722.78$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1187.598$

$D_x = 1.403$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 58161 reflections

$\theta = 2.1\text{--}31.7^\circ$

$\mu = 0.83$  mm<sup>-1</sup>

$T = 270$  K

Block, yellow

$0.34 \times 0.21 \times 0.17$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.537$ ,  $T_{\max} = 1.000$

80082 measured reflections

6761 independent reflections

6119 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.012$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.097$

$S = 1.03$

6761 reflections

375 parameters

9 restraints

56 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.7942P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.0003$

$\Delta\rho_{\max} = 1.01$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.76$  e Å<sup>-3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Fe1 | 0.347521 (14) | 0.321700 (16) | 0.198652 (13) | 0.04004 (7)                      |           |
| S2  | 0.53189 (3)   | 0.00397 (4)   | 0.33725 (4)   | 0.06522 (13)                     |           |
| S1  | 0.24516 (13)  | 0.32645 (13)  | 0.45987 (10)  | 0.1136 (3)                       |           |
| N3  | 0.39278 (9)   | 0.33383 (10)  | 0.07927 (9)   | 0.0449 (3)                       |           |
| N4  | 0.26071 (8)   | 0.46225 (9)   | 0.11989 (8)   | 0.0401 (2)                       |           |
| N5  | 0.21704 (8)   | 0.24312 (10)  | 0.11059 (8)   | 0.0427 (2)                       |           |
| N6  | 0.43603 (8)   | 0.46936 (10)  | 0.24625 (8)   | 0.0439 (3)                       |           |
| N1  | 0.29635 (11)  | 0.34131 (12)  | 0.30765 (10)  | 0.0581 (3)                       |           |
| N2  | 0.42824 (12)  | 0.18679 (13)  | 0.25317 (11)  | 0.0647 (4)                       |           |
| C13 | 0.14597 (10)  | 0.31284 (12)  | 0.07472 (10)  | 0.0444 (3)                       |           |
| C19 | 0.39075 (10)  | 0.56545 (12)  | 0.22761 (9)   | 0.0430 (3)                       |           |
| C7  | 0.35448 (10)  | 0.41464 (12)  | 0.02009 (9)   | 0.0440 (3)                       |           |
| C9  | 0.20378 (12)  | 0.13560 (12)  | 0.08988 (10)  | 0.0496 (3)                       |           |
| C18 | 0.43580 (12)  | 0.66594 (13)  | 0.25411 (12)  | 0.0546 (4)                       |           |

|      |               |              |               |             |           |
|------|---------------|--------------|---------------|-------------|-----------|
| C20  | 0.28758 (11)  | 0.55889 (12) | 0.18107 (11)  | 0.0500 (3)  |           |
| C12  | 0.05961 (11)  | 0.27740 (15) | 0.01723 (12)  | 0.0548 (4)  |           |
| C8   | 0.27673 (11)  | 0.48040 (14) | 0.03295 (10)  | 0.0515 (3)  |           |
| C2   | 0.47144 (10)  | 0.10975 (13) | 0.28759 (11)  | 0.0486 (3)  |           |
| C14  | 0.16381 (10)  | 0.43144 (12) | 0.10377 (12)  | 0.0490 (3)  |           |
| C15  | 0.52756 (11)  | 0.47028 (15) | 0.29029 (11)  | 0.0555 (4)  |           |
| C11  | 0.04609 (13)  | 0.16608 (15) | −0.00338 (13) | 0.0616 (4)  |           |
| C1   | 0.27505 (13)  | 0.33560 (14) | 0.37175 (11)  | 0.0551 (4)  |           |
| C10  | 0.11933 (13)  | 0.09393 (14) | 0.03347 (12)  | 0.0596 (4)  |           |
| C3   | 0.45979 (12)  | 0.27280 (16) | 0.06635 (13)  | 0.0599 (4)  |           |
| C17  | 0.53036 (14)  | 0.66590 (16) | 0.30221 (14)  | 0.0643 (5)  |           |
| C16  | 0.57749 (13)  | 0.56623 (17) | 0.32021 (13)  | 0.0653 (5)  |           |
| C6   | 0.38217 (13)  | 0.43626 (17) | −0.05434 (12) | 0.0628 (4)  |           |
| C4   | 0.49070 (14)  | 0.2896 (2)   | −0.00594 (15) | 0.0751 (6)  |           |
| Cl1  | 0.3992 (2)    | 0.00927 (19) | −0.0989 (2)   | 0.1588 (14) | 0.495 (4) |
| C26a | 0.3082 (4)    | 0.1130 (4)   | −0.2322 (4)   | 0.0738 (18) | 0.505 (4) |
| C5   | 0.45112 (15)  | 0.3726 (2)   | −0.06718 (15) | 0.0775 (6)  |           |
| C23a | 0.1962 (3)    | 0.2839 (4)   | −0.2125 (5)   | 0.087 (2)   | 0.505 (4) |
| C22a | 0.2366 (4)    | 0.2119 (4)   | −0.1402 (3)   | 0.0845 (19) | 0.505 (4) |
| C21a | 0.2926 (3)    | 0.1264 (3)   | −0.1500 (3)   | 0.0697 (16) | 0.505 (4) |
| C25a | 0.2678 (4)    | 0.1851 (4)   | −0.3045 (3)   | 0.0885 (16) | 0.505 (4) |
| C24a | 0.2117 (3)    | 0.2705 (4)   | −0.2947 (4)   | 0.0913 (18) | 0.505 (4) |
| Cl1a | 0.33847 (19)  | 0.03850 (16) | −0.06172 (14) | 0.1251 (11) | 0.505 (4) |
| C22  | 0.2702 (4)    | 0.1535 (6)   | −0.1048 (3)   | 0.102 (3)   | 0.495 (4) |
| C21  | 0.3186 (5)    | 0.1013 (5)   | −0.1539 (5)   | 0.099 (2)   | 0.495 (4) |
| C26  | 0.3042 (4)    | 0.1325 (5)   | −0.2437 (4)   | 0.105 (3)   | 0.495 (4) |
| C25  | 0.2414 (4)    | 0.2158 (5)   | −0.2845 (4)   | 0.096 (2)   | 0.495 (4) |
| C24  | 0.1930 (3)    | 0.2680 (5)   | −0.2354 (5)   | 0.114 (4)   | 0.495 (4) |
| C23  | 0.2074 (4)    | 0.2368 (7)   | −0.1456 (5)   | 0.116 (4)   | 0.495 (4) |
| H1   | 0.49103 (12)  | 0.20753 (16) | 0.11468 (13)  | 0.0718 (5)* |           |
| H2   | 0.54497 (14)  | 0.2385 (2)   | −0.01427 (15) | 0.0902 (7)* |           |
| H3   | 0.47373 (15)  | 0.3879 (2)   | −0.12471 (15) | 0.0930 (7)* |           |
| H4   | 0.35006 (13)  | 0.50217 (17) | −0.10163 (12) | 0.0753 (5)* |           |
| H13  | 0.26127 (12)  | 0.07926 (12) | 0.11835 (10)  | 0.0596 (4)* |           |
| H14  | 0.11083 (13)  | 0.00661 (14) | 0.01851 (12)  | 0.0715 (5)* |           |
| H15  | −0.02080 (13) | 0.13609 (15) | −0.04767 (13) | 0.0739 (5)* |           |
| H16  | 0.00360 (11)  | 0.33571 (15) | −0.01112 (12) | 0.0658 (4)* |           |
| H7   | 0.56437 (11)  | 0.39248 (15) | 0.30319 (11)  | 0.0666 (4)* |           |
| H8   | 0.65159 (13)  | 0.56362 (17) | 0.35682 (13)  | 0.0783 (5)* |           |
| H9   | 0.56701 (14)  | 0.74260 (16) | 0.32543 (14)  | 0.0771 (5)* |           |
| H10  | 0.39762 (12)  | 0.74267 (13) | 0.23744 (12)  | 0.0655 (4)* |           |
| H11  | 0.26290 (11)  | 0.63378 (12) | 0.14127 (11)  | 0.0599 (4)* |           |
| H12  | 0.25461 (11)  | 0.55359 (12) | 0.23274 (11)  | 0.0599 (4)* |           |
| H5   | 0.21281 (11)  | 0.46109 (14) | −0.02333 (10) | 0.0618 (4)* |           |
| H6   | 0.29113 (11)  | 0.56772 (14) | 0.02788 (10)  | 0.0618 (4)* |           |
| H18  | 0.14845 (10)  | 0.44429 (12) | 0.16641 (12)  | 0.0588 (4)* |           |
| H17  | 0.11778 (10)  | 0.48431 (12) | 0.05062 (12)  | 0.0588 (4)* |           |
| H27  | 0.3419 (4)    | 0.0919 (5)   | −0.2820 (4)   | 0.126 (4)*  | 0.495 (4) |

|     |            |            |             |              |           |
|-----|------------|------------|-------------|--------------|-----------|
| H25 | 0.2301 (4) | 0.2401 (5) | −0.3545 (4) | 0.115 (2)*   | 0.495 (4) |
| H23 | 0.1440 (3) | 0.3329 (5) | −0.2672 (5) | 0.136 (5)*   | 0.495 (4) |
| H21 | 0.1697 (4) | 0.2775 (7) | −0.1073 (5) | 0.139 (5)*   | 0.495 (4) |
| H19 | 0.2814 (4) | 0.1292 (6) | −0.0348 (3) | 0.122 (3)*   | 0.495 (4) |
| H20 | 0.2245 (4) | 0.2223 (4) | −0.0762 (3) | 0.101 (2)*   | 0.505 (4) |
| H22 | 0.1525 (3) | 0.3505 (4) | −0.2048 (5) | 0.104 (2)*   | 0.505 (4) |
| H24 | 0.1802 (3) | 0.3267 (4) | −0.3510 (4) | 0.110 (2)*   | 0.505 (4) |
| H26 | 0.2799 (4) | 0.1746 (4) | −0.3686 (3) | 0.1062 (19)* | 0.505 (4) |
| H28 | 0.3518 (4) | 0.0464 (4) | −0.2399 (4) | 0.089 (2)*   | 0.505 (4) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|      | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|------|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1  | 0.04324 (12) | 0.03672 (11) | 0.03753 (11) | 0.00309 (7)  | 0.01068 (8) | 0.00117 (7)  |
| S2   | 0.0566 (2)   | 0.0569 (3)   | 0.0871 (3)   | 0.01916 (19) | 0.0313 (2)  | 0.0207 (2)   |
| S1   | 0.1715 (8)   | 0.1225 (6)   | 0.0744 (4)   | −0.0789 (5)  | 0.0778 (5)  | −0.0436 (4)  |
| N3   | 0.0426 (6)   | 0.0504 (7)   | 0.0420 (6)   | 0.0057 (5)   | 0.0149 (5)  | −0.0025 (5)  |
| N4   | 0.0394 (5)   | 0.0348 (5)   | 0.0440 (6)   | 0.0045 (4)   | 0.0117 (5)  | −0.0021 (4)  |
| N5   | 0.0457 (6)   | 0.0386 (6)   | 0.0412 (6)   | 0.0002 (5)   | 0.0117 (5)  | −0.0013 (5)  |
| N6   | 0.0447 (6)   | 0.0410 (6)   | 0.0408 (6)   | −0.0001 (5)  | 0.0082 (5)  | 0.0010 (5)   |
| N1   | 0.0751 (9)   | 0.0568 (8)   | 0.0487 (7)   | −0.0105 (7)  | 0.0294 (7)  | −0.0050 (6)  |
| N2   | 0.0670 (9)   | 0.0550 (8)   | 0.0643 (9)   | 0.0190 (7)   | 0.0130 (7)  | 0.0150 (7)   |
| C13  | 0.0413 (7)   | 0.0424 (7)   | 0.0464 (7)   | −0.0014 (5)  | 0.0114 (6)  | −0.0006 (6)  |
| C19  | 0.0475 (7)   | 0.0376 (7)   | 0.0415 (6)   | −0.0018 (5)  | 0.0125 (6)  | −0.0033 (5)  |
| C7   | 0.0434 (7)   | 0.0490 (7)   | 0.0380 (6)   | −0.0046 (6)  | 0.0122 (5)  | −0.0025 (6)  |
| C9   | 0.0618 (9)   | 0.0379 (7)   | 0.0454 (7)   | −0.0020 (6)  | 0.0139 (7)  | 0.0009 (6)   |
| C18  | 0.0601 (10)  | 0.0427 (8)   | 0.0577 (9)   | −0.0079 (7)  | 0.0163 (7)  | −0.0079 (6)  |
| C20  | 0.0488 (8)   | 0.0380 (7)   | 0.0596 (9)   | 0.0043 (6)   | 0.0144 (7)  | −0.0097 (6)  |
| C12  | 0.0428 (8)   | 0.0546 (9)   | 0.0594 (9)   | −0.0059 (7)  | 0.0080 (7)  | 0.0018 (7)   |
| C8   | 0.0549 (8)   | 0.0508 (8)   | 0.0464 (7)   | 0.0103 (7)   | 0.0146 (6)  | 0.0085 (6)   |
| C2   | 0.0456 (7)   | 0.0466 (8)   | 0.0540 (8)   | 0.0077 (6)   | 0.0179 (6)  | 0.0083 (6)   |
| C14  | 0.0400 (7)   | 0.0431 (7)   | 0.0615 (9)   | 0.0050 (6)   | 0.0144 (6)  | −0.0047 (6)  |
| C15  | 0.0476 (8)   | 0.0569 (9)   | 0.0516 (8)   | 0.0013 (7)   | 0.0038 (6)  | 0.0033 (7)   |
| C11  | 0.0570 (10)  | 0.0573 (10)  | 0.0610 (10)  | −0.0173 (8)  | 0.0084 (8)  | −0.0019 (8)  |
| C1   | 0.0696 (10)  | 0.0526 (8)   | 0.0460 (8)   | −0.0191 (7)  | 0.0236 (7)  | −0.0109 (6)  |
| C10  | 0.0718 (11)  | 0.0439 (8)   | 0.0570 (9)   | −0.0144 (8)  | 0.0146 (8)  | −0.0033 (7)  |
| C3   | 0.0509 (8)   | 0.0698 (11)  | 0.0614 (10)  | 0.0123 (8)   | 0.0227 (7)  | −0.0067 (8)  |
| C17  | 0.0617 (10)  | 0.0583 (10)  | 0.0667 (11)  | −0.0189 (8)  | 0.0146 (8)  | −0.0113 (8)  |
| C16  | 0.0503 (9)   | 0.0716 (12)  | 0.0615 (10)  | −0.0118 (8)  | 0.0038 (7)  | −0.0040 (9)  |
| C6   | 0.0668 (10)  | 0.0778 (12)  | 0.0487 (8)   | −0.0071 (9)  | 0.0262 (8)  | 0.0040 (8)   |
| C4   | 0.0608 (11)  | 0.1001 (15)  | 0.0754 (12)  | 0.0064 (11)  | 0.0374 (10) | −0.0174 (12) |
| Cl1  | 0.209 (3)    | 0.1085 (14)  | 0.146 (2)    | 0.0329 (16)  | 0.0465 (19) | 0.0204 (13)  |
| C26a | 0.092 (4)    | 0.073 (3)    | 0.081 (4)    | −0.008 (2)   | 0.061 (3)   | −0.022 (2)   |
| C5   | 0.0740 (12)  | 0.1101 (18)  | 0.0627 (11)  | −0.0066 (12) | 0.0420 (10) | −0.0073 (11) |
| C23a | 0.071 (4)    | 0.082 (4)    | 0.111 (5)    | −0.008 (3)   | 0.037 (3)   | −0.023 (4)   |
| C22a | 0.082 (4)    | 0.072 (3)    | 0.114 (5)    | −0.018 (3)   | 0.053 (4)   | −0.031 (3)   |
| C21a | 0.088 (3)    | 0.056 (2)    | 0.072 (3)    | −0.027 (2)   | 0.036 (3)   | −0.018 (2)   |
| C25a | 0.092 (4)    | 0.077 (3)    | 0.114 (4)    | −0.010 (2)   | 0.058 (3)   | −0.014 (2)   |

|      |           |             |             |              |             |            |
|------|-----------|-------------|-------------|--------------|-------------|------------|
| C24a | 0.056 (3) | 0.078 (3)   | 0.134 (5)   | −0.008 (2)   | 0.026 (3)   | −0.019 (3) |
| Cl1a | 0.173 (2) | 0.0877 (11) | 0.1086 (12) | −0.0222 (12) | 0.0405 (12) | 0.0145 (9) |
| C22  | 0.096 (4) | 0.134 (6)   | 0.101 (4)   | −0.053 (4)   | 0.066 (4)   | −0.070 (5) |
| C21  | 0.123 (6) | 0.089 (5)   | 0.105 (6)   | −0.016 (4)   | 0.063 (4)   | −0.025 (4) |
| C26  | 0.113 (7) | 0.105 (5)   | 0.116 (7)   | 0.016 (5)    | 0.062 (5)   | 0.002 (5)  |
| C25  | 0.068 (3) | 0.098 (5)   | 0.129 (5)   | 0.011 (3)    | 0.043 (3)   | −0.016 (4) |
| C24  | 0.055 (4) | 0.105 (5)   | 0.189 (9)   | −0.017 (3)   | 0.051 (5)   | −0.073 (6) |
| C23  | 0.066 (4) | 0.145 (8)   | 0.153 (8)   | −0.042 (5)   | 0.058 (4)   | −0.092 (7) |

*Geometric parameters (Å, °)*

|         |             |           |           |
|---------|-------------|-----------|-----------|
| Fe1—N3  | 2.2034 (13) | C15—H7    | 1.0830    |
| Fe1—N4  | 2.2487 (11) | C11—C10   | 1.384 (3) |
| Fe1—N5  | 2.2143 (12) | C11—H15   | 1.0830    |
| Fe1—N6  | 2.2160 (12) | C10—H14   | 1.0830    |
| Fe1—N1  | 2.1165 (15) | C3—C4     | 1.380 (3) |
| Fe1—N2  | 2.0516 (14) | C3—H1     | 1.0830    |
| S2—C2   | 1.6155 (16) | C17—C16   | 1.387 (3) |
| S1—C1   | 1.594 (2)   | C17—H9    | 1.0830    |
| N3—C7   | 1.3343 (19) | C16—H8    | 1.0830    |
| N3—C3   | 1.3393 (19) | C6—C5     | 1.381 (3) |
| N4—C20  | 1.4763 (17) | C6—H4     | 1.0830    |
| N4—C8   | 1.4735 (19) | C4—C5     | 1.375 (3) |
| N4—C14  | 1.4706 (18) | C4—H2     | 1.0830    |
| N5—C13  | 1.3408 (18) | Cl1—C21   | 1.667 (5) |
| N5—C9   | 1.3424 (19) | C26a—C21a | 1.3900    |
| N6—C19  | 1.3371 (18) | C26a—C25a | 1.3900    |
| N6—C15  | 1.3343 (19) | C26a—H28  | 1.0830    |
| N1—C1   | 1.155 (2)   | C5—H3     | 1.0830    |
| N2—C2   | 1.164 (2)   | C23a—C22a | 1.3900    |
| C13—C12 | 1.387 (2)   | C23a—C24a | 1.3900    |
| C13—C14 | 1.505 (2)   | C23a—H22  | 1.0830    |
| C19—C18 | 1.392 (2)   | C22a—C21a | 1.3900    |
| C19—C20 | 1.500 (2)   | C22a—H20  | 1.0830    |
| C7—C8   | 1.507 (2)   | C21a—Cl1a | 1.689 (4) |
| C7—C6   | 1.391 (2)   | C25a—C24a | 1.3900    |
| C9—C10  | 1.386 (2)   | C25a—H26  | 1.0830    |
| C9—H13  | 1.0830      | C24a—H24  | 1.0830    |
| C18—C17 | 1.385 (3)   | C22—C21   | 1.3900    |
| C18—H10 | 1.0830      | C22—C23   | 1.3900    |
| C20—H11 | 1.0910      | C22—H19   | 1.0830    |
| C20—H12 | 1.0910      | C21—C26   | 1.3900    |
| C12—C11 | 1.387 (2)   | C26—C25   | 1.3900    |
| C12—H16 | 1.0830      | C26—H27   | 1.0830    |
| C8—H5   | 1.0910      | C25—C24   | 1.3900    |
| C8—H6   | 1.0910      | C25—H25   | 1.0830    |
| C14—H18 | 1.0910      | C24—C23   | 1.3900    |
| C14—H17 | 1.0910      | C24—H23   | 1.0830    |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| C15—C16     | 1.383 (2)   | C23—H21        | 1.0830      |
| N4—Fe1—N3   | 77.94 (4)   | H17—C14—C13    | 109.38 (8)  |
| N5—Fe1—N3   | 89.43 (4)   | H17—C14—H18    | 108.0       |
| N5—Fe1—N4   | 75.55 (4)   | C16—C15—N6     | 122.89 (16) |
| N6—Fe1—N3   | 83.74 (5)   | H7—C15—N6      | 118.56 (9)  |
| N6—Fe1—N4   | 75.43 (4)   | H7—C15—C16     | 118.56 (11) |
| N6—Fe1—N5   | 150.96 (4)  | C10—C11—C12    | 119.02 (16) |
| N1—Fe1—N3   | 169.21 (6)  | H15—C11—C12    | 120.49 (10) |
| N1—Fe1—N4   | 92.08 (5)   | H15—C11—C10    | 120.49 (10) |
| N1—Fe1—N5   | 92.05 (5)   | N1—C1—S1       | 179.38 (16) |
| N1—Fe1—N6   | 89.83 (5)   | C11—C10—C9     | 118.70 (16) |
| N2—Fe1—N3   | 94.55 (6)   | H14—C10—C9     | 120.65 (10) |
| N2—Fe1—N4   | 171.88 (6)  | H14—C10—C11    | 120.65 (10) |
| N2—Fe1—N5   | 101.56 (6)  | C4—C3—N3       | 122.77 (19) |
| N2—Fe1—N6   | 107.09 (6)  | H1—C3—N3       | 118.62 (10) |
| N2—Fe1—N1   | 95.62 (7)   | H1—C3—C4       | 118.62 (13) |
| C7—N3—Fe1   | 116.00 (9)  | C16—C17—C18    | 119.08 (16) |
| C3—N3—Fe1   | 125.04 (12) | H9—C17—C18     | 120.46 (10) |
| C3—N3—C7    | 118.74 (14) | H9—C17—C16     | 120.46 (10) |
| C20—N4—Fe1  | 105.70 (9)  | C17—C16—C15    | 118.41 (16) |
| C8—N4—Fe1   | 110.93 (8)  | H8—C16—C15     | 120.79 (11) |
| C8—N4—C20   | 112.03 (12) | H8—C16—C17     | 120.79 (10) |
| C14—N4—Fe1  | 105.93 (9)  | C5—C6—C7       | 119.16 (19) |
| C14—N4—C20  | 110.61 (11) | H4—C6—C7       | 120.42 (11) |
| C14—N4—C8   | 111.32 (12) | H4—C6—C5       | 120.42 (12) |
| C13—N5—Fe1  | 114.72 (9)  | C5—C4—C3       | 118.59 (18) |
| C9—N5—Fe1   | 126.63 (10) | H2—C4—C3       | 120.70 (13) |
| C9—N5—C13   | 118.65 (13) | H2—C4—C5       | 120.70 (11) |
| C19—N6—Fe1  | 114.81 (9)  | C25a—C26a—C21a | 120.0       |
| C15—N6—Fe1  | 126.45 (11) | H28—C26a—C21a  | 120.0 (2)   |
| C15—N6—C19  | 118.74 (13) | H28—C26a—C25a  | 120.0 (2)   |
| C1—N1—Fe1   | 168.88 (15) | C4—C5—C6       | 119.12 (18) |
| C2—N2—Fe1   | 176.15 (16) | H3—C5—C6       | 120.44 (12) |
| C12—C13—N5  | 122.15 (14) | H3—C5—C4       | 120.44 (11) |
| C14—C13—N5  | 116.18 (13) | C24a—C23a—C22a | 120.0       |
| C14—C13—C12 | 121.62 (14) | H22—C23a—C22a  | 120.0 (2)   |
| C18—C19—N6  | 122.11 (14) | H22—C23a—C24a  | 120.0 (2)   |
| C20—C19—N6  | 116.24 (12) | C21a—C22a—C23a | 120.0       |
| C20—C19—C18 | 121.56 (14) | H20—C22a—C23a  | 120.0 (2)   |
| C8—C7—N3    | 118.54 (13) | H20—C22a—C21a  | 120.0 (2)   |
| C6—C7—N3    | 121.62 (15) | C22a—C21a—C26a | 120.0       |
| C6—C7—C8    | 119.79 (15) | Cl1a—C21a—C26a | 121.2       |
| C10—C9—N5   | 122.52 (16) | Cl1a—C21a—C22a | 118.8       |
| H13—C9—N5   | 118.74 (9)  | C24a—C25a—C26a | 120.0       |
| H13—C9—C10  | 118.74 (10) | H26—C25a—C26a  | 120.0 (2)   |
| C17—C18—C19 | 118.73 (16) | H26—C25a—C24a  | 120.0 (2)   |
| H10—C18—C19 | 120.64 (10) | C25a—C24a—C23a | 120.0       |



|                 |              |                     |              |
|-----------------|--------------|---------------------|--------------|
| H10—C18—C17     | 120.64 (10)  | H24—C24a—C23a       | 120.0 (2)    |
| C19—C20—N4      | 111.46 (11)  | H24—C24a—C25a       | 120.0 (2)    |
| H11—C20—N4      | 109.33 (8)   | C23—C22—C21         | 120.0        |
| H11—C20—C19     | 109.33 (8)   | H19—C22—C21         | 120.0        |
| H12—C20—N4      | 109.33 (8)   | H19—C22—C23         | 120.0        |
| H12—C20—C19     | 109.33 (9)   | C22—C21—Cl1         | 117.8 (5)    |
| H12—C20—H11     | 108.0        | C26—C21—Cl1         | 122.0 (5)    |
| C11—C12—C13     | 118.96 (16)  | C26—C21—C22         | 120.0        |
| H16—C12—C13     | 120.52 (10)  | C25—C26—C21         | 120.0        |
| H16—C12—C11     | 120.52 (10)  | H27—C26—C21         | 120.0 (3)    |
| C7—C8—N4        | 115.84 (12)  | H27—C26—C25         | 120.0 (3)    |
| H5—C8—N4        | 108.32 (8)   | C24—C25—C26         | 120.0        |
| H5—C8—C7        | 108.32 (8)   | H25—C25—C26         | 120.0 (3)    |
| H6—C8—N4        | 108.32 (8)   | H25—C25—C24         | 120.0 (3)    |
| H6—C8—C7        | 108.32 (9)   | C23—C24—C25         | 120.0        |
| H6—C8—H5        | 107.4        | H23—C24—C25         | 120.0 (3)    |
| N2—C2—S2        | 178.91 (16)  | H23—C24—C23         | 120.0 (3)    |
| C13—C14—N4      | 111.26 (12)  | C24—C23—C22         | 120.0        |
| H18—C14—N4      | 109.38 (8)   | H21—C23—C22         | 120.0 (3)    |
| H18—C14—C13     | 109.38 (9)   | H21—C23—C24         | 120.0 (3)    |
| H17—C14—N4      | 109.38 (8)   |                     |              |
| Fe1—N3—C7—C8    | −7.36 (12)   | C7—N3—C3—C4         | −0.32 (18)   |
| Fe1—N3—C7—C6    | 175.15 (11)  | C7—C8—N4—C20        | 110.01 (14)  |
| Fe1—N3—C3—C4    | −174.64 (15) | C7—C8—N4—C14        | −125.55 (13) |
| Fe1—N4—C20—C19  | 43.49 (9)    | C7—C6—C5—C4         | −0.1 (2)     |
| Fe1—N4—C8—C7    | −7.85 (9)    | C9—N5—C13—C12       | 0.05 (16)    |
| Fe1—N4—C14—C13  | −43.58 (9)   | C9—N5—C13—C14       | 177.35 (14)  |
| Fe1—N5—C13—C12  | 179.29 (11)  | C9—C10—C11—C12      | 0.1 (2)      |
| Fe1—N5—C13—C14  | −3.41 (11)   | C18—C19—N6—C15      | −1.16 (18)   |
| Fe1—N5—C9—C10   | −179.63 (13) | C18—C17—C16—C15     | −0.9 (2)     |
| Fe1—N6—C19—C18  | 178.93 (11)  | C20—C19—N6—C15      | −177.84 (14) |
| Fe1—N6—C19—C20  | 2.25 (11)    | C20—C19—C18—C17     | 175.76 (17)  |
| Fe1—N6—C15—C16  | −178.00 (14) | C8—C7—N3—C3         | 177.81 (15)  |
| N3—C7—C8—N4     | 10.45 (14)   | C8—C7—C6—C5         | −177.58 (18) |
| N3—C7—C6—C5     | −0.12 (19)   | C14—C13—C12—C11     | −176.72 (17) |
| N3—C3—C4—C5     | 0.1 (2)      | C3—N3—C7—C6         | 0.32 (18)    |
| N4—C20—C19—N6   | −32.19 (15)  | C3—C4—C5—C6         | 0.1 (2)      |
| N4—C20—C19—C18  | 151.11 (13)  | Cl1—C21—C22—C23     | 175.11 (19)  |
| N4—C8—C7—C6     | −172.02 (14) | Cl1—C21—C26—C25     | −174.90 (19) |
| N4—C14—C13—N5   | 33.01 (15)   | C26a—C21a—C22a—C23a | −0.0 (4)     |
| N4—C14—C13—C12  | −149.67 (13) | C26a—C25a—C24a—C23a | 0.0 (4)      |
| N5—C13—C12—C11  | 0.43 (18)    | C23a—C22a—C21a—Cl1a | −178.47 (13) |
| N5—C9—C10—C11   | 0.44 (19)    | C22a—C23a—C24a—C25a | −0.0 (5)     |
| N6—C19—C18—C17  | −0.74 (18)   | C22a—C21a—C26a—C25a | 0.0 (4)      |
| N6—C15—C16—C17  | −1.1 (2)     | C21a—C26a—C25a—C24a | −0.0 (5)     |
| C13—N5—C9—C10   | −0.50 (16)   | C21a—C22a—C23a—C24a | −0.0 (4)     |
| C13—C12—C11—C10 | −0.47 (19)   | C25a—C26a—C21a—Cl1a | 178.43 (14)  |

|                 |              |                 |          |
|-----------------|--------------|-----------------|----------|
| C13—C14—N4—C20  | −157.66 (13) | C22—C21—C26—C25 | −0.0 (6) |
| C13—C14—N4—C8   | 77.10 (14)   | C22—C23—C24—C25 | −0.0 (6) |
| C19—N6—C15—C16  | 2.10 (17)    | C21—C22—C23—C24 | −0.0 (4) |
| C19—C18—C17—C16 | 1.7 (2)      | C21—C26—C25—C24 | 0.0 (6)  |
| C19—C20—N4—C8   | −77.45 (14)  | C26—C21—C22—C23 | 0.0 (6)  |
| C19—C20—N4—C14  | 157.71 (13)  | C26—C25—C24—C23 | 0.0 (4)  |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_100K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 14.2977$  (3) Å

$b = 12.4994$  (3) Å

$c = 15.3500$  (3) Å

$\beta = 110.853$  (2)°

$V = 2563.55$  (10) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.447$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 43965 reflections

$\theta = 2.1$ – $32.3^\circ$

$\mu = 0.79$  mm<sup>−1</sup>

$T = 100$  K

Block, red

$0.32 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.661$ ,  $T_{\max} = 1.000$

71141 measured reflections

6360 independent reflections

5950 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.122$

$S = 1.03$

6360 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 5.6203P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.0002$

$\Delta\rho_{\max} = 0.97$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.73$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | $x$          | $y$          | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.33711 (2)  | 0.35051 (3)  | 0.17835 (2)  | 0.01670 (9)                      |
| S2  | 0.52417 (5)  | 0.04242 (5)  | 0.31555 (4)  | 0.02621 (14)                     |
| S1  | 0.24383 (6)  | 0.32660 (7)  | 0.44461 (5)  | 0.0394 (2)                       |
| N5  | 0.22450 (14) | 0.25690 (16) | 0.10462 (12) | 0.0191 (4)                       |
| N6  | 0.42681 (14) | 0.47014 (16) | 0.23861 (12) | 0.0191 (4)                       |
| N4  | 0.24703 (14) | 0.46878 (16) | 0.11000 (13) | 0.0190 (4)                       |
| N3  | 0.38953 (14) | 0.35494 (16) | 0.07590 (13) | 0.0190 (4)                       |
| N2  | 0.42420 (15) | 0.23313 (17) | 0.24108 (13) | 0.0215 (4)                       |

|     |               |              |               |             |
|-----|---------------|--------------|---------------|-------------|
| N1  | 0.28461 (15)  | 0.35224 (17) | 0.28048 (13)  | 0.0223 (4)  |
| F1  | 0.22173 (17)  | 0.2780 (2)   | −0.36298 (15) | 0.0658 (7)  |
| C19 | 0.37964 (17)  | 0.56554 (19) | 0.22785 (15)  | 0.0197 (4)  |
| C15 | 0.52611 (17)  | 0.4661 (2)   | 0.28533 (15)  | 0.0223 (5)  |
| H7  | 0.56515 (17)  | 0.3888 (2)   | 0.29207 (15)  | 0.0267 (5)* |
| C13 | 0.13863 (17)  | 0.3122 (2)   | 0.06283 (15)  | 0.0204 (4)  |
| C18 | 0.43020 (18)  | 0.6600 (2)   | 0.26477 (16)  | 0.0232 (5)  |
| H10 | 0.39004 (18)  | 0.7371 (2)   | 0.25394 (16)  | 0.0279 (6)* |
| C14 | 0.14299 (17)  | 0.4288 (2)   | 0.08630 (16)  | 0.0222 (4)  |
| H18 | 0.11819 (17)  | 0.4412 (2)   | 0.14641 (16)  | 0.0266 (5)* |
| H17 | 0.09181 (17)  | 0.4742 (2)   | 0.02586 (16)  | 0.0266 (5)* |
| C7  | 0.34809 (17)  | 0.43022 (19) | 0.01097 (15)  | 0.0199 (4)  |
| C12 | 0.05072 (17)  | 0.2623 (2)   | 0.00630 (15)  | 0.0224 (5)  |
| H16 | −0.01788 (17) | 0.3095 (2)   | −0.02739 (15) | 0.0268 (5)* |
| C20 | 0.26858 (17)  | 0.5604 (2)   | 0.17636 (16)  | 0.0223 (5)  |
| H11 | 0.24192 (17)  | 0.6363 (2)   | 0.13770 (16)  | 0.0267 (5)* |
| H12 | 0.22921 (17)  | 0.5492 (2)   | 0.22638 (16)  | 0.0267 (5)* |
| C9  | 0.22397 (19)  | 0.15042 (19) | 0.09083 (16)  | 0.0224 (5)  |
| H13 | 0.29379 (19)  | 0.10508 (19) | 0.12432 (16)  | 0.0269 (5)* |
| C2  | 0.46576 (17)  | 0.15437 (19) | 0.27353 (15)  | 0.0200 (4)  |
| C10 | 0.13881 (19)  | 0.0955 (2)   | 0.03601 (16)  | 0.0249 (5)  |
| H14 | 0.14118 (19)  | 0.0081 (2)   | 0.02683 (16)  | 0.0299 (6)* |
| C16 | 0.58110 (18)  | 0.5570 (2)   | 0.32550 (16)  | 0.0246 (5)  |
| H8  | 0.66225 (18)  | 0.5511 (2)   | 0.36469 (16)  | 0.0296 (6)* |
| C17 | 0.53252 (19)  | 0.6549 (2)   | 0.31561 (16)  | 0.0258 (5)  |
| H9  | 0.57439 (19)  | 0.7277 (2)   | 0.34758 (16)  | 0.0310 (6)* |
| C3  | 0.46222 (18)  | 0.2923 (2)   | 0.06678 (16)  | 0.0240 (5)  |
| H1  | 0.49670 (18)  | 0.2310 (2)   | 0.12039 (16)  | 0.0288 (6)* |
| C8  | 0.26797 (18)  | 0.4982 (2)   | 0.02413 (15)  | 0.0227 (5)  |
| H5  | 0.19816 (18)  | 0.4896 (2)   | −0.03770 (15) | 0.0272 (5)* |
| H6  | 0.29127 (18)  | 0.5835 (2)   | 0.02885 (15)  | 0.0272 (5)* |
| C6  | 0.37704 (18)  | 0.4436 (2)   | −0.06599 (16) | 0.0259 (5)  |
| H4  | 0.34101 (18)  | 0.5051 (2)   | −0.11881 (16) | 0.0311 (6)* |
| C1  | 0.26732 (19)  | 0.3430 (2)   | 0.34873 (16)  | 0.0246 (5)  |
| C11 | 0.05067 (19)  | 0.1520 (2)   | −0.00708 (17) | 0.0254 (5)  |
| H15 | −0.01796 (19) | 0.1104 (2)   | −0.05099 (17) | 0.0305 (6)* |
| C4  | 0.49520 (19)  | 0.3020 (2)   | −0.00765 (17) | 0.0288 (5)  |
| H2  | 0.55504 (19)  | 0.2496 (2)   | −0.01298 (17) | 0.0345 (6)* |
| C5  | 0.4514 (2)    | 0.3790 (2)   | −0.07550 (17) | 0.0293 (5)  |
| H3  | 0.4755 (2)    | 0.3884 (2)   | −0.13578 (17) | 0.0352 (6)* |
| C25 | 0.2308 (2)    | 0.2147 (3)   | −0.1344 (2)   | 0.0354 (6)  |
| H22 | 0.2006 (2)    | 0.2444 (3)   | −0.0820 (2)   | 0.0425 (7)* |
| C26 | 0.2059 (2)    | 0.2674 (3)   | −0.2182 (2)   | 0.0393 (7)  |
| H23 | 0.1565 (2)    | 0.3381 (3)   | −0.2339 (2)   | 0.0472 (8)* |
| C24 | 0.2936 (2)    | 0.1241 (3)   | −0.1131 (3)   | 0.0453 (8)  |
| H21 | 0.3113 (2)    | 0.0819 (3)   | −0.0460 (3)   | 0.0544 (9)* |
| C21 | 0.2450 (2)    | 0.2283 (3)   | −0.2819 (2)   | 0.0415 (7)  |
| C22 | 0.3073 (3)    | 0.1406 (3)   | −0.2645 (3)   | 0.0490 (9)  |

|     |            |            |             |              |
|-----|------------|------------|-------------|--------------|
| H19 | 0.3363 (3) | 0.1115 (3) | −0.3178 (3) | 0.0588 (10)* |
| C23 | 0.3328 (3) | 0.0894 (3) | −0.1795 (3) | 0.0523 (9)   |
| H20 | 0.3846 (3) | 0.0207 (3) | −0.1640 (3) | 0.0627 (10)* |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Fe1 | 0.01753 (16) | 0.02264 (17) | 0.01050 (15) | 0.00244 (12) | 0.00569 (11) | 0.00041 (11) |
| S2  | 0.0265 (3)   | 0.0259 (3)   | 0.0284 (3)   | 0.0055 (2)   | 0.0125 (2)   | 0.0076 (2)   |
| S1  | 0.0565 (5)   | 0.0496 (4)   | 0.0208 (3)   | −0.0276 (4)  | 0.0245 (3)   | −0.0133 (3)  |
| N5  | 0.0206 (9)   | 0.0238 (10)  | 0.0126 (8)   | 0.0013 (7)   | 0.0057 (7)   | −0.0003 (7)  |
| N6  | 0.0196 (9)   | 0.0253 (10)  | 0.0127 (8)   | 0.0014 (7)   | 0.0060 (7)   | −0.0002 (7)  |
| N4  | 0.0185 (9)   | 0.0239 (10)  | 0.0143 (8)   | 0.0027 (7)   | 0.0056 (7)   | −0.0019 (7)  |
| N3  | 0.0197 (9)   | 0.0257 (10)  | 0.0125 (8)   | 0.0015 (7)   | 0.0070 (7)   | −0.0006 (7)  |
| N2  | 0.0227 (9)   | 0.0246 (10)  | 0.0163 (9)   | 0.0049 (8)   | 0.0060 (7)   | 0.0034 (7)   |
| N1  | 0.0258 (10)  | 0.0296 (10)  | 0.0138 (9)   | −0.0018 (8)  | 0.0100 (7)   | −0.0007 (7)  |
| F1  | 0.0568 (13)  | 0.101 (2)    | 0.0423 (11)  | −0.0182 (13) | 0.0206 (10)  | 0.0003 (12)  |
| C19 | 0.0203 (10)  | 0.0257 (11)  | 0.0129 (9)   | 0.0015 (9)   | 0.0055 (8)   | −0.0013 (8)  |
| C15 | 0.0202 (10)  | 0.0296 (12)  | 0.0159 (10)  | 0.0026 (9)   | 0.0050 (8)   | 0.0013 (9)   |
| C13 | 0.0197 (10)  | 0.0267 (12)  | 0.0157 (9)   | 0.0008 (9)   | 0.0073 (8)   | −0.0012 (8)  |
| C18 | 0.0258 (12)  | 0.0253 (12)  | 0.0180 (10)  | −0.0000 (9)  | 0.0070 (9)   | −0.0022 (9)  |
| C14 | 0.0176 (10)  | 0.0267 (12)  | 0.0213 (11)  | 0.0046 (9)   | 0.0059 (8)   | −0.0022 (9)  |
| C7  | 0.0203 (10)  | 0.0264 (11)  | 0.0133 (9)   | 0.0007 (8)   | 0.0065 (8)   | −0.0012 (8)  |
| C12 | 0.0201 (11)  | 0.0298 (12)  | 0.0173 (10)  | −0.0017 (9)  | 0.0068 (8)   | 0.0000 (9)   |
| C20 | 0.0204 (10)  | 0.0262 (12)  | 0.0185 (10)  | 0.0032 (9)   | 0.0049 (8)   | −0.0034 (9)  |
| C9  | 0.0268 (12)  | 0.0240 (11)  | 0.0170 (10)  | 0.0014 (9)   | 0.0084 (9)   | 0.0006 (8)   |
| C2  | 0.0198 (10)  | 0.0254 (11)  | 0.0152 (9)   | 0.0014 (9)   | 0.0067 (8)   | 0.0012 (8)   |
| C10 | 0.0315 (12)  | 0.0230 (12)  | 0.0208 (11)  | −0.0022 (10) | 0.0101 (9)   | −0.0001 (9)  |
| C16 | 0.0212 (11)  | 0.0333 (13)  | 0.0180 (10)  | −0.0013 (10) | 0.0051 (8)   | −0.0006 (9)  |
| C17 | 0.0251 (12)  | 0.0334 (13)  | 0.0184 (10)  | −0.0040 (10) | 0.0072 (9)   | −0.0049 (9)  |
| C3  | 0.0227 (11)  | 0.0341 (13)  | 0.0179 (10)  | 0.0055 (9)   | 0.0107 (9)   | −0.0006 (9)  |
| C8  | 0.0249 (11)  | 0.0282 (12)  | 0.0147 (10)  | 0.0051 (9)   | 0.0067 (8)   | 0.0045 (9)   |
| C6  | 0.0274 (12)  | 0.0376 (14)  | 0.0144 (10)  | −0.0016 (10) | 0.0094 (9)   | 0.0013 (9)   |
| C1  | 0.0294 (12)  | 0.0324 (13)  | 0.0151 (10)  | −0.0097 (10) | 0.0117 (9)   | −0.0049 (9)  |
| C11 | 0.0262 (12)  | 0.0305 (13)  | 0.0198 (11)  | −0.0057 (10) | 0.0087 (9)   | −0.0015 (9)  |
| C4  | 0.0259 (12)  | 0.0441 (15)  | 0.0209 (11)  | 0.0031 (11)  | 0.0142 (9)   | −0.0051 (10) |
| C5  | 0.0296 (12)  | 0.0447 (15)  | 0.0183 (11)  | −0.0006 (11) | 0.0143 (10)  | −0.0019 (10) |
| C25 | 0.0323 (13)  | 0.0455 (16)  | 0.0337 (14)  | −0.0087 (12) | 0.0180 (11)  | −0.0122 (12) |
| C26 | 0.0235 (12)  | 0.0553 (19)  | 0.0415 (16)  | −0.0069 (12) | 0.0144 (11)  | −0.0147 (14) |
| C24 | 0.0405 (16)  | 0.0502 (19)  | 0.0482 (18)  | −0.0063 (14) | 0.0195 (14)  | −0.0113 (15) |
| C21 | 0.0361 (15)  | 0.059 (2)    | 0.0327 (14)  | −0.0140 (14) | 0.0169 (12)  | −0.0126 (14) |
| C22 | 0.0463 (18)  | 0.055 (2)    | 0.056 (2)    | −0.0064 (15) | 0.0299 (16)  | −0.0273 (17) |
| C23 | 0.0497 (19)  | 0.0468 (19)  | 0.068 (2)    | 0.0001 (15)  | 0.0303 (18)  | −0.0102 (17) |

*Geometric parameters (Å, °)*

|        |           |         |           |
|--------|-----------|---------|-----------|
| Fe1—N5 | 1.986 (2) | C12—H16 | 1.1030    |
| Fe1—N6 | 1.973 (2) | C12—C11 | 1.393 (4) |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| Fe1—N4     | 1.996 (2)   | C20—H11     | 1.1110      |
| Fe1—N3     | 1.9691 (19) | C20—H12     | 1.1110      |
| Fe1—N2     | 1.943 (2)   | C9—H13      | 1.1030      |
| Fe1—N1     | 1.965 (2)   | C9—C10      | 1.390 (3)   |
| S2—C2      | 1.640 (2)   | C10—H14     | 1.1030      |
| S1—C1      | 1.635 (2)   | C10—C11     | 1.390 (4)   |
| N5—C13     | 1.355 (3)   | C16—H8      | 1.1030      |
| N5—C9      | 1.347 (3)   | C16—C17     | 1.389 (4)   |
| N6—C19     | 1.351 (3)   | C17—H9      | 1.1030      |
| N6—C15     | 1.344 (3)   | C3—H1       | 1.1030      |
| N4—C14     | 1.486 (3)   | C3—C4       | 1.388 (3)   |
| N4—C20     | 1.491 (3)   | C8—H5       | 1.1110      |
| N4—C8      | 1.498 (3)   | C8—H6       | 1.1110      |
| N3—C7      | 1.345 (3)   | C6—H4       | 1.1030      |
| N3—C3      | 1.348 (3)   | C6—C5       | 1.383 (4)   |
| N2—C2      | 1.166 (3)   | C11—H15     | 1.1030      |
| N1—C1      | 1.165 (3)   | C4—H2       | 1.1030      |
| F1—C21     | 1.323 (4)   | C4—C5       | 1.393 (4)   |
| C19—C18    | 1.395 (3)   | C5—H3       | 1.1030      |
| C19—C20    | 1.502 (3)   | C25—H22     | 1.1030      |
| C15—H7     | 1.1030      | C25—C26     | 1.375 (5)   |
| C15—C16    | 1.393 (3)   | C25—C24     | 1.410 (5)   |
| C13—C14    | 1.498 (3)   | C26—H23     | 1.1030      |
| C13—C12    | 1.395 (3)   | C26—C21     | 1.377 (4)   |
| C18—H10    | 1.1030      | C24—H21     | 1.1030      |
| C18—C17    | 1.393 (3)   | C24—C23     | 1.397 (5)   |
| C14—H18    | 1.1110      | C21—C22     | 1.377 (5)   |
| C14—H17    | 1.1110      | C22—H19     | 1.1030      |
| C7—C8      | 1.497 (3)   | C22—C23     | 1.379 (6)   |
| C7—C6      | 1.394 (3)   | C23—H20     | 1.1030      |
|            |             |             |             |
| N6—Fe1—N5  | 166.79 (8)  | H11—C20—C19 | 110.03 (13) |
| N4—Fe1—N5  | 84.05 (8)   | H12—C20—N4  | 110.03 (12) |
| N4—Fe1—N6  | 82.83 (8)   | H12—C20—C19 | 110.03 (12) |
| N3—Fe1—N5  | 91.34 (8)   | H12—C20—H11 | 108.4       |
| N3—Fe1—N6  | 89.11 (8)   | H13—C9—N5   | 118.73 (14) |
| N3—Fe1—N4  | 85.25 (8)   | C10—C9—N5   | 122.5 (2)   |
| N2—Fe1—N5  | 94.82 (8)   | C10—C9—H13  | 118.73 (15) |
| N2—Fe1—N6  | 98.34 (8)   | N2—C2—S2    | 178.0 (2)   |
| N2—Fe1—N4  | 178.16 (8)  | H14—C10—C9  | 120.41 (15) |
| N2—Fe1—N3  | 93.33 (8)   | C11—C10—C9  | 119.2 (2)   |
| N1—Fe1—N5  | 89.92 (8)   | C11—C10—H14 | 120.41 (15) |
| N1—Fe1—N6  | 89.24 (8)   | H8—C16—C15  | 120.39 (14) |
| N1—Fe1—N4  | 93.04 (8)   | C17—C16—C15 | 119.2 (2)   |
| N1—Fe1—N3  | 177.76 (9)  | C17—C16—H8  | 120.39 (14) |
| N1—Fe1—N2  | 88.41 (8)   | C16—C17—C18 | 119.1 (2)   |
| C13—N5—Fe1 | 112.56 (16) | H9—C17—C18  | 120.46 (15) |
| C9—N5—Fe1  | 129.02 (17) | H9—C17—C16  | 120.46 (14) |

|                |              |                 |             |
|----------------|--------------|-----------------|-------------|
| C9—N5—C13      | 118.4 (2)    | H1—C3—N3        | 118.83 (13) |
| C19—N6—Fe1     | 113.50 (15)  | C4—C3—N3        | 122.3 (2)   |
| C15—N6—Fe1     | 127.50 (17)  | C4—C3—H1        | 118.83 (16) |
| C15—N6—C19     | 119.0 (2)    | C7—C8—N4        | 112.42 (18) |
| C14—N4—Fe1     | 106.39 (14)  | H5—C8—N4        | 109.12 (12) |
| C20—N4—Fe1     | 105.81 (13)  | H5—C8—C7        | 109.12 (12) |
| C20—N4—C14     | 111.78 (17)  | H6—C8—N4        | 109.12 (12) |
| C8—N4—Fe1      | 110.54 (14)  | H6—C8—C7        | 109.12 (13) |
| C8—N4—C14      | 111.30 (17)  | H6—C8—H5        | 107.9       |
| C8—N4—C20      | 110.79 (19)  | H4—C6—C7        | 120.31 (15) |
| C7—N3—Fe1      | 114.76 (15)  | C5—C6—C7        | 119.4 (2)   |
| C3—N3—Fe1      | 126.39 (16)  | C5—C6—H4        | 120.31 (15) |
| C3—N3—C7       | 118.8 (2)    | N1—C1—S1        | 178.5 (2)   |
| C2—N2—Fe1      | 171.22 (19)  | C10—C11—C12     | 118.7 (2)   |
| C1—N1—Fe1      | 168.7 (2)    | H15—C11—C12     | 120.66 (15) |
| C18—C19—N6     | 122.2 (2)    | H15—C11—C10     | 120.66 (15) |
| C20—C19—N6     | 114.5 (2)    | H2—C4—C3        | 120.60 (16) |
| C20—C19—C18    | 123.2 (2)    | C5—C4—C3        | 118.8 (2)   |
| H7—C15—N6      | 119.06 (14)  | C5—C4—H2        | 120.60 (14) |
| C16—C15—N6     | 121.9 (2)    | C4—C5—C6        | 118.9 (2)   |
| C16—C15—H7     | 119.06 (14)  | H3—C5—C6        | 120.57 (15) |
| C14—C13—N5     | 115.4 (2)    | H3—C5—C4        | 120.57 (14) |
| C12—C13—N5     | 122.1 (2)    | C26—C25—H22     | 118.90 (18) |
| C12—C13—C14    | 122.4 (2)    | C24—C25—H22     | 118.90 (19) |
| H10—C18—C19    | 120.73 (14)  | C24—C25—C26     | 122.2 (3)   |
| C17—C18—C19    | 118.5 (2)    | H23—C26—C25     | 121.33 (18) |
| C17—C18—H10    | 120.73 (15)  | C21—C26—C25     | 117.3 (3)   |
| C13—C14—N4     | 109.78 (18)  | C21—C26—H23     | 121.3 (2)   |
| H18—C14—N4     | 109.71 (11)  | H21—C24—C25     | 121.04 (19) |
| H18—C14—C13    | 109.71 (12)  | C23—C24—C25     | 117.9 (3)   |
| H17—C14—N4     | 109.71 (12)  | C23—C24—H21     | 121.0 (2)   |
| H17—C14—C13    | 109.71 (12)  | C26—C21—F1      | 118.1 (3)   |
| H17—C14—H18    | 108.2        | C22—C21—F1      | 119.1 (3)   |
| C8—C7—N3       | 116.99 (19)  | C22—C21—C26     | 122.8 (3)   |
| C6—C7—N3       | 121.8 (2)    | H19—C22—C21     | 120.4 (2)   |
| C6—C7—C8       | 121.2 (2)    | C23—C22—C21     | 119.2 (3)   |
| H16—C12—C13    | 120.42 (14)  | C23—C22—H19     | 120.4 (2)   |
| C11—C12—C13    | 119.2 (2)    | C22—C23—C24     | 120.4 (4)   |
| C11—C12—H16    | 120.42 (15)  | H20—C23—C24     | 119.8 (2)   |
| C19—C20—N4     | 108.35 (18)  | H20—C23—C22     | 119.8 (2)   |
| H11—C20—N4     | 110.03 (12)  |                 |             |
| Fe1—N5—C13—C14 | −4.83 (16)   | C19—C20—N4—C14  | 155.89 (18) |
| Fe1—N5—C13—C12 | 178.85 (14)  | C19—C20—N4—C8   | −79.3 (2)   |
| Fe1—N5—C9—C10  | −179.22 (19) | C15—N6—C19—C18  | −0.9 (2)    |
| Fe1—N6—C19—C18 | −179.55 (15) | C15—N6—C19—C20  | −178.8 (2)  |
| Fe1—N6—C19—C20 | 2.62 (16)    | C15—C16—C17—C18 | −0.8 (3)    |
| Fe1—N6—C15—C16 | −179.52 (18) | C13—N5—C9—C10   | −0.4 (2)    |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Fe1—N4—C14—C13  | −36.44 (14)  | C13—C14—N4—C20  | −151.48 (18) |
| Fe1—N4—C20—C19  | 40.49 (14)   | C13—C14—N4—C8   | 84.0 (2)     |
| Fe1—N4—C8—C7    | 1.46 (15)    | C13—C12—C11—C10 | −0.5 (3)     |
| Fe1—N3—C7—C8    | −0.93 (18)   | C14—N4—C8—C7    | −116.54 (19) |
| Fe1—N3—C7—C6    | −179.97 (16) | C14—C13—N5—C9   | 176.15 (19)  |
| Fe1—N3—C3—C4    | −179.3 (2)   | C14—C13—C12—C11 | −175.5 (2)   |
| N5—C13—C14—N4   | 28.2 (2)     | C7—N3—C3—C4     | −0.6 (3)     |
| N5—C13—C12—C11  | 0.6 (3)      | C7—C8—N4—C20    | 118.4 (2)    |
| N5—C9—C10—C11   | 0.5 (3)      | C7—C6—C5—C4     | 0.1 (3)      |
| N6—C19—C18—C17  | −1.0 (3)     | C12—C13—N5—C9   | −0.2 (3)     |
| N6—C19—C20—N4   | −29.3 (2)    | C12—C11—C10—C9  | −0.0 (3)     |
| N6—C15—C16—C17  | −1.2 (3)     | C20—C19—C18—C17 | 176.6 (2)    |
| N4—C14—C13—C12  | −155.51 (17) | C3—N3—C7—C8     | −179.8 (2)   |
| N4—C20—C19—C18  | 152.89 (18)  | C3—N3—C7—C6     | 1.2 (3)      |
| N4—C8—C7—N3     | −0.4 (2)     | C3—C4—C5—C6     | 0.4 (3)      |
| N4—C8—C7—C6     | 178.66 (19)  | C8—C7—C6—C5     | −180.0 (2)   |
| N3—C7—C6—C5     | −1.0 (3)     | C25—C26—C21—C22 | −0.3 (3)     |
| N3—C3—C4—C5     | −0.2 (3)     | C25—C24—C23—C22 | −2.4 (4)     |
| F1—C21—C26—C25  | 180.0 (3)    | C26—C25—C24—C23 | 1.5 (3)      |
| F1—C21—C22—C23  | 179.1 (3)    | C26—C21—C22—C23 | −0.6 (4)     |
| C19—N6—C15—C16  | 2.1 (2)      | C24—C25—C26—C21 | −0.1 (3)     |
| C19—C18—C17—C16 | 1.9 (3)      | C24—C23—C22—C21 | 2.0 (4)      |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_120K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 14.3180$  (3) Å

$b = 12.5119$  (3) Å

$c = 15.3591$  (3) Å

$\beta = 110.824$  (2)°

$V = 2571.77$  (10) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.442$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 43288 reflections

$\theta = 2.1$ – $32.4$ °

$\mu = 0.78$  mm<sup>−1</sup>

$T = 120$  K

Block, red

$0.32 \times 0.2 \times 0.12$  mm

## Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.660$ ,  $T_{\max} = 1.000$

72319 measured reflections

6382 independent reflections

5941 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 28.3$ °,  $\theta_{\min} = 2.2$ °

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.130$

$S = 0.96$

6382 reflections



325 parameters  
 0 restraints  
 43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 5.3461P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.0004$   
 $\Delta\rho_{\max} = 0.92 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>     | <i>z</i>      | <i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub> |
|-----|---------------|--------------|---------------|---------------------------------------------------------------|
| Fe1 | 0.33700 (2)   | 0.35020 (3)  | 0.17853 (2)   | 0.01873 (10)                                                  |
| S2  | 0.52429 (5)   | 0.04299 (5)  | 0.31600 (5)   | 0.02953 (15)                                                  |
| S1  | 0.24437 (7)   | 0.32609 (7)  | 0.44486 (5)   | 0.0439 (2)                                                    |
| N5  | 0.22449 (14)  | 0.25674 (16) | 0.10491 (12)  | 0.0212 (4)                                                    |
| N6  | 0.42651 (14)  | 0.46980 (16) | 0.23866 (12)  | 0.0213 (4)                                                    |
| N4  | 0.24707 (14)  | 0.46826 (16) | 0.11027 (13)  | 0.0211 (4)                                                    |
| N3  | 0.38920 (14)  | 0.35467 (16) | 0.07609 (13)  | 0.0215 (4)                                                    |
| N2  | 0.42402 (15)  | 0.23314 (17) | 0.24117 (13)  | 0.0241 (4)                                                    |
| N1  | 0.28470 (15)  | 0.35186 (16) | 0.28063 (13)  | 0.0245 (4)                                                    |
| F1  | 0.22214 (18)  | 0.2772 (3)   | −0.36262 (16) | 0.0769 (8)                                                    |
| C19 | 0.37940 (17)  | 0.56496 (19) | 0.22775 (14)  | 0.0218 (4)                                                    |
| C13 | 0.13875 (17)  | 0.31173 (19) | 0.06322 (15)  | 0.0221 (4)                                                    |
| C15 | 0.52554 (17)  | 0.4661 (2)   | 0.28525 (15)  | 0.0250 (5)                                                    |
| H7  | 0.56450 (17)  | 0.3888 (2)   | 0.29216 (15)  | 0.0300 (6)*                                                   |
| C7  | 0.34788 (17)  | 0.43006 (19) | 0.01125 (14)  | 0.0220 (4)                                                    |
| C18 | 0.42996 (19)  | 0.6595 (2)   | 0.26436 (16)  | 0.0260 (5)                                                    |
| H10 | 0.38987 (19)  | 0.7365 (2)   | 0.25330 (16)  | 0.0312 (6)*                                                   |
| C14 | 0.14309 (16)  | 0.4284 (2)   | 0.08669 (16)  | 0.0245 (4)                                                    |
| H18 | 0.11839 (16)  | 0.4409 (2)   | 0.14682 (16)  | 0.0293 (5)*                                                   |
| H17 | 0.09198 (16)  | 0.4738 (2)   | 0.02632 (16)  | 0.0293 (5)*                                                   |
| C20 | 0.26859 (17)  | 0.5599 (2)   | 0.17656 (16)  | 0.0253 (5)                                                    |
| H11 | 0.24191 (17)  | 0.6357 (2)   | 0.13793 (16)  | 0.0303 (6)*                                                   |
| H12 | 0.22938 (17)  | 0.5487 (2)   | 0.22667 (16)  | 0.0303 (6)*                                                   |
| C2  | 0.46579 (17)  | 0.15464 (19) | 0.27380 (15)  | 0.0222 (4)                                                    |
| C9  | 0.22409 (19)  | 0.15045 (19) | 0.09106 (16)  | 0.0248 (5)                                                    |
| H13 | 0.29385 (19)  | 0.10519 (19) | 0.12436 (16)  | 0.0297 (6)*                                                   |
| C10 | 0.13878 (19)  | 0.0955 (2)   | 0.03619 (16)  | 0.0280 (5)                                                    |
| H14 | 0.14104 (19)  | 0.0083 (2)   | 0.02691 (16)  | 0.0336 (6)*                                                   |
| C12 | 0.05111 (17)  | 0.2619 (2)   | 0.00680 (15)  | 0.0252 (5)                                                    |
| H16 | −0.01742 (17) | 0.3090 (2)   | −0.02687 (15) | 0.0302 (6)*                                                   |
| C3  | 0.46204 (18)  | 0.2923 (2)   | 0.06696 (16)  | 0.0268 (5)                                                    |
| H1  | 0.49666 (18)  | 0.2312 (2)   | 0.12058 (16)  | 0.0322 (6)*                                                   |
| C6  | 0.37668 (19)  | 0.4435 (2)   | −0.06563 (16) | 0.0290 (5)                                                    |
| H4  | 0.34051 (19)  | 0.5048 (2)   | −0.11850 (16) | 0.0348 (6)*                                                   |
| C17 | 0.53215 (19)  | 0.6545 (2)   | 0.31516 (17)  | 0.0289 (5)                                                    |
| H9  | 0.57395 (19)  | 0.7272 (2)   | 0.34703 (17)  | 0.0347 (6)*                                                   |
| C8  | 0.26772 (18)  | 0.4976 (2)   | 0.02438 (15)  | 0.0253 (5)                                                    |
| H5  | 0.19800 (18)  | 0.4886 (2)   | −0.03730 (15) | 0.0304 (6)*                                                   |
| H6  | 0.29062 (18)  | 0.5829 (2)   | 0.02886 (15)  | 0.0304 (6)*                                                   |
| C16 | 0.58057 (18)  | 0.5566 (2)   | 0.32506 (16)  | 0.0282 (5)                                                    |

|     |               |            |               |              |
|-----|---------------|------------|---------------|--------------|
| H8  | 0.66163 (18)  | 0.5507 (2) | 0.36399 (16)  | 0.0339 (6)*  |
| C11 | 0.05125 (19)  | 0.1519 (2) | −0.00646 (17) | 0.0284 (5)   |
| H15 | −0.01728 (19) | 0.1103 (2) | −0.05026 (17) | 0.0341 (6)*  |
| C1  | 0.26747 (19)  | 0.3425 (2) | 0.34893 (17)  | 0.0274 (5)   |
| C4  | 0.4950 (2)    | 0.3020 (2) | −0.00730 (18) | 0.0324 (6)   |
| H2  | 0.5546 (2)    | 0.2497 (2) | −0.01274 (18) | 0.0389 (7)*  |
| C5  | 0.4512 (2)    | 0.3792 (2) | −0.07497 (17) | 0.0332 (6)   |
| H3  | 0.4756 (2)    | 0.3888 (2) | −0.13505 (17) | 0.0398 (7)*  |
| C25 | 0.2308 (2)    | 0.2144 (3) | −0.1344 (2)   | 0.0404 (7)   |
| H22 | 0.2005 (2)    | 0.2438 (3) | −0.0822 (2)   | 0.0485 (8)*  |
| C26 | 0.2059 (2)    | 0.2671 (3) | −0.2184 (2)   | 0.0437 (7)   |
| H23 | 0.1566 (2)    | 0.3376 (3) | −0.2341 (2)   | 0.0525 (9)*  |
| C24 | 0.2936 (3)    | 0.1240 (3) | −0.1132 (3)   | 0.0517 (8)   |
| H21 | 0.3114 (3)    | 0.0818 (3) | −0.0461 (3)   | 0.0620 (10)* |
| C21 | 0.2453 (2)    | 0.2280 (3) | −0.2817 (2)   | 0.0474 (8)   |
| C22 | 0.3074 (3)    | 0.1408 (3) | −0.2645 (3)   | 0.0558 (10)  |
| H19 | 0.3366 (3)    | 0.1120 (3) | −0.3177 (3)   | 0.0670 (12)* |
| C23 | 0.3326 (3)    | 0.0897 (3) | −0.1798 (3)   | 0.0604 (10)  |
| H20 | 0.3841 (3)    | 0.0209 (3) | −0.1644 (3)   | 0.0725 (12)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Fe1 | 0.01977 (16) | 0.02475 (18) | 0.01233 (15) | 0.00299 (11) | 0.00651 (11) | 0.00054 (11) |
| S2  | 0.0297 (3)   | 0.0288 (3)   | 0.0325 (3)   | 0.0062 (2)   | 0.0139 (2)   | 0.0086 (2)   |
| S1  | 0.0640 (5)   | 0.0541 (4)   | 0.0237 (3)   | −0.0300 (4)  | 0.0279 (3)   | −0.0144 (3)  |
| N5  | 0.0234 (9)   | 0.0255 (9)   | 0.0152 (8)   | 0.0017 (7)   | 0.0075 (7)   | −0.0004 (7)  |
| N6  | 0.0222 (9)   | 0.0277 (10)  | 0.0141 (8)   | 0.0013 (7)   | 0.0065 (7)   | 0.0002 (7)   |
| N4  | 0.0207 (9)   | 0.0259 (10)  | 0.0165 (8)   | 0.0031 (7)   | 0.0065 (7)   | −0.0022 (7)  |
| N3  | 0.0227 (9)   | 0.0282 (10)  | 0.0146 (8)   | 0.0016 (7)   | 0.0081 (7)   | −0.0001 (7)  |
| N2  | 0.0242 (9)   | 0.0288 (10)  | 0.0181 (8)   | 0.0060 (8)   | 0.0061 (7)   | 0.0039 (7)   |
| N1  | 0.0283 (10)  | 0.0317 (10)  | 0.0159 (9)   | −0.0019 (8)  | 0.0109 (7)   | −0.0015 (7)  |
| F1  | 0.0655 (15)  | 0.119 (2)    | 0.0487 (12)  | −0.0218 (15) | 0.0237 (11)  | 0.0003 (13)  |
| C19 | 0.0228 (10)  | 0.0271 (11)  | 0.0151 (9)   | 0.0019 (8)   | 0.0061 (8)   | −0.0008 (8)  |
| C13 | 0.0213 (10)  | 0.0278 (11)  | 0.0181 (9)   | 0.0009 (9)   | 0.0080 (8)   | −0.0010 (8)  |
| C15 | 0.0221 (10)  | 0.0331 (12)  | 0.0183 (10)  | 0.0032 (9)   | 0.0053 (8)   | 0.0014 (9)   |
| C7  | 0.0235 (10)  | 0.0282 (11)  | 0.0144 (9)   | 0.0006 (8)   | 0.0070 (8)   | −0.0004 (8)  |
| C18 | 0.0290 (12)  | 0.0280 (12)  | 0.0205 (10)  | −0.0000 (9)  | 0.0083 (9)   | −0.0020 (9)  |
| C14 | 0.0200 (10)  | 0.0283 (12)  | 0.0244 (11)  | 0.0046 (8)   | 0.0072 (8)   | −0.0026 (9)  |
| C20 | 0.0241 (11)  | 0.0288 (12)  | 0.0212 (10)  | 0.0039 (9)   | 0.0060 (8)   | −0.0039 (9)  |
| C2  | 0.0218 (10)  | 0.0282 (12)  | 0.0168 (9)   | 0.0016 (8)   | 0.0074 (8)   | 0.0014 (8)   |
| C9  | 0.0307 (12)  | 0.0251 (11)  | 0.0191 (10)  | 0.0022 (9)   | 0.0097 (9)   | 0.0008 (8)   |
| C10 | 0.0358 (13)  | 0.0255 (12)  | 0.0232 (11)  | −0.0027 (10) | 0.0111 (10)  | −0.0002 (9)  |
| C12 | 0.0229 (11)  | 0.0329 (13)  | 0.0197 (10)  | −0.0018 (9)  | 0.0075 (8)   | 0.0001 (9)   |
| C3  | 0.0249 (11)  | 0.0376 (13)  | 0.0211 (10)  | 0.0049 (10)  | 0.0119 (9)   | −0.0007 (9)  |
| C6  | 0.0317 (12)  | 0.0405 (14)  | 0.0167 (10)  | −0.0011 (10) | 0.0108 (9)   | 0.0018 (9)   |
| C17 | 0.0276 (12)  | 0.0359 (14)  | 0.0220 (11)  | −0.0037 (10) | 0.0074 (9)   | −0.0046 (9)  |
| C8  | 0.0278 (11)  | 0.0310 (12)  | 0.0167 (10)  | 0.0058 (9)   | 0.0072 (8)   | 0.0047 (9)   |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C16 | 0.0241 (11) | 0.0370 (13) | 0.0212 (10) | −0.0015 (10) | 0.0052 (9)  | −0.0010 (9)  |
| C11 | 0.0300 (12) | 0.0329 (13) | 0.0229 (11) | −0.0064 (10) | 0.0101 (9)  | −0.0024 (9)  |
| C1  | 0.0325 (12) | 0.0355 (13) | 0.0176 (10) | −0.0105 (10) | 0.0129 (9)  | −0.0053 (9)  |
| C4  | 0.0298 (12) | 0.0497 (16) | 0.0234 (11) | 0.0038 (11)  | 0.0164 (10) | −0.0052 (11) |
| C5  | 0.0343 (13) | 0.0501 (16) | 0.0209 (11) | −0.0013 (12) | 0.0169 (10) | −0.0023 (11) |
| C25 | 0.0364 (14) | 0.0520 (18) | 0.0383 (15) | −0.0104 (13) | 0.0200 (12) | −0.0151 (13) |
| C26 | 0.0264 (13) | 0.060 (2)   | 0.0471 (17) | −0.0076 (13) | 0.0161 (12) | −0.0165 (15) |
| C24 | 0.0460 (17) | 0.056 (2)   | 0.057 (2)   | −0.0083 (15) | 0.0233 (16) | −0.0136 (17) |
| C21 | 0.0397 (16) | 0.071 (2)   | 0.0364 (15) | −0.0176 (15) | 0.0193 (13) | −0.0150 (15) |
| C22 | 0.053 (2)   | 0.063 (2)   | 0.063 (2)   | −0.0087 (17) | 0.0345 (18) | −0.0322 (19) |
| C23 | 0.057 (2)   | 0.051 (2)   | 0.083 (3)   | −0.0011 (17) | 0.036 (2)   | −0.013 (2)   |

*Geometric parameters (Å, °)*

|           |             |             |           |
|-----------|-------------|-------------|-----------|
| Fe1—N5    | 1.986 (2)   | C20—H11     | 1.1110    |
| Fe1—N6    | 1.973 (2)   | C20—H12     | 1.1110    |
| Fe1—N4    | 1.9949 (19) | C9—H13      | 1.1030    |
| Fe1—N3    | 1.9681 (19) | C9—C10      | 1.394 (3) |
| Fe1—N2    | 1.942 (2)   | C10—H14     | 1.1030    |
| Fe1—N1    | 1.963 (2)   | C10—C11     | 1.383 (4) |
| S2—C2     | 1.639 (2)   | C12—H16     | 1.1030    |
| S1—C1     | 1.634 (2)   | C12—C11     | 1.392 (4) |
| N5—C13    | 1.353 (3)   | C3—H1       | 1.1030    |
| N5—C9     | 1.346 (3)   | C3—C4       | 1.386 (3) |
| N6—C19    | 1.349 (3)   | C6—H4       | 1.1030    |
| N6—C15    | 1.343 (3)   | C6—C5       | 1.385 (4) |
| N4—C14    | 1.487 (3)   | C17—H9      | 1.1030    |
| N4—C20    | 1.491 (3)   | C17—C16     | 1.389 (4) |
| N4—C8     | 1.497 (3)   | C8—H5       | 1.1110    |
| N3—C7     | 1.346 (3)   | C8—H6       | 1.1110    |
| N3—C3     | 1.350 (3)   | C16—H8      | 1.1030    |
| N2—C2     | 1.166 (3)   | C11—H15     | 1.1030    |
| N1—C1     | 1.166 (3)   | C4—H2       | 1.1030    |
| F1—C21    | 1.319 (4)   | C4—C5       | 1.393 (4) |
| C19—C18   | 1.396 (3)   | C5—H3       | 1.1030    |
| C19—C20   | 1.501 (3)   | C25—H22     | 1.1030    |
| C13—C14   | 1.500 (3)   | C25—C26     | 1.377 (5) |
| C13—C12   | 1.393 (3)   | C25—C24     | 1.409 (5) |
| C15—H7    | 1.1030      | C26—H23     | 1.1030    |
| C15—C16   | 1.390 (3)   | C26—C21     | 1.376 (4) |
| C7—C6     | 1.392 (3)   | C24—H21     | 1.1030    |
| C7—C8     | 1.496 (3)   | C24—C23     | 1.395 (5) |
| C18—H10   | 1.1030      | C21—C22     | 1.372 (5) |
| C18—C17   | 1.393 (3)   | C22—H19     | 1.1030    |
| C14—H18   | 1.1110      | C22—C23     | 1.378 (6) |
| C14—H17   | 1.1110      | C23—H20     | 1.1030    |
| N6—Fe1—N5 | 166.71 (8)  | H12—C20—H11 | 108.4     |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N4—Fe1—N5   | 83.99 (8)   | N2—C2—S2    | 178.0 (2)   |
| N4—Fe1—N6   | 82.81 (8)   | H13—C9—N5   | 118.79 (14) |
| N3—Fe1—N5   | 91.34 (8)   | C10—C9—N5   | 122.4 (2)   |
| N3—Fe1—N6   | 89.11 (8)   | C10—C9—H13  | 118.79 (15) |
| N3—Fe1—N4   | 85.20 (8)   | H14—C10—C9  | 120.44 (15) |
| N2—Fe1—N5   | 94.92 (8)   | C11—C10—C9  | 119.1 (2)   |
| N2—Fe1—N6   | 98.31 (8)   | C11—C10—H14 | 120.44 (15) |
| N2—Fe1—N4   | 178.16 (8)  | H16—C12—C13 | 120.47 (14) |
| N2—Fe1—N3   | 93.35 (8)   | C11—C12—C13 | 119.1 (2)   |
| N1—Fe1—N5   | 89.91 (8)   | C11—C12—H16 | 120.47 (15) |
| N1—Fe1—N6   | 89.25 (8)   | H1—C3—N3    | 118.79 (13) |
| N1—Fe1—N4   | 93.08 (8)   | C4—C3—N3    | 122.4 (2)   |
| N1—Fe1—N3   | 177.76 (9)  | C4—C3—H1    | 118.79 (16) |
| N1—Fe1—N2   | 88.40 (8)   | H4—C6—C7    | 120.36 (15) |
| C13—N5—Fe1  | 112.69 (16) | C5—C6—C7    | 119.3 (2)   |
| C9—N5—Fe1   | 128.93 (17) | C5—C6—H4    | 120.36 (15) |
| C9—N5—C13   | 118.4 (2)   | H9—C17—C18  | 120.51 (15) |
| C19—N6—Fe1  | 113.47 (15) | C16—C17—C18 | 119.0 (2)   |
| C15—N6—Fe1  | 127.62 (17) | C16—C17—H9  | 120.51 (14) |
| C15—N6—C19  | 118.9 (2)   | C7—C8—N4    | 112.44 (18) |
| C14—N4—Fe1  | 106.45 (14) | H5—C8—N4    | 109.11 (12) |
| C20—N4—Fe1  | 105.82 (13) | H5—C8—C7    | 109.11 (12) |
| C20—N4—C14  | 111.71 (17) | H6—C8—N4    | 109.11 (12) |
| C8—N4—Fe1   | 110.61 (14) | H6—C8—C7    | 109.11 (13) |
| C8—N4—C14   | 111.21 (17) | H6—C8—H5    | 107.8       |
| C8—N4—C20   | 110.82 (19) | C17—C16—C15 | 119.1 (2)   |
| C7—N3—Fe1   | 114.80 (15) | H8—C16—C15  | 120.43 (14) |
| C3—N3—Fe1   | 126.42 (16) | H8—C16—C17  | 120.43 (14) |
| C3—N3—C7    | 118.8 (2)   | C12—C11—C10 | 118.9 (2)   |
| C2—N2—Fe1   | 171.4 (2)   | H15—C11—C10 | 120.56 (15) |
| C1—N1—Fe1   | 168.6 (2)   | H15—C11—C12 | 120.56 (15) |
| C18—C19—N6  | 122.2 (2)   | N1—C1—S1    | 178.5 (2)   |
| C20—C19—N6  | 114.6 (2)   | H2—C4—C3    | 120.63 (16) |
| C20—C19—C18 | 123.1 (2)   | C5—C4—C3    | 118.7 (2)   |
| C14—C13—N5  | 115.3 (2)   | C5—C4—H2    | 120.63 (15) |
| C12—C13—N5  | 122.1 (2)   | C4—C5—C6    | 119.0 (2)   |
| C12—C13—C14 | 122.4 (2)   | H3—C5—C6    | 120.52 (15) |
| H7—C15—N6   | 118.92 (14) | H3—C5—C4    | 120.52 (15) |
| C16—C15—N6  | 122.2 (2)   | C26—C25—H22 | 118.91 (19) |
| C16—C15—H7  | 118.92 (14) | C24—C25—H22 | 118.9 (2)   |
| C6—C7—N3    | 121.8 (2)   | C24—C25—C26 | 122.2 (3)   |
| C8—C7—N3    | 116.91 (19) | H23—C26—C25 | 121.39 (19) |
| C8—C7—C6    | 121.2 (2)   | C21—C26—C25 | 117.2 (3)   |
| H10—C18—C19 | 120.71 (14) | C21—C26—H23 | 121.4 (2)   |
| C17—C18—C19 | 118.6 (2)   | H21—C24—C25 | 121.1 (2)   |
| C17—C18—H10 | 120.71 (15) | C23—C24—C25 | 117.8 (4)   |
| C13—C14—N4  | 109.70 (18) | C23—C24—H21 | 121.1 (3)   |
| H18—C14—N4  | 109.73 (11) | C26—C21—F1  | 118.1 (4)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| H18—C14—C13     | 109.73 (12)  | C22—C21—F1      | 118.8 (3)    |
| H17—C14—N4      | 109.73 (12)  | C22—C21—C26     | 123.1 (3)    |
| H17—C14—C13     | 109.73 (12)  | H19—C22—C21     | 120.5 (2)    |
| H17—C14—H18     | 108.2        | C23—C22—C21     | 119.0 (3)    |
| C19—C20—N4      | 108.25 (18)  | C23—C22—H19     | 120.5 (2)    |
| H11—C20—N4      | 110.05 (12)  | C22—C23—C24     | 120.7 (4)    |
| H11—C20—C19     | 110.05 (13)  | H20—C23—C24     | 119.6 (3)    |
| H12—C20—N4      | 110.05 (12)  | H20—C23—C22     | 119.6 (2)    |
| H12—C20—C19     | 110.05 (12)  |                 |              |
| Fe1—N5—C13—C14  | −4.81 (16)   | C19—C20—N4—C14  | 155.98 (18)  |
| Fe1—N5—C13—C12  | 178.79 (14)  | C19—C20—N4—C8   | −79.4 (2)    |
| Fe1—N5—C9—C10   | −179.22 (19) | C13—N5—C9—C10   | −0.5 (2)     |
| Fe1—N6—C19—C18  | −179.35 (15) | C13—C14—N4—C20  | −151.53 (18) |
| Fe1—N6—C19—C20  | 2.81 (16)    | C13—C14—N4—C8   | 84.1 (2)     |
| Fe1—N6—C15—C16  | −179.74 (19) | C13—C12—C11—C10 | −0.6 (3)     |
| Fe1—N4—C14—C13  | −36.48 (14)  | C15—N6—C19—C18  | −0.9 (2)     |
| Fe1—N4—C20—C19  | 40.54 (14)   | C15—N6—C19—C20  | −178.7 (2)   |
| Fe1—N4—C8—C7    | 1.20 (15)    | C15—C16—C17—C18 | −1.0 (3)     |
| Fe1—N3—C7—C6    | −180.00 (16) | C7—N3—C3—C4     | −0.8 (3)     |
| Fe1—N3—C7—C8    | −1.24 (18)   | C7—C6—C5—C4     | 0.4 (3)      |
| Fe1—N3—C3—C4    | −179.2 (2)   | C7—C8—N4—C14    | −116.9 (2)   |
| N5—C13—C14—N4   | 28.2 (2)     | C7—C8—N4—C20    | 118.2 (2)    |
| N5—C13—C12—C11  | 0.7 (3)      | C14—C13—N5—C9   | 176.26 (19)  |
| N5—C9—C10—C11   | 0.6 (3)      | C14—C13—C12—C11 | −175.5 (2)   |
| N6—C19—C18—C17  | −1.1 (3)     | C20—C19—C18—C17 | 176.5 (2)    |
| N6—C19—C20—N4   | −29.5 (2)    | C9—N5—C13—C12   | −0.1 (2)     |
| N6—C15—C16—C17  | −1.1 (3)     | C9—C10—C11—C12  | −0.0 (3)     |
| N4—C14—C13—C12  | −155.45 (17) | C3—N3—C7—C6     | 1.5 (3)      |
| N4—C20—C19—C18  | 152.74 (18)  | C3—N3—C7—C8     | −179.8 (2)   |
| N4—C8—C7—N3     | −0.0 (2)     | C3—C4—C5—C6     | 0.2 (3)      |
| N4—C8—C7—C6     | 178.76 (19)  | C8—C7—C6—C5     | −180.0 (2)   |
| N3—C7—C6—C5     | −1.3 (3)     | C25—C26—C21—C22 | −0.3 (3)     |
| N3—C3—C4—C5     | 0.0 (3)      | C25—C24—C23—C22 | −2.1 (4)     |
| F1—C21—C26—C25  | 179.8 (3)    | C26—C25—C24—C23 | 1.3 (4)      |
| F1—C21—C22—C23  | 179.4 (3)    | C26—C21—C22—C23 | −0.5 (4)     |
| C19—N6—C15—C16  | 2.0 (2)      | C24—C25—C26—C21 | −0.1 (3)     |
| C19—C18—C17—C16 | 2.0 (3)      | C24—C23—C22—C21 | 1.7 (4)      |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_140K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 14.3456$  (3) Å

$b = 12.5246$  (3) Å

$c = 15.3694$  (3) Å

$\beta = 110.785$  (2)°

$V = 2581.74$  (10) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.437$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 41564 reflections

$\theta = 2.1$ –32.4°

$\mu = 0.78 \text{ mm}^{-1}$   
 $T = 140 \text{ K}$

Block, red  
 $0.32 \times 0.2 \times 0.12 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.656$ ,  $T_{\max} = 1.000$   
 72682 measured reflections  
 6413 independent reflections  
 5895 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -16 \rightarrow 16$   
 $l = -17 \rightarrow 20$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.128$   
 $S = 1.04$   
 6413 reflections  
 325 parameters  
 0 restraints

43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 4.3195P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = -0.0002$   
 $\Delta\rho_{\max} = 0.94 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.70 \text{ e } \text{\AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.66309 (2)  | 0.34974 (3)  | 0.82120 (2)  | 0.02119 (10)                     |
| S2  | 0.47565 (5)  | 0.04337 (5)  | 0.68355 (5)  | 0.03361 (15)                     |
| S1  | 0.75502 (7)  | 0.32551 (7)  | 0.55482 (5)  | 0.0494 (2)                       |
| N5  | 0.77560 (14) | 0.25642 (16) | 0.89476 (12) | 0.0237 (4)                       |
| N4  | 0.75302 (14) | 0.46755 (16) | 0.88940 (13) | 0.0232 (4)                       |
| N3  | 0.61101 (14) | 0.35426 (16) | 0.92379 (13) | 0.0237 (4)                       |
| N6  | 0.57369 (14) | 0.46933 (16) | 0.76131 (12) | 0.0239 (4)                       |
| N2  | 0.57622 (15) | 0.23290 (17) | 0.75868 (13) | 0.0273 (4)                       |
| N1  | 0.71528 (15) | 0.35155 (17) | 0.71901 (14) | 0.0278 (4)                       |
| C13 | 0.86106 (17) | 0.3114 (2)   | 0.93625 (15) | 0.0245 (4)                       |
| C19 | 0.62076 (17) | 0.56461 (19) | 0.77235 (15) | 0.0247 (4)                       |
| C18 | 0.57031 (19) | 0.6589 (2)   | 0.73611 (16) | 0.0296 (5)                       |
| H10 | 0.61035 (19) | 0.7357 (2)   | 0.74739 (16) | 0.0355 (6)*                      |
| C15 | 0.47502 (17) | 0.4658 (2)   | 0.71487 (15) | 0.0282 (5)                       |
| H7  | 0.43611 (17) | 0.3885 (2)   | 0.70777 (15) | 0.0339 (6)*                      |
| F1  | 0.7777 (2)   | 0.2767 (3)   | 1.36206 (17) | 0.0920 (9)                       |
| C7  | 0.65231 (17) | 0.42944 (19) | 0.98846 (15) | 0.0248 (4)                       |
| C14 | 0.85663 (17) | 0.4278 (2)   | 0.91257 (17) | 0.0278 (5)                       |
| H18 | 0.88096 (17) | 0.4400 (2)   | 0.85224 (17) | 0.0334 (6)*                      |
| H17 | 0.90783 (17) | 0.4732 (2)   | 0.97265 (17) | 0.0334 (6)*                      |
| C20 | 0.73134 (17) | 0.5596 (2)   | 0.82338 (16) | 0.0278 (5)                       |
| H11 | 0.75793 (17) | 0.6351 (2)   | 0.86223 (16) | 0.0333 (6)*                      |
| H12 | 0.77043 (17) | 0.5487 (2)   | 0.77321 (16) | 0.0333 (6)*                      |
| C12 | 0.94844 (18) | 0.2616 (2)   | 0.99261 (16) | 0.0281 (5)                       |

|     |              |              |              |              |
|-----|--------------|--------------|--------------|--------------|
| H16 | 1.01680 (18) | 0.3087 (2)   | 1.02615 (16) | 0.0338 (6)*  |
| C2  | 0.53424 (17) | 0.1547 (2)   | 0.72606 (15) | 0.0252 (4)   |
| C10 | 0.8610 (2)   | 0.0952 (2)   | 0.96329 (17) | 0.0312 (5)   |
| H14 | 0.8587 (2)   | 0.0080 (2)   | 0.97234 (17) | 0.0375 (6)*  |
| C3  | 0.53828 (18) | 0.2922 (2)   | 0.93290 (17) | 0.0303 (5)   |
| H1  | 0.50367 (18) | 0.2312 (2)   | 0.87938 (17) | 0.0363 (6)*  |
| C9  | 0.77586 (19) | 0.15028 (19) | 0.90864 (16) | 0.0276 (5)   |
| H13 | 0.70615 (19) | 0.10519 (19) | 0.87555 (16) | 0.0331 (6)*  |
| C6  | 0.62334 (19) | 0.4432 (2)   | 1.06510 (16) | 0.0328 (5)   |
| H4  | 0.65927 (19) | 0.5047 (2)   | 1.11769 (16) | 0.0394 (6)*  |
| C17 | 0.46849 (19) | 0.6542 (2)   | 0.68542 (17) | 0.0327 (5)   |
| H9  | 0.42687 (19) | 0.7269 (2)   | 0.65377 (17) | 0.0392 (6)*  |
| C16 | 0.42010 (19) | 0.5562 (2)   | 0.67535 (16) | 0.0318 (5)   |
| H8  | 0.33921 (19) | 0.5504 (2)   | 0.63652 (16) | 0.0381 (6)*  |
| C11 | 0.9485 (2)   | 0.1517 (2)   | 1.00610 (17) | 0.0320 (5)   |
| H15 | 1.0168 (2)   | 0.1103 (2)   | 1.05003 (17) | 0.0385 (6)*  |
| C8  | 0.73258 (18) | 0.4968 (2)   | 0.97543 (15) | 0.0284 (5)   |
| H5  | 0.80213 (18) | 0.4874 (2)   | 1.03697 (15) | 0.0341 (6)*  |
| H6  | 0.71000 (18) | 0.5821 (2)   | 0.97118 (15) | 0.0341 (6)*  |
| C1  | 0.73216 (19) | 0.3419 (2)   | 0.65075 (17) | 0.0301 (5)   |
| C4  | 0.5055 (2)   | 0.3022 (2)   | 1.00710 (18) | 0.0363 (6)   |
| H2  | 0.4457 (2)   | 0.2502 (2)   | 1.01251 (18) | 0.0436 (7)*  |
| C5  | 0.5492 (2)   | 0.3791 (3)   | 1.07473 (18) | 0.0374 (6)   |
| H3  | 0.5251 (2)   | 0.3888 (3)   | 1.13491 (18) | 0.0448 (7)*  |
| C25 | 0.7695 (2)   | 0.2142 (3)   | 1.1350 (2)   | 0.0460 (7)   |
| H22 | 0.7998 (2)   | 0.2435 (3)   | 1.0828 (2)   | 0.0551 (9)*  |
| C26 | 0.7942 (2)   | 0.2669 (3)   | 1.2186 (2)   | 0.0492 (8)   |
| H23 | 0.8434 (2)   | 0.3374 (3)   | 1.2342 (2)   | 0.0591 (9)*  |
| C21 | 0.7550 (3)   | 0.2281 (3)   | 1.2819 (2)   | 0.0551 (9)   |
| C24 | 0.7066 (3)   | 0.1237 (3)   | 1.1136 (3)   | 0.0601 (9)   |
| H21 | 0.6888 (3)   | 0.0814 (3)   | 1.0467 (3)   | 0.0721 (11)* |
| C22 | 0.6930 (3)   | 0.1412 (3)   | 1.2649 (3)   | 0.0646 (11)  |
| H19 | 0.6637 (3)   | 0.1128 (3)   | 1.3182 (3)   | 0.0775 (13)* |
| C23 | 0.6677 (3)   | 0.0898 (3)   | 1.1804 (4)   | 0.0700 (11)  |
| H20 | 0.6163 (3)   | 0.0211 (3)   | 1.1653 (4)   | 0.0839 (14)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02254 (16) | 0.02754 (18) | 0.01416 (15) | −0.00347 (12) | 0.00732 (11) | −0.00055 (11) |
| S2  | 0.0331 (3)   | 0.0327 (3)   | 0.0373 (3)   | −0.0070 (2)   | 0.0153 (3)   | −0.0100 (3)   |
| S1  | 0.0727 (5)   | 0.0597 (5)   | 0.0270 (3)   | 0.0332 (4)    | 0.0315 (3)   | 0.0159 (3)    |
| N5  | 0.0262 (9)   | 0.0281 (10)  | 0.0171 (8)   | −0.0019 (8)   | 0.0081 (7)   | 0.0002 (7)    |
| N4  | 0.0238 (9)   | 0.0276 (10)  | 0.0182 (8)   | −0.0040 (7)   | 0.0073 (7)   | 0.0010 (7)    |
| N3  | 0.0246 (9)   | 0.0314 (10)  | 0.0162 (8)   | −0.0017 (7)   | 0.0086 (7)   | 0.0001 (7)    |
| N6  | 0.0247 (9)   | 0.0307 (10)  | 0.0160 (8)   | −0.0018 (8)   | 0.0069 (7)   | −0.0008 (7)   |
| N2  | 0.0278 (10)  | 0.0322 (11)  | 0.0204 (9)   | −0.0063 (8)   | 0.0069 (7)   | −0.0044 (8)   |
| N1  | 0.0323 (10)  | 0.0354 (11)  | 0.0182 (9)   | 0.0017 (8)    | 0.0120 (8)   | 0.0014 (8)    |



|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C13 | 0.0236 (10) | 0.0306 (12) | 0.0199 (10) | −0.0010 (9)  | 0.0085 (8)  | 0.0013 (8)   |
| C19 | 0.0269 (11) | 0.0306 (12) | 0.0169 (9)  | −0.0024 (9)  | 0.0078 (8)  | 0.0012 (8)   |
| C18 | 0.0322 (12) | 0.0319 (12) | 0.0236 (11) | −0.0000 (10) | 0.0086 (9)  | 0.0028 (9)   |
| C15 | 0.0255 (11) | 0.0368 (13) | 0.0204 (10) | −0.0034 (10) | 0.0056 (8)  | −0.0016 (9)  |
| F1  | 0.0790 (17) | 0.144 (3)   | 0.0564 (14) | 0.0285 (18)  | 0.0286 (13) | −0.0006 (16) |
| C7  | 0.0255 (10) | 0.0321 (12) | 0.0165 (9)  | −0.0005 (9)  | 0.0073 (8)  | 0.0010 (8)   |
| C14 | 0.0228 (10) | 0.0325 (12) | 0.0271 (11) | −0.0062 (9)  | 0.0076 (9)  | 0.0023 (9)   |
| C20 | 0.0264 (11) | 0.0308 (12) | 0.0238 (11) | −0.0047 (9)  | 0.0062 (9)  | 0.0041 (9)   |
| C12 | 0.0252 (11) | 0.0360 (13) | 0.0226 (10) | 0.0018 (9)   | 0.0077 (9)  | −0.0001 (9)  |
| C2  | 0.0240 (10) | 0.0328 (12) | 0.0191 (10) | −0.0018 (9)  | 0.0081 (8)  | −0.0016 (9)  |
| C10 | 0.0413 (14) | 0.0265 (12) | 0.0261 (11) | 0.0029 (10)  | 0.0123 (10) | −0.0003 (9)  |
| C3  | 0.0286 (11) | 0.0422 (14) | 0.0232 (11) | −0.0067 (10) | 0.0130 (9)  | 0.0003 (10)  |
| C9  | 0.0340 (12) | 0.0287 (12) | 0.0208 (10) | −0.0022 (9)  | 0.0107 (9)  | −0.0008 (8)  |
| C6  | 0.0351 (13) | 0.0459 (15) | 0.0193 (10) | 0.0022 (11)  | 0.0119 (9)  | −0.0023 (10) |
| C17 | 0.0317 (12) | 0.0407 (14) | 0.0243 (11) | 0.0047 (11)  | 0.0083 (9)  | 0.0050 (10)  |
| C16 | 0.0269 (11) | 0.0417 (14) | 0.0240 (11) | 0.0017 (10)  | 0.0057 (9)  | 0.0004 (10)  |
| C11 | 0.0337 (13) | 0.0367 (13) | 0.0256 (11) | 0.0070 (10)  | 0.0104 (10) | 0.0027 (10)  |
| C8  | 0.0316 (12) | 0.0335 (12) | 0.0193 (10) | −0.0069 (10) | 0.0079 (9)  | −0.0061 (9)  |
| C1  | 0.0360 (12) | 0.0380 (13) | 0.0196 (10) | 0.0110 (10)  | 0.0138 (9)  | 0.0056 (9)   |
| C4  | 0.0334 (12) | 0.0541 (17) | 0.0282 (12) | −0.0041 (12) | 0.0192 (10) | 0.0062 (11)  |
| C5  | 0.0384 (13) | 0.0570 (17) | 0.0233 (11) | 0.0025 (12)  | 0.0190 (10) | 0.0034 (11)  |
| C25 | 0.0430 (15) | 0.0564 (19) | 0.0453 (16) | 0.0133 (14)  | 0.0242 (13) | 0.0186 (14)  |
| C26 | 0.0305 (13) | 0.067 (2)   | 0.0535 (18) | 0.0103 (14)  | 0.0191 (13) | 0.0195 (16)  |
| C21 | 0.0455 (17) | 0.084 (3)   | 0.0406 (16) | 0.0218 (17)  | 0.0212 (14) | 0.0175 (17)  |
| C24 | 0.0515 (19) | 0.063 (2)   | 0.070 (2)   | 0.0098 (17)  | 0.0265 (18) | 0.0185 (19)  |
| C22 | 0.059 (2)   | 0.073 (3)   | 0.076 (3)   | 0.0103 (19)  | 0.041 (2)   | 0.039 (2)    |
| C23 | 0.066 (2)   | 0.057 (2)   | 0.097 (3)   | 0.0026 (19)  | 0.042 (2)   | 0.017 (2)    |

*Geometric parameters (Å, °)*

|        |             |         |           |
|--------|-------------|---------|-----------|
| Fe1—N5 | 1.989 (2)   | C20—H11 | 1.1110    |
| Fe1—N4 | 1.9952 (19) | C20—H12 | 1.1110    |
| Fe1—N3 | 1.9706 (19) | C12—H16 | 1.1030    |
| Fe1—N6 | 1.974 (2)   | C12—C11 | 1.393 (4) |
| Fe1—N2 | 1.941 (2)   | C10—H14 | 1.1030    |
| Fe1—N1 | 1.965 (2)   | C10—C9  | 1.394 (3) |
| S2—C2  | 1.639 (2)   | C10—C11 | 1.386 (4) |
| S1—C1  | 1.633 (2)   | C3—H1   | 1.1030    |
| N5—C13 | 1.353 (3)   | C3—C4   | 1.385 (3) |
| N5—C9  | 1.346 (3)   | C9—H13  | 1.1030    |
| N4—C14 | 1.486 (3)   | C6—H4   | 1.1030    |
| N4—C20 | 1.493 (3)   | C6—C5   | 1.381 (4) |
| N4—C8  | 1.498 (3)   | C17—H9  | 1.1030    |
| N3—C7  | 1.344 (3)   | C17—C16 | 1.391 (4) |
| N3—C3  | 1.348 (3)   | C16—H8  | 1.1030    |
| N6—C19 | 1.352 (3)   | C11—H15 | 1.1030    |
| N6—C15 | 1.341 (3)   | C8—H5   | 1.1110    |
| N2—C2  | 1.166 (3)   | C8—H6   | 1.1110    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| N1—C1      | 1.164 (3)   | C4—H2       | 1.1030      |
| C13—C14    | 1.498 (3)   | C4—C5       | 1.392 (4)   |
| C13—C12    | 1.393 (3)   | C5—H3       | 1.1030      |
| C19—C18    | 1.393 (3)   | C25—H22     | 1.1030      |
| C19—C20    | 1.501 (3)   | C25—C26     | 1.374 (5)   |
| C18—H10    | 1.1030      | C25—C24     | 1.413 (5)   |
| C18—C17    | 1.392 (3)   | C26—H23     | 1.1030      |
| C15—H7     | 1.1030      | C26—C21     | 1.373 (4)   |
| C15—C16    | 1.390 (4)   | C21—C22     | 1.370 (6)   |
| F1—C21     | 1.307 (4)   | C24—H21     | 1.1030      |
| C7—C6      | 1.392 (3)   | C24—C23     | 1.396 (5)   |
| C7—C8      | 1.497 (3)   | C22—H19     | 1.1030      |
| C14—H18    | 1.1110      | C22—C23     | 1.378 (6)   |
| C14—H17    | 1.1110      | C23—H20     | 1.1030      |
| N4—Fe1—N5  | 83.85 (8)   | H12—C20—H11 | 108.4       |
| N3—Fe1—N5  | 91.36 (8)   | H16—C12—C13 | 120.39 (15) |
| N3—Fe1—N4  | 85.21 (8)   | C11—C12—C13 | 119.2 (2)   |
| N6—Fe1—N5  | 166.63 (8)  | C11—C12—H16 | 120.39 (15) |
| N6—Fe1—N4  | 82.87 (8)   | N2—C2—S2    | 178.2 (2)   |
| N6—Fe1—N3  | 89.04 (8)   | C9—C10—H14  | 120.47 (15) |
| N2—Fe1—N5  | 95.01 (8)   | C11—C10—H14 | 120.47 (15) |
| N2—Fe1—N4  | 178.15 (8)  | C11—C10—C9  | 119.1 (2)   |
| N2—Fe1—N3  | 93.37 (8)   | H1—C3—N3    | 118.84 (13) |
| N2—Fe1—N6  | 98.31 (8)   | C4—C3—N3    | 122.3 (2)   |
| N1—Fe1—N5  | 89.92 (8)   | C4—C3—H1    | 118.84 (16) |
| N1—Fe1—N4  | 93.01 (8)   | C10—C9—N5   | 122.4 (2)   |
| N1—Fe1—N3  | 177.69 (9)  | H13—C9—N5   | 118.78 (14) |
| N1—Fe1—N6  | 89.27 (8)   | H13—C9—C10  | 118.78 (15) |
| N1—Fe1—N2  | 88.44 (8)   | H4—C6—C7    | 120.28 (15) |
| C13—N5—Fe1 | 112.68 (16) | C5—C6—C7    | 119.4 (2)   |
| C9—N5—Fe1  | 128.80 (17) | C5—C6—H4    | 120.28 (15) |
| C9—N5—C13  | 118.5 (2)   | H9—C17—C18  | 120.58 (16) |
| C14—N4—Fe1 | 106.48 (14) | C16—C17—C18 | 118.8 (2)   |
| C20—N4—Fe1 | 105.89 (13) | C16—C17—H9  | 120.58 (15) |
| C20—N4—C14 | 111.75 (18) | C17—C16—C15 | 119.1 (2)   |
| C8—N4—Fe1  | 110.61 (14) | H8—C16—C15  | 120.43 (14) |
| C8—N4—C14  | 111.28 (18) | H8—C16—C17  | 120.43 (15) |
| C8—N4—C20  | 110.61 (19) | C10—C11—C12 | 118.8 (2)   |
| C7—N3—Fe1  | 114.76 (15) | H15—C11—C12 | 120.62 (15) |
| C3—N3—Fe1  | 126.42 (16) | H15—C11—C10 | 120.62 (15) |
| C3—N3—C7   | 118.8 (2)   | C7—C8—N4    | 112.39 (18) |
| C19—N6—Fe1 | 113.47 (15) | H5—C8—N4    | 109.12 (12) |
| C15—N6—Fe1 | 127.70 (17) | H5—C8—C7    | 109.12 (12) |
| C15—N6—C19 | 118.8 (2)   | H6—C8—N4    | 109.12 (12) |
| C2—N2—Fe1  | 171.6 (2)   | H6—C8—C7    | 109.12 (14) |
| C1—N1—Fe1  | 168.4 (2)   | H6—C8—H5    | 107.9       |
| C14—C13—N5 | 115.3 (2)   | N1—C1—S1    | 178.7 (3)   |

|                |              |                 |             |
|----------------|--------------|-----------------|-------------|
| C12—C13—N5     | 122.0 (2)    | H2—C4—C3        | 120.56 (16) |
| C12—C13—C14    | 122.5 (2)    | C5—C4—C3        | 118.9 (2)   |
| C18—C19—N6     | 122.2 (2)    | C5—C4—H2        | 120.56 (15) |
| C20—C19—N6     | 114.6 (2)    | C4—C5—C6        | 118.8 (2)   |
| C20—C19—C18    | 123.2 (2)    | H3—C5—C6        | 120.60 (15) |
| H10—C18—C19    | 120.61 (14)  | H3—C5—C4        | 120.60 (15) |
| C17—C18—C19    | 118.8 (2)    | C26—C25—H22     | 118.85 (19) |
| C17—C18—H10    | 120.61 (16)  | C24—C25—H22     | 118.9 (2)   |
| H7—C15—N6      | 118.89 (14)  | C24—C25—C26     | 122.3 (3)   |
| C16—C15—N6     | 122.2 (2)    | H23—C26—C25     | 121.37 (19) |
| C16—C15—H7     | 118.89 (14)  | C21—C26—C25     | 117.3 (4)   |
| C6—C7—N3       | 121.8 (2)    | C21—C26—H23     | 121.4 (2)   |
| C8—C7—N3       | 117.00 (19)  | C26—C21—F1      | 118.3 (4)   |
| C8—C7—C6       | 121.2 (2)    | C22—C21—F1      | 118.5 (3)   |
| C13—C14—N4     | 109.67 (18)  | C22—C21—C26     | 123.2 (4)   |
| H18—C14—N4     | 109.74 (12)  | H21—C24—C25     | 121.3 (2)   |
| H18—C14—C13    | 109.74 (13)  | C23—C24—C25     | 117.5 (4)   |
| H17—C14—N4     | 109.74 (12)  | C23—C24—H21     | 121.3 (3)   |
| H17—C14—C13    | 109.74 (12)  | H19—C22—C21     | 120.5 (2)   |
| H17—C14—H18    | 108.2        | C23—C22—C21     | 119.1 (3)   |
| C19—C20—N4     | 108.31 (18)  | C23—C22—H19     | 120.5 (2)   |
| H11—C20—N4     | 110.03 (12)  | C22—C23—C24     | 120.7 (4)   |
| H11—C20—C19    | 110.03 (13)  | H20—C23—C24     | 119.6 (3)   |
| H12—C20—N4     | 110.03 (12)  | H20—C23—C22     | 119.6 (2)   |
| H12—C20—C19    | 110.03 (12)  |                 |             |
| Fe1—N5—C13—C14 | −4.93 (16)   | C19—N6—C15—C16  | 2.0 (2)     |
| Fe1—N5—C13—C12 | 178.70 (14)  | C19—C18—C17—C16 | 2.1 (3)     |
| Fe1—N5—C9—C10  | −179.26 (19) | C19—C20—N4—C14  | 155.88 (19) |
| Fe1—N4—C14—C13 | −36.75 (14)  | C19—C20—N4—C8   | −79.5 (2)   |
| Fe1—N4—C20—C19 | 40.34 (14)   | C18—C19—N6—C15  | −0.8 (3)    |
| Fe1—N4—C8—C7   | 1.06 (15)    | C18—C17—C16—C15 | −1.0 (3)    |
| Fe1—N3—C7—C6   | 179.74 (16)  | C15—N6—C19—C20  | −178.7 (2)  |
| Fe1—N3—C7—C8   | −1.38 (18)   | F1—C21—C26—C25  | 179.8 (3)   |
| Fe1—N3—C3—C4   | −179.0 (2)   | F1—C21—C22—C23  | 179.4 (3)   |
| Fe1—N6—C19—C18 | −179.26 (15) | C7—N3—C3—C4     | −0.8 (3)    |
| Fe1—N6—C19—C20 | 2.84 (16)    | C7—C6—C5—C4     | 0.1 (3)     |
| Fe1—N6—C15—C16 | −179.81 (19) | C7—C8—N4—C14    | −117.1 (2)  |
| N5—C13—C14—N4  | 28.4 (2)     | C7—C8—N4—C20    | 118.1 (2)   |
| N5—C13—C12—C11 | 0.7 (3)      | C14—C13—N5—C9   | 176.21 (19) |
| N5—C9—C10—C11  | 0.9 (3)      | C14—C13—C12—C11 | −175.4 (2)  |
| N4—C14—C13—C12 | −155.25 (18) | C20—C19—C18—C17 | 176.5 (2)   |
| N4—C20—C19—N6  | −29.3 (2)    | C12—C13—N5—C9   | −0.2 (3)    |
| N4—C20—C19—C18 | 152.82 (18)  | C12—C11—C10—C9  | −0.3 (3)    |
| N4—C8—C7—N3    | 0.2 (2)      | C3—N3—C7—C6     | 1.3 (3)     |
| N4—C8—C7—C6    | 179.1 (2)    | C3—N3—C7—C8     | −179.8 (2)  |
| N3—C7—C6—C5    | −0.9 (3)     | C3—C4—C5—C6     | 0.4 (3)     |
| N3—C3—C4—C5    | −0.1 (3)     | C8—C7—C6—C5     | −179.8 (2)  |

|                 |              |                 |          |
|-----------------|--------------|-----------------|----------|
| N6—C19—C18—C17  | −1.2 (3)     | C25—C26—C21—C22 | −0.5 (4) |
| N6—C15—C16—C17  | −1.1 (3)     | C25—C24—C23—C22 | −2.1 (4) |
| C13—N5—C9—C10   | −0.6 (2)     | C26—C25—C24—C23 | 1.3 (4)  |
| C13—C14—N4—C20  | −151.93 (18) | C26—C21—C22—C23 | −0.3 (4) |
| C13—C14—N4—C8   | 83.9 (2)     | C21—C26—C25—C24 | −0.0 (4) |
| C13—C12—C11—C10 | −0.4 (3)     | C21—C22—C23—C24 | 1.6 (4)  |

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) fluorobenzene monosolvate (f\_150K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 14.3703$  (3) Å

$b = 12.5251$  (3) Å

$c = 15.3745$  (4) Å

$\beta = 110.774$  (3)°

$V = 2587.34$  (12) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.434$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 40012 reflections

$\theta = 2.1$ – $32.3^\circ$

$\mu = 0.78$  mm<sup>−1</sup>

$T = 150$  K

Block, red

$0.32 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.662$ ,  $T_{\max} = 1.000$

74232 measured reflections

6434 independent reflections

5915 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.133$

$S = 1.04$

6434 reflections

325 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 4.4152P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.82$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.74$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | $x$          | $y$          | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.66316 (2)  | 0.34947 (3)  | 0.82099 (2)  | 0.02276 (10)                     |
| S2  | 0.47568 (5)  | 0.04333 (6)  | 0.68334 (5)  | 0.03607 (16)                     |
| S1  | 0.75489 (7)  | 0.32550 (8)  | 0.55463 (5)  | 0.0527 (2)                       |
| N4  | 0.77571 (15) | 0.25628 (17) | 0.89461 (13) | 0.0249 (4)                       |
| N6  | 0.57388 (15) | 0.46914 (17) | 0.76133 (13) | 0.0254 (4)                       |
| N5  | 0.75294 (14) | 0.46732 (16) | 0.88917 (13) | 0.0247 (4)                       |
| N3  | 0.61121 (15) | 0.35394 (16) | 0.92366 (13) | 0.0252 (4)                       |
| N1  | 0.71522 (16) | 0.35163 (17) | 0.71879 (14) | 0.0294 (4)                       |

|     |              |              |              |              |
|-----|--------------|--------------|--------------|--------------|
| N2  | 0.57645 (15) | 0.23265 (18) | 0.75877 (14) | 0.0293 (4)   |
| C19 | 0.62077 (18) | 0.5644 (2)   | 0.77244 (15) | 0.0260 (5)   |
| C13 | 0.86100 (17) | 0.3112 (2)   | 0.93591 (15) | 0.0259 (5)   |
| C15 | 0.47541 (18) | 0.4658 (2)   | 0.71483 (16) | 0.0300 (5)   |
| H7  | 0.43664 (18) | 0.3885 (2)   | 0.70742 (16) | 0.0360 (6)*  |
| C18 | 0.5703 (2)   | 0.6587 (2)   | 0.73632 (17) | 0.0314 (5)   |
| H10 | 0.6102 (2)   | 0.7356 (2)   | 0.74762 (17) | 0.0376 (6)*  |
| C7  | 0.65238 (17) | 0.4291 (2)   | 0.98832 (15) | 0.0259 (5)   |
| C2  | 0.53449 (17) | 0.1543 (2)   | 0.72599 (16) | 0.0269 (5)   |
| C12 | 0.94834 (18) | 0.2618 (2)   | 0.99239 (17) | 0.0303 (5)   |
| H16 | 1.01646 (18) | 0.3091 (2)   | 1.02598 (17) | 0.0364 (6)*  |
| C14 | 0.85634 (17) | 0.4279 (2)   | 0.91214 (17) | 0.0294 (5)   |
| H18 | 0.88047 (17) | 0.4401 (2)   | 0.85175 (17) | 0.0353 (6)*  |
| H17 | 0.90744 (17) | 0.4734 (2)   | 0.97214 (17) | 0.0353 (6)*  |
| F1  | 0.7778 (2)   | 0.2764 (3)   | 1.36190 (19) | 0.1019 (11)  |
| C20 | 0.73101 (18) | 0.5594 (2)   | 0.82328 (17) | 0.0296 (5)   |
| H11 | 0.75747 (18) | 0.6349 (2)   | 0.86219 (17) | 0.0355 (6)*  |
| H12 | 0.76996 (18) | 0.5488 (2)   | 0.77304 (17) | 0.0355 (6)*  |
| C16 | 0.42037 (19) | 0.5562 (2)   | 0.67562 (17) | 0.0340 (5)   |
| H8  | 0.33959 (19) | 0.5503 (2)   | 0.63701 (17) | 0.0408 (7)*  |
| C10 | 0.8611 (2)   | 0.0953 (2)   | 0.96315 (18) | 0.0338 (5)   |
| H14 | 0.8590 (2)   | 0.0081 (2)   | 0.97220 (18) | 0.0405 (6)*  |
| C6  | 0.6233 (2)   | 0.4431 (2)   | 1.06484 (17) | 0.0343 (6)   |
| H4  | 0.6591 (2)   | 0.5048 (2)   | 1.11731 (17) | 0.0412 (7)*  |
| C3  | 0.53842 (19) | 0.2919 (2)   | 0.93276 (17) | 0.0322 (5)   |
| H1  | 0.50378 (19) | 0.2310 (2)   | 0.87927 (17) | 0.0387 (6)*  |
| C11 | 0.9484 (2)   | 0.1518 (2)   | 1.00583 (18) | 0.0342 (6)   |
| H15 | 1.0166 (2)   | 0.1104 (2)   | 1.04967 (18) | 0.0410 (7)*  |
| C8  | 0.73266 (19) | 0.4962 (2)   | 0.97532 (16) | 0.0299 (5)   |
| H5  | 0.80211 (19) | 0.4863 (2)   | 1.03674 (16) | 0.0359 (6)*  |
| H6  | 0.71048 (19) | 0.5815 (2)   | 0.97146 (16) | 0.0359 (6)*  |
| C9  | 0.7760 (2)   | 0.1503 (2)   | 0.90857 (17) | 0.0295 (5)   |
| H13 | 0.7065 (2)   | 0.1052 (2)   | 0.87565 (17) | 0.0354 (6)*  |
| C1  | 0.7320 (2)   | 0.3418 (2)   | 0.65047 (17) | 0.0318 (5)   |
| C17 | 0.4687 (2)   | 0.6541 (2)   | 0.68582 (18) | 0.0349 (6)   |
| H9  | 0.4272 (2)   | 0.7269 (2)   | 0.65436 (18) | 0.0418 (7)*  |
| C4  | 0.5057 (2)   | 0.3022 (3)   | 1.00683 (19) | 0.0386 (6)   |
| H2  | 0.4461 (2)   | 0.2502 (3)   | 1.01223 (19) | 0.0464 (7)*  |
| C5  | 0.5492 (2)   | 0.3790 (3)   | 1.07442 (19) | 0.0399 (6)   |
| H3  | 0.5250 (2)   | 0.3886 (3)   | 1.13452 (19) | 0.0479 (8)*  |
| C25 | 0.7695 (2)   | 0.2144 (3)   | 1.1353 (2)   | 0.0493 (8)   |
| H22 | 0.7998 (2)   | 0.2439 (3)   | 1.0831 (2)   | 0.0592 (9)*  |
| C26 | 0.7943 (2)   | 0.2670 (3)   | 1.2188 (3)   | 0.0528 (9)   |
| H23 | 0.8434 (2)   | 0.3375 (3)   | 1.2344 (3)   | 0.0634 (10)* |
| C24 | 0.7068 (3)   | 0.1239 (3)   | 1.1137 (3)   | 0.0644 (10)  |
| H21 | 0.6889 (3)   | 0.0816 (3)   | 1.0468 (3)   | 0.0772 (13)* |
| C21 | 0.7552 (3)   | 0.2281 (4)   | 1.2819 (2)   | 0.0601 (10)  |
| C22 | 0.6936 (3)   | 0.1416 (4)   | 1.2656 (3)   | 0.0715 (13)  |

|     |            |            |            |              |
|-----|------------|------------|------------|--------------|
| H19 | 0.6645 (3) | 0.1132 (4) | 1.3189 (3) | 0.0858 (16)* |
| C23 | 0.6683 (3) | 0.0903 (4) | 1.1807 (4) | 0.0768 (13)  |
| H20 | 0.6169 (3) | 0.0217 (4) | 1.1657 (4) | 0.0921 (16)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.02414 (17) | 0.02953 (19) | 0.01534 (16) | −0.00344 (13) | 0.00791 (12) | −0.00034 (12) |
| S2  | 0.0354 (3)   | 0.0355 (3)   | 0.0397 (4)   | −0.0072 (3)   | 0.0163 (3)   | −0.0105 (3)   |
| S1  | 0.0775 (6)   | 0.0634 (5)   | 0.0290 (3)   | 0.0351 (5)    | 0.0333 (4)   | 0.0167 (3)    |
| N4  | 0.0276 (10)  | 0.0299 (10)  | 0.0175 (8)   | −0.0021 (8)   | 0.0083 (7)   | 0.0005 (7)    |
| N6  | 0.0268 (9)   | 0.0321 (11)  | 0.0174 (8)   | −0.0013 (8)   | 0.0081 (7)   | −0.0008 (7)   |
| N5  | 0.0244 (9)   | 0.0300 (10)  | 0.0195 (8)   | −0.0046 (8)   | 0.0074 (7)   | 0.0011 (7)    |
| N3  | 0.0270 (9)   | 0.0321 (10)  | 0.0174 (8)   | −0.0023 (8)   | 0.0090 (7)   | 0.0001 (7)    |
| N1  | 0.0345 (11)  | 0.0371 (11)  | 0.0196 (9)   | 0.0014 (9)    | 0.0134 (8)   | 0.0012 (8)    |
| N2  | 0.0304 (10)  | 0.0347 (11)  | 0.0214 (9)   | −0.0072 (9)   | 0.0075 (8)   | −0.0046 (8)   |
| C19 | 0.0289 (11)  | 0.0317 (12)  | 0.0176 (9)   | −0.0023 (9)   | 0.0084 (8)   | 0.0008 (8)    |
| C13 | 0.0246 (11)  | 0.0328 (12)  | 0.0207 (10)  | −0.0009 (9)   | 0.0087 (8)   | 0.0011 (9)    |
| C15 | 0.0270 (11)  | 0.0395 (14)  | 0.0215 (10)  | −0.0037 (10)  | 0.0062 (9)   | −0.0025 (9)   |
| C18 | 0.0343 (13)  | 0.0338 (13)  | 0.0244 (11)  | −0.0004 (10)  | 0.0084 (10)  | 0.0032 (9)    |
| C7  | 0.0277 (11)  | 0.0335 (12)  | 0.0171 (10)  | 0.0004 (9)    | 0.0085 (8)   | 0.0005 (9)    |
| C2  | 0.0255 (11)  | 0.0356 (13)  | 0.0204 (10)  | −0.0017 (9)   | 0.0091 (8)   | −0.0016 (9)   |
| C12 | 0.0271 (11)  | 0.0384 (14)  | 0.0247 (11)  | 0.0018 (10)   | 0.0083 (9)   | 0.0005 (10)   |
| C14 | 0.0240 (11)  | 0.0343 (13)  | 0.0288 (12)  | −0.0056 (9)   | 0.0079 (9)   | 0.0021 (10)   |
| F1  | 0.0865 (19)  | 0.162 (3)    | 0.0606 (15)  | 0.033 (2)     | 0.0305 (14)  | −0.0003 (18)  |
| C20 | 0.0292 (12)  | 0.0311 (12)  | 0.0259 (11)  | −0.0056 (10)  | 0.0068 (9)   | 0.0039 (9)    |
| C16 | 0.0285 (12)  | 0.0450 (15)  | 0.0259 (11)  | 0.0014 (11)   | 0.0063 (9)   | 0.0003 (10)   |
| C10 | 0.0444 (15)  | 0.0289 (13)  | 0.0284 (12)  | 0.0035 (11)   | 0.0133 (11)  | 0.0001 (10)   |
| C6  | 0.0375 (13)  | 0.0470 (15)  | 0.0203 (11)  | 0.0020 (12)   | 0.0127 (10)  | −0.0023 (10)  |
| C3  | 0.0300 (12)  | 0.0447 (15)  | 0.0254 (11)  | −0.0070 (11)  | 0.0138 (9)   | 0.0008 (10)   |
| C11 | 0.0360 (13)  | 0.0391 (14)  | 0.0278 (12)  | 0.0076 (11)   | 0.0118 (10)  | 0.0029 (10)   |
| C8  | 0.0338 (12)  | 0.0347 (13)  | 0.0205 (10)  | −0.0074 (10)  | 0.0085 (9)   | −0.0059 (9)   |
| C9  | 0.0367 (13)  | 0.0297 (12)  | 0.0233 (11)  | −0.0034 (10)  | 0.0121 (10)  | −0.0020 (9)   |
| C1  | 0.0381 (13)  | 0.0392 (13)  | 0.0214 (11)  | 0.0117 (11)   | 0.0145 (10)  | 0.0061 (10)   |
| C17 | 0.0344 (13)  | 0.0436 (15)  | 0.0264 (12)  | 0.0063 (11)   | 0.0105 (10)  | 0.0063 (10)   |
| C4  | 0.0356 (13)  | 0.0577 (18)  | 0.0294 (12)  | −0.0043 (13)  | 0.0198 (11)  | 0.0064 (12)   |
| C5  | 0.0416 (14)  | 0.0599 (18)  | 0.0253 (12)  | 0.0014 (13)   | 0.0206 (11)  | 0.0021 (12)   |
| C25 | 0.0469 (17)  | 0.061 (2)    | 0.0475 (17)  | 0.0138 (15)   | 0.0260 (14)  | 0.0189 (15)   |
| C26 | 0.0310 (14)  | 0.073 (2)    | 0.058 (2)    | 0.0112 (15)   | 0.0199 (14)  | 0.0203 (18)   |
| C24 | 0.054 (2)    | 0.066 (2)    | 0.078 (3)    | 0.0108 (18)   | 0.0298 (19)  | 0.022 (2)     |
| C21 | 0.0502 (19)  | 0.093 (3)    | 0.0435 (17)  | 0.027 (2)     | 0.0241 (15)  | 0.0218 (18)   |
| C22 | 0.065 (2)    | 0.080 (3)    | 0.086 (3)    | 0.012 (2)     | 0.046 (2)    | 0.045 (3)     |
| C23 | 0.067 (3)    | 0.062 (3)    | 0.111 (4)    | 0.003 (2)     | 0.043 (3)    | 0.018 (3)     |

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

|        |           |         |           |
|--------|-----------|---------|-----------|
| Fe1—N4 | 1.990 (2) | C14—H17 | 1.1110    |
| Fe1—N6 | 1.975 (2) | F1—C21  | 1.304 (5) |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| Fe1—N5     | 1.996 (2)   | C20—H11     | 1.1110      |
| Fe1—N3     | 1.972 (2)   | C20—H12     | 1.1110      |
| Fe1—N1     | 1.965 (2)   | C16—H8      | 1.1030      |
| Fe1—N2     | 1.940 (2)   | C16—C17     | 1.391 (4)   |
| S2—C2      | 1.638 (3)   | C10—H14     | 1.1030      |
| S1—C1      | 1.632 (3)   | C10—C11     | 1.385 (4)   |
| N4—C13     | 1.351 (3)   | C10—C9      | 1.395 (4)   |
| N4—C9      | 1.344 (3)   | C6—H4       | 1.1030      |
| N6—C19     | 1.351 (3)   | C6—C5       | 1.383 (4)   |
| N6—C15     | 1.341 (3)   | C3—H1       | 1.1030      |
| N5—C14     | 1.485 (3)   | C3—C4       | 1.383 (3)   |
| N5—C20     | 1.493 (3)   | C11—H15     | 1.1030      |
| N5—C8      | 1.498 (3)   | C8—H5       | 1.1110      |
| N3—C7      | 1.344 (3)   | C8—H6       | 1.1110      |
| N3—C3      | 1.349 (3)   | C9—H13      | 1.1030      |
| N1—C1      | 1.165 (3)   | C17—H9      | 1.1030      |
| N2—C2      | 1.168 (3)   | C4—H2       | 1.1030      |
| C19—C18    | 1.395 (4)   | C4—C5       | 1.391 (4)   |
| C19—C20    | 1.499 (3)   | C5—H3       | 1.1030      |
| C13—C12    | 1.392 (3)   | C25—H22     | 1.1030      |
| C13—C14    | 1.502 (4)   | C25—C26     | 1.373 (5)   |
| C15—H7     | 1.1030      | C25—C24     | 1.413 (6)   |
| C15—C16    | 1.390 (4)   | C26—H23     | 1.1030      |
| C18—H10    | 1.1030      | C26—C21     | 1.371 (5)   |
| C18—C17    | 1.390 (4)   | C24—H21     | 1.1030      |
| C7—C6      | 1.392 (3)   | C24—C23     | 1.395 (6)   |
| C7—C8      | 1.497 (3)   | C21—C22     | 1.366 (6)   |
| C12—H16    | 1.1030      | C22—H19     | 1.1030      |
| C12—C11    | 1.394 (4)   | C22—C23     | 1.382 (7)   |
| C14—H18    | 1.1110      | C23—H20     | 1.1030      |
|            |             |             |             |
| N6—Fe1—N4  | 166.51 (8)  | H11—C20—N5  | 110.02 (12) |
| N5—Fe1—N4  | 83.77 (8)   | H11—C20—C19 | 110.02 (14) |
| N5—Fe1—N6  | 82.82 (8)   | H12—C20—N5  | 110.02 (13) |
| N3—Fe1—N4  | 91.34 (8)   | H12—C20—C19 | 110.02 (13) |
| N3—Fe1—N6  | 88.98 (8)   | H12—C20—H11 | 108.4       |
| N3—Fe1—N5  | 85.20 (8)   | H8—C16—C15  | 120.52 (15) |
| N1—Fe1—N4  | 89.99 (8)   | C17—C16—C15 | 119.0 (2)   |
| N1—Fe1—N6  | 89.26 (8)   | C17—C16—H8  | 120.52 (15) |
| N1—Fe1—N5  | 92.94 (8)   | C11—C10—H14 | 120.46 (16) |
| N1—Fe1—N3  | 177.58 (9)  | C9—C10—H14  | 120.46 (16) |
| N2—Fe1—N4  | 95.08 (9)   | C9—C10—C11  | 119.1 (2)   |
| N2—Fe1—N6  | 98.37 (9)   | H4—C6—C7    | 120.34 (16) |
| N2—Fe1—N5  | 178.04 (8)  | C5—C6—C7    | 119.3 (3)   |
| N2—Fe1—N3  | 93.25 (8)   | C5—C6—H4    | 120.34 (16) |
| N2—Fe1—N1  | 88.64 (9)   | H1—C3—N3    | 118.86 (14) |
| C13—N4—Fe1 | 112.79 (16) | C4—C3—N3    | 122.3 (3)   |
| C9—N4—Fe1  | 128.75 (18) | C4—C3—H1    | 118.86 (17) |



|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C9—N4—C13      | 118.4 (2)    | C10—C11—C12     | 118.8 (2)    |
| C19—N6—Fe1     | 113.50 (16)  | H15—C11—C12     | 120.60 (16)  |
| C15—N6—Fe1     | 127.82 (18)  | H15—C11—C10     | 120.60 (16)  |
| C15—N6—C19     | 118.7 (2)    | C7—C8—N5        | 112.57 (19)  |
| C14—N5—Fe1     | 106.54 (15)  | H5—C8—N5        | 109.08 (12)  |
| C20—N5—Fe1     | 105.87 (14)  | H5—C8—C7        | 109.08 (13)  |
| C20—N5—C14     | 111.78 (18)  | H6—C8—N5        | 109.08 (13)  |
| C8—N5—Fe1      | 110.53 (14)  | H6—C8—C7        | 109.08 (14)  |
| C8—N5—C14      | 111.23 (18)  | H6—C8—H5        | 107.8        |
| C8—N5—C20      | 110.7 (2)    | C10—C9—N4       | 122.4 (2)    |
| C7—N3—Fe1      | 114.80 (16)  | H13—C9—N4       | 118.79 (14)  |
| C3—N3—Fe1      | 126.45 (17)  | H13—C9—C10      | 118.79 (16)  |
| C3—N3—C7       | 118.7 (2)    | N1—C1—S1        | 178.8 (3)    |
| C1—N1—Fe1      | 168.2 (2)    | C16—C17—C18     | 118.9 (2)    |
| C2—N2—Fe1      | 171.6 (2)    | H9—C17—C18      | 120.54 (16)  |
| C18—C19—N6     | 122.1 (2)    | H9—C17—C16      | 120.54 (15)  |
| C20—C19—N6     | 114.7 (2)    | H2—C4—C3        | 120.49 (17)  |
| C20—C19—C18    | 123.2 (2)    | C5—C4—C3        | 119.0 (3)    |
| C12—C13—N4     | 122.3 (2)    | C5—C4—H2        | 120.49 (16)  |
| C14—C13—N4     | 115.2 (2)    | C4—C5—C6        | 118.8 (2)    |
| C14—C13—C12    | 122.4 (2)    | H3—C5—C6        | 120.62 (16)  |
| H7—C15—N6      | 118.77 (15)  | H3—C5—C4        | 120.62 (16)  |
| C16—C15—N6     | 122.5 (2)    | C26—C25—H22     | 118.8 (2)    |
| C16—C15—H7     | 118.77 (15)  | C24—C25—H22     | 118.8 (2)    |
| H10—C18—C19    | 120.59 (15)  | C24—C25—C26     | 122.4 (3)    |
| C17—C18—C19    | 118.8 (2)    | H23—C26—C25     | 121.4 (2)    |
| C17—C18—H10    | 120.59 (16)  | C21—C26—C25     | 117.2 (4)    |
| C6—C7—N3       | 121.9 (2)    | C21—C26—H23     | 121.4 (3)    |
| C8—C7—N3       | 116.9 (2)    | H21—C24—C25     | 121.4 (2)    |
| C8—C7—C6       | 121.2 (2)    | C23—C24—C25     | 117.1 (4)    |
| N2—C2—S2       | 178.1 (2)    | C23—C24—H21     | 121.4 (3)    |
| H16—C12—C13    | 120.51 (15)  | C26—C21—F1      | 118.4 (4)    |
| C11—C12—C13    | 119.0 (2)    | C22—C21—F1      | 118.0 (4)    |
| C11—C12—H16    | 120.51 (16)  | C22—C21—C26     | 123.6 (4)    |
| C13—C14—N5     | 109.64 (19)  | H19—C22—C21     | 120.7 (3)    |
| H18—C14—N5     | 109.74 (12)  | C23—C22—C21     | 118.6 (4)    |
| H18—C14—C13    | 109.74 (13)  | C23—C22—H19     | 120.7 (3)    |
| H17—C14—N5     | 109.74 (13)  | C22—C23—C24     | 121.1 (4)    |
| H17—C14—C13    | 109.74 (13)  | H20—C23—C24     | 119.5 (3)    |
| H17—C14—H18    | 108.2        | H20—C23—C22     | 119.5 (3)    |
| C19—C20—N5     | 108.38 (19)  |                 |              |
| Fe1—N4—C13—C12 | 178.57 (15)  | C13—N4—C9—C10   | −0.6 (2)     |
| Fe1—N4—C13—C14 | −4.79 (17)   | C13—C12—C11—C10 | −0.6 (3)     |
| Fe1—N4—C9—C10  | −179.3 (2)   | C13—C14—N5—C20  | −152.02 (19) |
| Fe1—N6—C19—C18 | −179.26 (16) | C13—C14—N5—C8   | 83.7 (2)     |
| Fe1—N6—C19—C20 | 2.86 (17)    | C15—N6—C19—C18  | −0.7 (2)     |
| Fe1—N6—C15—C16 | −179.9 (2)   | C15—N6—C19—C20  | −178.6 (2)   |

|                 |              |                 |            |
|-----------------|--------------|-----------------|------------|
| Fe1—N5—C14—C13  | −36.81 (14)  | C15—C16—C17—C18 | −1.1 (3)   |
| Fe1—N5—C20—C19  | 40.26 (15)   | C7—N3—C3—C4     | −0.9 (3)   |
| Fe1—N5—C8—C7    | 0.73 (16)    | C7—C6—C5—C4     | −0.1 (3)   |
| Fe1—N3—C7—C6    | 179.52 (17)  | C7—C8—N5—C14    | −117.4 (2) |
| Fe1—N3—C7—C8    | −1.59 (19)   | C7—C8—N5—C20    | 117.7 (2)  |
| Fe1—N3—C3—C4    | −178.9 (2)   | C12—C13—N4—C9   | −0.3 (3)   |
| N4—C13—C12—C11  | 0.9 (3)      | C12—C11—C10—C9  | −0.3 (3)   |
| N4—C13—C14—N5   | 28.3 (2)     | C14—C13—N4—C9   | 176.3 (2)  |
| N4—C9—C10—C11   | 1.0 (3)      | C14—C13—C12—C11 | −175.5 (2) |
| N6—C19—C18—C17  | −1.2 (3)     | F1—C21—C26—C25  | 179.8 (3)  |
| N6—C19—C20—N5   | −29.3 (2)    | F1—C21—C22—C23  | 179.4 (4)  |
| N6—C15—C16—C17  | −0.8 (3)     | C20—C19—C18—C17 | 176.5 (2)  |
| N5—C14—C13—C12  | −155.02 (18) | C6—C7—N3—C3     | 1.2 (3)    |
| N5—C20—C19—C18  | 152.86 (19)  | C6—C5—C4—C3     | 0.4 (3)    |
| N5—C8—C7—N3     | 0.5 (2)      | C3—N3—C7—C8     | −179.9 (2) |
| N5—C8—C7—C6     | 179.4 (2)    | C8—C7—C6—C5     | −179.6 (3) |
| N3—C7—C6—C5     | −0.8 (3)     | C25—C26—C21—C22 | −0.4 (4)   |
| N3—C3—C4—C5     | 0.0 (3)      | C25—C24—C23—C22 | −2.1 (4)   |
| C19—N6—C15—C16  | 1.7 (2)      | C26—C25—C24—C23 | 1.3 (4)    |
| C19—C18—C17—C16 | 2.1 (3)      | C26—C21—C22—C23 | −0.4 (4)   |
| C19—C20—N5—C14  | 155.89 (19)  | C24—C25—C26—C21 | −0.1 (4)   |
| C19—C20—N5—C8   | −79.5 (2)    | C24—C23—C22—C21 | 1.7 (5)    |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_155K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.1594$  (3) Å

$b = 12.0053$  (2) Å

$c = 15.4313$  (3) Å

$\beta = 110.511$  (2)°

$V = 2630.35$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.410$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 57132 reflections

$\theta = 2.1$ – $32.6^\circ$

$\mu = 0.77$  mm<sup>−1</sup>

$T = 155$  K

Block, yellow

$0.32 \times 0.2 \times 0.12$  mm

## Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.661$ ,  $T_{\max} = 1.000$

76654 measured reflections

6525 independent reflections

6159 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.103$

$S = 1.05$

6525 reflections

408 parameters  
 149 restraints  
 76 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 2.8798P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.26 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.87 \text{ e } \text{\AA}^{-3}$

*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 | 0.344261 (18) | 0.33272 (2)  | 0.195523 (17) | 0.02688 (8)                      |           |
| S2  | 0.52840 (4)   | 0.00390 (5)  | 0.32415 (5)   | 0.04698 (15)                     |           |
| S1  | 0.23412 (13)  | 0.34440 (13) | 0.45481 (10)  | 0.0623 (2)                       |           |
| N4  | 0.25840 (10)  | 0.47331 (12) | 0.11546 (10)  | 0.0246 (3)                       |           |
| N5  | 0.21476 (11)  | 0.25241 (12) | 0.10817 (10)  | 0.0260 (3)                       |           |
| N3  | 0.39192 (11)  | 0.34110 (14) | 0.07830 (10)  | 0.0295 (3)                       |           |
| N6  | 0.43442 (11)  | 0.47717 (13) | 0.24452 (10)  | 0.0266 (3)                       |           |
| N1  | 0.29065 (13)  | 0.35533 (14) | 0.30227 (13)  | 0.0370 (4)                       |           |
| N2  | 0.42385 (13)  | 0.19508 (17) | 0.25271 (13)  | 0.0431 (4)                       |           |
| C19 | 0.39032 (13)  | 0.57566 (15) | 0.22673 (12)  | 0.0261 (3)                       |           |
| C13 | 0.14197 (12)  | 0.32201 (14) | 0.07040 (12)  | 0.0253 (3)                       |           |
| C18 | 0.43752 (14)  | 0.67613 (16) | 0.25616 (13)  | 0.0326 (4)                       |           |
| H10 | 0.39942 (14)  | 0.75607 (16) | 0.23963 (13)  | 0.0391 (5)*                      |           |
| C7  | 0.35380 (13)  | 0.42146 (16) | 0.01637 (12)  | 0.0289 (4)                       |           |
| C9  | 0.20168 (15)  | 0.14307 (15) | 0.08885 (12)  | 0.0311 (4)                       |           |
| H13 | 0.26137 (15)  | 0.08571 (15) | 0.11945 (12)  | 0.0374 (5)*                      |           |
| C12 | 0.05439 (13)  | 0.28456 (16) | 0.01205 (13)  | 0.0311 (4)                       |           |
| H16 | −0.00374 (13) | 0.34396 (16) | −0.01861 (13) | 0.0373 (4)*                      |           |
| C14 | 0.15945 (12)  | 0.44248 (15) | 0.09779 (13)  | 0.0295 (4)                       |           |
| H18 | 0.14234 (12)  | 0.45762 (15) | 0.16116 (13)  | 0.0354 (4)*                      |           |
| H17 | 0.11248 (12)  | 0.49571 (15) | 0.04161 (13)  | 0.0354 (4)*                      |           |
| C20 | 0.28535 (13)  | 0.57148 (15) | 0.17702 (13)  | 0.0300 (4)                       |           |
| H11 | 0.26196 (13)  | 0.64857 (15) | 0.13546 (13)  | 0.0360 (4)*                      |           |
| H12 | 0.24939 (13)  | 0.56770 (15) | 0.22852 (13)  | 0.0360 (4)*                      |           |
| C10 | 0.11617 (15)  | 0.09941 (16) | 0.03202 (13)  | 0.0360 (4)                       |           |
| H14 | 0.10799 (15)  | 0.00916 (16) | 0.01817 (13)  | 0.0432 (5)*                      |           |
| C15 | 0.52750 (14)  | 0.47531 (18) | 0.29095 (13)  | 0.0340 (4)                       |           |
| H7  | 0.56422 (14)  | 0.39434 (18) | 0.30359 (13)  | 0.0408 (5)*                      |           |
| C8  | 0.27585 (14)  | 0.49116 (17) | 0.02781 (12)  | 0.0326 (4)                       |           |
| H5  | 0.20974 (14)  | 0.47323 (17) | −0.03086 (12) | 0.0392 (5)*                      |           |
| H6  | 0.29307 (14)  | 0.58044 (17) | 0.02334 (12)  | 0.0392 (5)*                      |           |
| C17 | 0.53321 (15)  | 0.67355 (18) | 0.30655 (14)  | 0.0382 (4)                       |           |
| H9  | 0.57215 (15)  | 0.75135 (18) | 0.33266 (14)  | 0.0459 (5)*                      |           |
| C11 | 0.04112 (15)  | 0.17166 (17) | −0.00705 (14) | 0.0354 (4)                       |           |
| H15 | −0.02782 (15) | 0.13978 (17) | −0.05248 (14) | 0.0425 (5)*                      |           |
| C2  | 0.46752 (14)  | 0.11485 (18) | 0.28243 (13)  | 0.0343 (4)                       |           |
| C16 | 0.57950 (15)  | 0.57137 (19) | 0.32375 (14)  | 0.0386 (4)                       |           |
| H8  | 0.65587 (15)  | 0.56679 (19) | 0.36262 (14)  | 0.0464 (5)*                      |           |
| C1  | 0.26681 (15)  | 0.35225 (17) | 0.36609 (14)  | 0.0361 (4)                       |           |
| C3  | 0.45957 (15)  | 0.2768 (2)   | 0.06693 (14)  | 0.0411 (5)                       |           |

|      |              |             |               |              |           |
|------|--------------|-------------|---------------|--------------|-----------|
| H1   | 0.49126 (15) | 0.2109 (2)  | 0.11824 (14)  | 0.0493 (6)*  |           |
| C6   | 0.38178 (15) | 0.4398 (2)  | −0.05917 (14) | 0.0423 (5)   |           |
| H4   | 0.34914 (15) | 0.5066 (2)  | −0.10916 (14) | 0.0508 (6)*  |           |
| C4   | 0.49117 (17) | 0.2897 (3)  | −0.00637 (16) | 0.0523 (6)   |           |
| H2   | 0.54677 (17) | 0.2351 (3)  | −0.01353 (16) | 0.0628 (8)*  |           |
| C5   | 0.45134 (17) | 0.3727 (3)  | −0.07072 (16) | 0.0540 (7)   |           |
| H3   | 0.47465 (17) | 0.3853 (3)  | −0.13017 (16) | 0.0648 (8)*  |           |
| C24a | 0.2215 (4)   | 0.2209 (5)  | −0.1307 (4)   | 0.0499 (14)  | 0.128 (6) |
| C25a | 0.1931 (3)   | 0.2801 (4)  | −0.2119 (3)   | 0.0519 (10)  | 0.128 (6) |
| C23a | 0.2782 (5)   | 0.1285 (5)  | −0.1196 (5)   | 0.0792 (18)  | 0.128 (6) |
| C26a | 0.2237 (3)   | 0.2483 (5)  | −0.2810 (3)   | 0.0728 (14)  | 0.128 (6) |
| F1   | 0.1990 (3)   | 0.2980 (4)  | −0.35892 (19) | 0.143 (2)    | 0.752 (6) |
| C21a | 0.2807 (4)   | 0.1577 (6)  | −0.2727 (5)   | 0.102 (2)    | 0.128 (6) |
| C22a | 0.3076 (4)   | 0.0983 (4)  | −0.1922 (6)   | 0.109 (3)    | 0.128 (6) |
| C23  | 0.3076 (4)   | 0.0983 (4)  | −0.1922 (6)   | 0.109 (3)    | 0.752 (6) |
| C22  | 0.2807 (4)   | 0.1577 (6)  | −0.2727 (5)   | 0.102 (2)    | 0.752 (6) |
| F1a  | 0.2911 (12)  | 0.1497 (14) | −0.3562 (10)  | 0.086 (7)    | 0.128 (6) |
| C21  | 0.2237 (3)   | 0.2483 (5)  | −0.2810 (3)   | 0.0728 (14)  | 0.752 (6) |
| C26  | 0.1931 (3)   | 0.2801 (4)  | −0.2119 (3)   | 0.0519 (10)  | 0.752 (6) |
| C25  | 0.2215 (4)   | 0.2209 (5)  | −0.1307 (4)   | 0.0499 (14)  | 0.752 (6) |
| C24  | 0.2782 (5)   | 0.1285 (5)  | −0.1196 (5)   | 0.0792 (18)  | 0.752 (6) |
| F1b  | 0.3785 (12)  | 0.0051 (14) | −0.1200 (11)  | 0.089 (5)    | 0.12      |
| C21b | 0.3128 (17)  | 0.0845 (19) | −0.1568 (13)  | 0.045 (6)    | 0.12      |
| C26b | 0.2965 (19)  | 0.113 (2)   | −0.2477 (14)  | 0.070 (9)    | 0.12      |
| C25b | 0.2352 (19)  | 0.1999 (18) | −0.2841 (15)  | 0.040 (5)    | 0.12      |
| C24b | 0.195 (2)    | 0.255 (2)   | −0.2299 (17)  | 0.039 (6)    | 0.12      |
| C23b | 0.209 (2)    | 0.223 (3)   | −0.1397 (18)  | 0.027 (6)    | 0.12      |
| C22b | 0.2729 (19)  | 0.138 (2)   | −0.1014 (15)  | 0.027 (4)    | 0.12      |
| H21a | 0.1987 (4)   | 0.2477 (5)  | −0.0735 (4)   | 0.0599 (17)* | 0.128 (6) |
| H20a | 0.2997 (5)   | 0.0798 (5)  | −0.0549 (5)   | 0.095 (2)*   | 0.128 (6) |
| H19a | 0.3536 (4)   | 0.0250 (4)  | −0.1846 (6)   | 0.131 (3)*   | 0.128 (6) |
| H23a | 0.2020 (3)   | 0.2970 (5)  | −0.3457 (3)   | 0.0874 (17)* | 0.128 (6) |
| H22a | 0.1460 (3)   | 0.3526 (4)  | −0.2209 (3)   | 0.0622 (12)* | 0.128 (6) |
| H20b | 0.171 (2)    | 0.264 (3)   | −0.0994 (18)  | 0.033 (7)*   | 0.12      |
| H19b | 0.2911 (19)  | 0.115 (2)   | −0.0282 (15)  | 0.032 (5)*   | 0.12      |
| H23b | 0.3309 (19)  | 0.068 (2)   | −0.2896 (14)  | 0.084 (11)*  | 0.12      |
| H22b | 0.2188 (19)  | 0.2248 (18) | −0.3569 (15)  | 0.048 (7)*   | 0.12      |
| H21b | 0.149 (2)    | 0.328 (2)   | −0.2589 (17)  | 0.047 (7)*   | 0.12      |
| H23  | 0.1460 (3)   | 0.3526 (4)  | −0.2209 (3)   | 0.0622 (12)* | 0.752 (6) |
| H22  | 0.1987 (4)   | 0.2477 (5)  | −0.0735 (4)   | 0.0599 (17)* | 0.752 (6) |
| H21  | 0.2997 (5)   | 0.0798 (5)  | −0.0549 (5)   | 0.095 (2)*   | 0.752 (6) |
| H20  | 0.3536 (4)   | 0.0250 (4)  | −0.1846 (6)   | 0.131 (3)*   | 0.752 (6) |
| H19  | 0.3044 (4)   | 0.1331 (6)  | −0.3298 (5)   | 0.123 (3)*   | 0.752 (6) |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| Fe1 | 0.02935 (14) | 0.02685 (14) | 0.02502 (13) | −0.00080 (10) | 0.01024 (10) | −0.00532 (9) |

|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| S2   | 0.0391 (3)  | 0.0445 (3)  | 0.0611 (4)  | 0.0077 (2)   | 0.0222 (3)  | 0.0054 (3)   |
| S1   | 0.0936 (6)  | 0.0617 (4)  | 0.0436 (3)  | −0.0418 (4)  | 0.0391 (4)  | −0.0236 (3)  |
| N4   | 0.0274 (7)  | 0.0211 (7)  | 0.0247 (7)  | 0.0029 (5)   | 0.0082 (5)  | −0.0004 (5)  |
| N5   | 0.0315 (7)  | 0.0230 (7)  | 0.0220 (6)  | 0.0037 (6)   | 0.0075 (6)  | −0.0006 (5)  |
| N3   | 0.0303 (7)  | 0.0363 (8)  | 0.0223 (7)  | 0.0031 (6)   | 0.0097 (6)  | −0.0014 (6)  |
| N6   | 0.0306 (7)  | 0.0247 (7)  | 0.0233 (7)  | 0.0016 (6)   | 0.0079 (6)  | 0.0018 (6)   |
| N1   | 0.0452 (9)  | 0.0307 (8)  | 0.0385 (9)  | −0.0057 (7)  | 0.0187 (8)  | −0.0052 (7)  |
| N2   | 0.0431 (10) | 0.0460 (11) | 0.0372 (9)  | 0.0012 (8)   | 0.0103 (8)  | 0.0002 (8)   |
| C19  | 0.0316 (9)  | 0.0221 (8)  | 0.0238 (7)  | 0.0003 (7)   | 0.0087 (7)  | −0.0019 (6)  |
| C13  | 0.0267 (8)  | 0.0227 (8)  | 0.0259 (8)  | 0.0017 (6)   | 0.0084 (6)  | 0.0001 (6)   |
| C18  | 0.0385 (10) | 0.0252 (9)  | 0.0329 (9)  | −0.0037 (7)  | 0.0111 (8)  | −0.0040 (7)  |
| C7   | 0.0292 (8)  | 0.0369 (10) | 0.0201 (7)  | −0.0037 (7)  | 0.0078 (6)  | −0.0014 (7)  |
| C9   | 0.0428 (10) | 0.0217 (8)  | 0.0267 (8)  | 0.0042 (7)   | 0.0094 (8)  | 0.0011 (7)   |
| C12  | 0.0272 (9)  | 0.0300 (9)  | 0.0335 (9)  | −0.0014 (7)  | 0.0075 (7)  | 0.0006 (7)   |
| C14  | 0.0271 (8)  | 0.0237 (8)  | 0.0365 (9)  | 0.0036 (7)   | 0.0097 (7)  | −0.0024 (7)  |
| C20  | 0.0317 (9)  | 0.0214 (8)  | 0.0346 (9)  | 0.0033 (7)   | 0.0086 (7)  | −0.0047 (7)  |
| C10  | 0.0497 (12) | 0.0241 (9)  | 0.0320 (9)  | −0.0050 (8)  | 0.0115 (8)  | −0.0013 (7)  |
| C15  | 0.0320 (9)  | 0.0357 (10) | 0.0301 (9)  | 0.0039 (8)   | 0.0056 (7)  | 0.0040 (8)   |
| C8   | 0.0366 (10) | 0.0354 (10) | 0.0250 (8)  | 0.0047 (8)   | 0.0096 (7)  | 0.0076 (7)   |
| C17  | 0.0385 (11) | 0.0379 (11) | 0.0360 (10) | −0.0115 (9)  | 0.0102 (8)  | −0.0085 (8)  |
| C11  | 0.0376 (10) | 0.0317 (10) | 0.0339 (9)  | −0.0075 (8)  | 0.0086 (8)  | −0.0011 (8)  |
| C2   | 0.0323 (9)  | 0.0367 (10) | 0.0336 (9)  | 0.0040 (8)   | 0.0111 (8)  | 0.0022 (8)   |
| C16  | 0.0329 (10) | 0.0434 (12) | 0.0338 (10) | −0.0038 (9)  | 0.0045 (8)  | −0.0007 (8)  |
| C1   | 0.0457 (11) | 0.0331 (10) | 0.0311 (9)  | −0.0131 (8)  | 0.0157 (8)  | −0.0086 (8)  |
| C3   | 0.0375 (10) | 0.0542 (13) | 0.0322 (10) | 0.0125 (9)   | 0.0129 (8)  | −0.0029 (9)  |
| C6   | 0.0405 (11) | 0.0627 (14) | 0.0255 (9)  | −0.0040 (10) | 0.0138 (8)  | 0.0048 (9)   |
| C4   | 0.0408 (12) | 0.0818 (18) | 0.0391 (11) | 0.0111 (12)  | 0.0199 (9)  | −0.0105 (12) |
| C5   | 0.0425 (12) | 0.095 (2)   | 0.0318 (10) | −0.0001 (13) | 0.0216 (9)  | −0.0037 (12) |
| C24a | 0.050 (3)   | 0.060 (3)   | 0.046 (2)   | −0.0164 (19) | 0.0251 (19) | −0.0175 (18) |
| C25a | 0.0383 (16) | 0.062 (2)   | 0.059 (2)   | −0.0112 (15) | 0.0216 (15) | −0.0189 (19) |
| C23a | 0.064 (3)   | 0.058 (3)   | 0.111 (5)   | −0.015 (2)   | 0.025 (3)   | −0.014 (3)   |
| C26a | 0.067 (2)   | 0.113 (4)   | 0.0467 (19) | −0.049 (3)   | 0.0305 (17) | −0.035 (2)   |
| F1   | 0.131 (3)   | 0.240 (5)   | 0.0501 (16) | −0.100 (3)   | 0.0220 (16) | 0.010 (2)    |
| C21a | 0.094 (4)   | 0.114 (5)   | 0.139 (5)   | −0.059 (4)   | 0.091 (4)   | −0.093 (4)   |
| C22a | 0.077 (4)   | 0.060 (3)   | 0.215 (9)   | −0.017 (2)   | 0.082 (5)   | −0.055 (4)   |
| C23  | 0.077 (4)   | 0.060 (3)   | 0.215 (9)   | −0.017 (2)   | 0.082 (5)   | −0.055 (4)   |
| C22  | 0.094 (4)   | 0.114 (5)   | 0.139 (5)   | −0.059 (4)   | 0.091 (4)   | −0.093 (4)   |
| F1a  | 0.102 (13)  | 0.097 (12)  | 0.080 (10)  | −0.038 (9)   | 0.060 (9)   | −0.052 (9)   |
| C21  | 0.067 (2)   | 0.113 (4)   | 0.0467 (19) | −0.049 (3)   | 0.0305 (17) | −0.035 (2)   |
| C26  | 0.0383 (16) | 0.062 (2)   | 0.059 (2)   | −0.0112 (15) | 0.0216 (15) | −0.0189 (19) |
| C25  | 0.050 (3)   | 0.060 (3)   | 0.046 (2)   | −0.0164 (19) | 0.0251 (19) | −0.0175 (18) |
| C24  | 0.064 (3)   | 0.058 (3)   | 0.111 (5)   | −0.015 (2)   | 0.025 (3)   | −0.014 (3)   |
| F1b  | 0.106 (10)  | 0.096 (10)  | 0.073 (8)   | 0.058 (7)    | 0.042 (6)   | 0.019 (6)    |
| C21b | 0.051 (10)  | 0.050 (10)  | 0.037 (6)   | 0.011 (6)    | 0.019 (4)   | −0.004 (4)   |
| C26b | 0.099 (16)  | 0.074 (13)  | 0.046 (7)   | 0.042 (8)    | 0.035 (5)   | 0.009 (5)    |
| C25b | 0.050 (10)  | 0.040 (10)  | 0.032 (7)   | 0.002 (6)    | 0.016 (5)   | −0.005 (5)   |
| C24b | 0.044 (10)  | 0.042 (10)  | 0.031 (8)   | −0.002 (6)   | 0.013 (5)   | −0.006 (5)   |
| C23b | 0.025 (9)   | 0.028 (10)  | 0.028 (8)   | −0.007 (5)   | 0.007 (4)   | −0.010 (5)   |

|      |           |           |           |            |           |            |
|------|-----------|-----------|-----------|------------|-----------|------------|
| C22b | 0.025 (7) | 0.030 (8) | 0.024 (6) | −0.006 (5) | 0.005 (4) | −0.008 (4) |
|------|-----------|-----------|-----------|------------|-----------|------------|

*Geometric parameters (Å, °)*

|         |             |           |            |
|---------|-------------|-----------|------------|
| Fe1—N4  | 2.2231 (15) | C16—H8    | 1.1030     |
| Fe1—N5  | 2.1785 (15) | C3—H1     | 1.1030     |
| Fe1—N3  | 2.1726 (15) | C3—C4     | 1.383 (3)  |
| Fe1—N6  | 2.1733 (16) | C6—H4     | 1.1030     |
| Fe1—N1  | 2.0943 (18) | C6—C5     | 1.387 (3)  |
| Fe1—N2  | 2.054 (2)   | C4—H2     | 1.1030     |
| S2—C2   | 1.620 (2)   | C4—C5     | 1.387 (4)  |
| S1—C1   | 1.614 (2)   | C5—H3     | 1.1030     |
| N4—C14  | 1.475 (2)   | C24a—C25a | 1.371 (5)  |
| N4—C20  | 1.479 (2)   | C24a—C23a | 1.376 (6)  |
| N4—C8   | 1.482 (2)   | C24a—H21a | 1.1030     |
| N5—C13  | 1.343 (2)   | C25a—C26a | 1.358 (5)  |
| N5—C9   | 1.345 (2)   | C25a—H22a | 1.1030     |
| N3—C7   | 1.338 (2)   | C23a—C22a | 1.392 (8)  |
| N3—C3   | 1.343 (3)   | C23a—H20a | 1.1030     |
| N6—C19  | 1.339 (2)   | C26a—C21a | 1.367 (8)  |
| N6—C15  | 1.340 (2)   | C26a—H23a | 1.1030     |
| N1—C1   | 1.162 (3)   | F1—C21    | 1.275 (6)  |
| N2—C2   | 1.168 (3)   | C21a—C22a | 1.364 (9)  |
| C19—C18 | 1.395 (2)   | C21a—F1a  | 1.357 (13) |
| C19—C20 | 1.506 (3)   | C22a—H19a | 1.1030     |
| C13—C12 | 1.392 (2)   | C23—C22   | 1.364 (9)  |
| C13—C14 | 1.504 (2)   | C23—C24   | 1.392 (8)  |
| C18—H10 | 1.1030      | C23—H20   | 1.1030     |
| C18—C17 | 1.385 (3)   | C22—C21   | 1.367 (8)  |
| C7—C8   | 1.508 (3)   | C22—H19   | 1.1030     |
| C7—C6   | 1.390 (3)   | C21—C26   | 1.358 (5)  |
| C9—H13  | 1.1030      | C26—C25   | 1.371 (5)  |
| C9—C10  | 1.388 (3)   | C26—H23   | 1.1030     |
| C12—H16 | 1.1030      | C25—C24   | 1.376 (6)  |
| C12—C11 | 1.387 (3)   | C25—H22   | 1.1030     |
| C14—H18 | 1.1110      | C24—H21   | 1.1030     |
| C14—H17 | 1.1110      | F1b—C21b  | 1.351 (15) |
| C20—H11 | 1.1110      | C21b—C26b | 1.380 (13) |
| C20—H12 | 1.1110      | C21b—C22b | 1.368 (13) |
| C10—H14 | 1.1030      | C26b—C25b | 1.380 (13) |
| C10—C11 | 1.389 (3)   | C26b—H23b | 1.1030     |
| C15—H7  | 1.1030      | C25b—C24b | 1.372 (13) |
| C15—C16 | 1.388 (3)   | C25b—H22b | 1.1030     |
| C8—H5   | 1.1110      | C24b—C23b | 1.386 (14) |
| C8—H6   | 1.1110      | C24b—H21b | 1.1030     |
| C17—H9  | 1.1030      | C23b—C22b | 1.388 (14) |
| C17—C16 | 1.392 (3)   | C23b—H20b | 1.1030     |
| C11—H15 | 1.1030      | C22b—H19b | 1.1030     |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N5—Fe1—N4   | 76.52 (5)   | C16—C17—H9     | 120.48 (12) |
| N3—Fe1—N4   | 78.63 (6)   | C10—C11—C12    | 118.84 (18) |
| N3—Fe1—N5   | 89.76 (6)   | H15—C11—C12    | 120.58 (12) |
| N6—Fe1—N4   | 76.23 (5)   | H15—C11—C10    | 120.58 (12) |
| N6—Fe1—N5   | 152.74 (6)  | N2—C2—S2       | 179.7 (2)   |
| N6—Fe1—N3   | 84.30 (6)   | C17—C16—C15    | 118.60 (18) |
| N1—Fe1—N4   | 91.47 (6)   | H8—C16—C15     | 120.70 (12) |
| N1—Fe1—N5   | 91.73 (6)   | H8—C16—C17     | 120.70 (12) |
| N1—Fe1—N3   | 169.36 (7)  | N1—C1—S1       | 178.5 (2)   |
| N1—Fe1—N6   | 89.60 (6)   | H1—C3—N3       | 118.60 (12) |
| N2—Fe1—N4   | 172.28 (7)  | C4—C3—N3       | 122.8 (2)   |
| N2—Fe1—N5   | 100.15 (7)  | C4—C3—H1       | 118.60 (15) |
| N2—Fe1—N3   | 94.49 (7)   | H4—C6—C7       | 120.49 (13) |
| N2—Fe1—N6   | 106.82 (7)  | C5—C6—C7       | 119.0 (2)   |
| N2—Fe1—N1   | 95.61 (7)   | C5—C6—H4       | 120.49 (14) |
| C14—N4—Fe1  | 105.77 (11) | H2—C4—C3       | 120.75 (15) |
| C20—N4—Fe1  | 105.78 (10) | C5—C4—C3       | 118.5 (2)   |
| C20—N4—C14  | 110.67 (14) | C5—C4—H2       | 120.75 (13) |
| C8—N4—Fe1   | 111.11 (11) | C4—C5—C6       | 119.0 (2)   |
| C8—N4—C14   | 111.27 (14) | H3—C5—C6       | 120.49 (14) |
| C8—N4—C20   | 111.94 (15) | H3—C5—C4       | 120.49 (13) |
| C13—N5—Fe1  | 114.73 (12) | C23a—C24a—C25a | 120.9 (5)   |
| C9—N5—Fe1   | 126.86 (13) | H21a—C24a—C25a | 119.5 (3)   |
| C9—N5—C13   | 118.41 (16) | H21a—C24a—C23a | 119.5 (3)   |
| C7—N3—Fe1   | 116.21 (12) | C26a—C25a—C24a | 119.1 (4)   |
| C3—N3—Fe1   | 125.03 (14) | H22a—C25a—C24a | 120.4 (3)   |
| C3—N3—C7    | 118.65 (17) | H22a—C25a—C26a | 120.4 (3)   |
| C19—N6—Fe1  | 115.18 (12) | C22a—C23a—C24a | 118.2 (6)   |
| C15—N6—Fe1  | 126.06 (13) | H20a—C23a—C24a | 120.9 (3)   |
| C15—N6—C19  | 118.75 (16) | H20a—C23a—C22a | 120.9 (4)   |
| C1—N1—Fe1   | 169.67 (18) | C21a—C26a—C25a | 121.9 (5)   |
| C2—N2—Fe1   | 177.66 (18) | H23a—C26a—C25a | 119.0 (3)   |
| C18—C19—N6  | 122.32 (17) | H23a—C26a—C21a | 119.0 (3)   |
| C20—C19—N6  | 115.96 (15) | C22a—C21a—C26a | 118.7 (4)   |
| C20—C19—C18 | 121.65 (16) | F1a—C21a—C26a  | 104.4 (10)  |
| C12—C13—N5  | 122.15 (16) | F1a—C21a—C22a  | 136.9 (10)  |
| C14—C13—N5  | 116.05 (15) | C21a—C22a—C23a | 121.2 (5)   |
| C14—C13—C12 | 121.77 (16) | H19a—C22a—C23a | 119.4 (4)   |
| H10—C18—C19 | 120.65 (11) | H19a—C22a—C21a | 119.4 (3)   |
| C17—C18—C19 | 118.70 (18) | C24—C23—C22    | 121.2 (5)   |
| C17—C18—H10 | 120.65 (12) | H20—C23—C22    | 119.4 (3)   |
| C8—C7—N3    | 118.45 (16) | H20—C23—C24    | 119.4 (4)   |
| C6—C7—N3    | 122.03 (19) | C21—C22—C23    | 118.7 (4)   |
| C6—C7—C8    | 119.48 (18) | H19—C22—C23    | 120.7 (3)   |
| H13—C9—N5   | 118.63 (11) | H19—C22—C21    | 120.7 (3)   |
| C10—C9—N5   | 122.74 (18) | C22—C21—F1     | 115.3 (5)   |
| C10—C9—H13  | 118.63 (11) | C26—C21—F1     | 122.8 (6)   |



|                |              |                     |              |
|----------------|--------------|---------------------|--------------|
| H16—C12—C13    | 120.41 (11)  | C26—C21—C22         | 121.9 (5)    |
| C11—C12—C13    | 119.18 (18)  | C25—C26—C21         | 119.1 (4)    |
| C11—C12—H16    | 120.41 (12)  | H23—C26—C21         | 120.4 (3)    |
| C13—C14—N4     | 111.15 (14)  | H23—C26—C25         | 120.4 (3)    |
| H18—C14—N4     | 109.40 (9)   | C24—C25—C26         | 120.9 (5)    |
| H18—C14—C13    | 109.40 (10)  | H22—C25—C26         | 119.5 (3)    |
| H17—C14—N4     | 109.40 (9)   | H22—C25—C24         | 119.5 (3)    |
| H17—C14—C13    | 109.40 (10)  | C25—C24—C23         | 118.2 (6)    |
| H17—C14—H18    | 108.0        | H21—C24—C23         | 120.9 (4)    |
| C19—C20—N4     | 111.02 (14)  | H21—C24—C25         | 120.9 (3)    |
| H11—C20—N4     | 109.43 (9)   | C26b—C21b—F1b       | 116.3 (16)   |
| H11—C20—C19    | 109.43 (10)  | C22b—C21b—F1b       | 119.5 (16)   |
| H12—C20—N4     | 109.43 (10)  | C22b—C21b—C26b      | 123.9 (16)   |
| H12—C20—C19    | 109.43 (10)  | C25b—C26b—C21b      | 117.1 (16)   |
| H12—C20—H11    | 108.0        | H23b—C26b—C21b      | 121.4 (11)   |
| H14—C10—C9     | 120.66 (11)  | H23b—C26b—C25b      | 121.4 (11)   |
| C11—C10—C9     | 118.68 (18)  | C24b—C25b—C26b      | 120.1 (16)   |
| C11—C10—H14    | 120.66 (12)  | H22b—C25b—C26b      | 120.0 (11)   |
| H7—C15—N6      | 118.73 (11)  | H22b—C25b—C24b      | 120.0 (11)   |
| C16—C15—N6     | 122.54 (19)  | C23b—C24b—C25b      | 122.0 (16)   |
| C16—C15—H7     | 118.73 (12)  | H21b—C24b—C25b      | 119.0 (11)   |
| C7—C8—N4       | 115.11 (15)  | H21b—C24b—C23b      | 119.0 (11)   |
| H5—C8—N4       | 108.49 (10)  | C22b—C23b—C24b      | 118.3 (17)   |
| H5—C8—C7       | 108.49 (10)  | H20b—C23b—C24b      | 120.9 (12)   |
| H6—C8—N4       | 108.49 (10)  | H20b—C23b—C22b      | 120.9 (12)   |
| H6—C8—C7       | 108.49 (11)  | C23b—C22b—C21b      | 118.4 (16)   |
| H6—C8—H5       | 107.5        | H19b—C22b—C21b      | 120.8 (11)   |
| H9—C17—C18     | 120.48 (12)  | H19b—C22b—C23b      | 120.8 (12)   |
| C16—C17—C18    | 119.04 (18)  |                     |              |
| Fe1—N4—C14—C13 | −42.59 (11)  | C7—C8—N4—C20        | 111.48 (17)  |
| Fe1—N4—C20—C19 | 42.37 (11)   | C7—C6—C5—C4         | −0.2 (3)     |
| Fe1—N4—C8—C7   | −6.54 (12)   | C9—N5—C13—C12       | −0.32 (19)   |
| Fe1—N5—C13—C12 | 179.33 (12)  | C9—N5—C13—C14       | 177.37 (16)  |
| Fe1—N5—C13—C14 | −2.98 (13)   | C9—C10—C11—C12      | 0.1 (2)      |
| Fe1—N5—C9—C10  | −179.91 (15) | C14—C13—C12—C11     | −176.76 (18) |
| Fe1—N3—C7—C8   | −5.63 (15)   | C20—C19—N6—C15      | −178.12 (16) |
| Fe1—N3—C7—C6   | 176.49 (14)  | C20—C19—C18—C17     | 176.02 (18)  |
| Fe1—N3—C3—C4   | −176.20 (19) | C8—C7—N3—C3         | 178.17 (18)  |
| Fe1—N6—C19—C18 | 177.88 (12)  | C8—C7—C6—C5         | −177.9 (2)   |
| Fe1—N6—C19—C20 | 0.87 (13)    | C3—N3—C7—C6         | 0.3 (2)      |
| Fe1—N6—C15—C16 | −176.88 (16) | C3—C4—C5—C6         | 0.2 (3)      |
| N4—C14—C13—N5  | 32.04 (17)   | C24a—C25a—C26a—C21a | 1.3 (5)      |
| N4—C14—C13—C12 | −150.26 (15) | C24a—C23a—C22a—C21a | −0.6 (6)     |
| N4—C20—C19—N6  | −30.45 (17)  | C25a—C24a—C23a—C22a | 1.5 (7)      |
| N4—C20—C19—C18 | 152.52 (15)  | C25a—C26a—C21a—C22a | −0.4 (5)     |
| N4—C8—C7—N3    | 8.3 (2)      | C25a—C26a—C21a—F1a  | 176.7 (7)    |
| N4—C8—C7—C6    | −173.76 (17) | C23a—C24a—C25a—C26a | −1.8 (5)     |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N5—C13—C12—C11  | 0.8 (2)      | C23a—C22a—C21a—C26a | −0.0 (6)    |
| N5—C9—C10—C11   | 0.4 (2)      | C23a—C22a—C21a—F1a  | −175.8 (10) |
| N3—C7—C6—C5     | −0.0 (2)     | F1—C21—C22—C23      | −178.0 (4)  |
| N3—C3—C4—C5     | 0.1 (3)      | F1—C21—C26—C25      | 178.8 (5)   |
| N6—C19—C18—C17  | −0.8 (2)     | C23—C22—C21—C26     | −0.4 (5)    |
| N6—C15—C16—C17  | −0.9 (2)     | C23—C24—C25—C26     | 1.5 (7)     |
| C19—N6—C15—C16  | 1.99 (19)    | C22—C23—C24—C25     | −0.6 (6)    |
| C19—C18—C17—C16 | 1.9 (2)      | C22—C21—C26—C25     | 1.3 (5)     |
| C19—C20—N4—C14  | 156.48 (15)  | C21—C22—C23—C24     | −0.0 (5)    |
| C19—C20—N4—C8   | −78.78 (16)  | C21—C26—C25—C24     | −1.8 (5)    |
| C13—N5—C9—C10   | −0.31 (19)   | F1b—C21b—C26b—C25b  | −174.9 (17) |
| C13—C12—C11—C10 | −0.6 (2)     | F1b—C21b—C22b—C23b  | 177 (2)     |
| C13—C14—N4—C20  | −156.70 (15) | C21b—C26b—C25b—C24b | 1 (3)       |
| C13—C14—N4—C8   | 78.17 (16)   | C21b—C22b—C23b—C24b | −5 (3)      |
| C18—C19—N6—C15  | −1.1 (2)     | C26b—C21b—C22b—C23b | 3 (3)       |
| C18—C17—C16—C15 | −1.1 (2)     | C26b—C25b—C24b—C23b | −4 (3)      |
| C7—N3—C3—C4     | −0.4 (2)     | C25b—C26b—C21b—C22b | −1 (3)      |
| C7—C8—N4—C14    | −124.11 (16) | C25b—C24b—C23b—C22b | 6 (4)       |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_165K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.2066$  (3) Å

$b = 12.0024$  (2) Å

$c = 15.4362$  (3) Å

$\beta = 110.469$  (2)°

$V = 2639.47$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.405$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 58161 reflections

$\theta = 2.1$ – $32.6^\circ$

$\mu = 0.76$  mm<sup>−1</sup>

$T = 165$  K

Block, yellow

$0.31 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.668$ ,  $T_{\max} = 1.000$

76127 measured reflections

6545 independent reflections

6148 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 17$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.094$

$S = 1.05$

6545 reflections

408 parameters

155 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 1.8169P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.00$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.51$  e Å<sup>−3</sup>

*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 | 0.344491 (15) | 0.331216 (18) | 0.196423 (14) | 0.02604 (7)                      |           |
| S2  | 0.52861 (3)   | 0.00268 (4)   | 0.32500 (4)   | 0.04644 (13)                     |           |
| S1  | 0.23444 (11)  | 0.34397 (12)  | 0.45595 (9)   | 0.0640 (2)                       |           |
| N4  | 0.25876 (9)   | 0.47253 (10)  | 0.11581 (8)   | 0.0249 (2)                       |           |
| N5  | 0.21422 (9)   | 0.25151 (11)  | 0.10848 (8)   | 0.0264 (3)                       |           |
| N6  | 0.43450 (9)   | 0.47702 (11)  | 0.24479 (8)   | 0.0267 (3)                       |           |
| N3  | 0.39186 (9)   | 0.33993 (12)  | 0.07831 (9)   | 0.0302 (3)                       |           |
| N1  | 0.29108 (11)  | 0.35459 (12)  | 0.30366 (11)  | 0.0373 (3)                       |           |
| N2  | 0.42373 (12)  | 0.19342 (14)  | 0.25312 (11)  | 0.0428 (4)                       |           |
| C19 | 0.39038 (11)  | 0.57547 (12)  | 0.22649 (10)  | 0.0264 (3)                       |           |
| C13 | 0.14216 (11)  | 0.32170 (12)  | 0.07088 (10)  | 0.0260 (3)                       |           |
| C18 | 0.43740 (12)  | 0.67597 (13)  | 0.25537 (11)  | 0.0332 (3)                       |           |
| H10 | 0.39936 (12)  | 0.75586 (13)  | 0.23845 (11)  | 0.0398 (4)*                      |           |
| C12 | 0.05470 (11)  | 0.28485 (14)  | 0.01254 (11)  | 0.0319 (3)                       |           |
| H16 | −0.00295 (11) | 0.34468 (14)  | −0.01785 (11) | 0.0383 (4)*                      |           |
| C8  | 0.27601 (12)  | 0.48976 (15)  | 0.02815 (11)  | 0.0335 (3)                       |           |
| H5  | 0.21017 (12)  | 0.47114 (15)  | −0.03028 (11) | 0.0402 (4)*                      |           |
| H6  | 0.29270 (12)  | 0.57912 (15)  | 0.02312 (11)  | 0.0402 (4)*                      |           |
| C7  | 0.35389 (11)  | 0.42049 (14)  | 0.01683 (10)  | 0.0298 (3)                       |           |
| C14 | 0.16014 (10)  | 0.44223 (13)  | 0.09846 (11)  | 0.0302 (3)                       |           |
| H18 | 0.14337 (10)  | 0.45742 (13)  | 0.16198 (11)  | 0.0362 (4)*                      |           |
| H17 | 0.11341 (10)  | 0.49575 (13)  | 0.04252 (11)  | 0.0362 (4)*                      |           |
| C9  | 0.20080 (12)  | 0.14252 (13)  | 0.08883 (11)  | 0.0312 (3)                       |           |
| H13 | 0.26010 (12)  | 0.08480 (13)  | 0.11909 (11)  | 0.0374 (4)*                      |           |
| C20 | 0.28583 (11)  | 0.57089 (13)  | 0.17700 (11)  | 0.0309 (3)                       |           |
| H11 | 0.26239 (11)  | 0.64781 (13)  | 0.13524 (11)  | 0.0371 (4)*                      |           |
| H12 | 0.25012 (11)  | 0.56743 (13)  | 0.22863 (11)  | 0.0371 (4)*                      |           |
| C10 | 0.11530 (13)  | 0.09939 (14)  | 0.03197 (11)  | 0.0363 (4)                       |           |
| H14 | 0.10676 (13)  | 0.00916 (14)  | 0.01803 (11)  | 0.0435 (4)*                      |           |
| C15 | 0.52727 (12)  | 0.47566 (15)  | 0.29103 (11)  | 0.0345 (3)                       |           |
| H7  | 0.56401 (12)  | 0.39477 (15)  | 0.30395 (11)  | 0.0414 (4)*                      |           |
| C2  | 0.46756 (11)  | 0.11340 (14)  | 0.28295 (11)  | 0.0336 (3)                       |           |
| C11 | 0.04088 (13)  | 0.17196 (14)  | −0.00695 (12) | 0.0361 (4)                       |           |
| H15 | −0.02793 (13) | 0.14055 (14)  | −0.05248 (12) | 0.0434 (4)*                      |           |
| C1  | 0.26722 (13)  | 0.35159 (14)  | 0.36725 (12)  | 0.0359 (4)                       |           |
| C17 | 0.53270 (13)  | 0.67377 (15)  | 0.30570 (12)  | 0.0387 (4)                       |           |
| H9  | 0.57135 (13)  | 0.75178 (15)  | 0.33144 (12)  | 0.0464 (5)*                      |           |
| C16 | 0.57900 (12)  | 0.57178 (16)  | 0.32333 (12)  | 0.0389 (4)                       |           |
| H8  | 0.65513 (12)  | 0.56742 (16)  | 0.36212 (12)  | 0.0467 (4)*                      |           |
| C3  | 0.45930 (13)  | 0.27570 (18)  | 0.06677 (13)  | 0.0427 (4)                       |           |
| H1  | 0.49087 (13)  | 0.20963 (18)  | 0.11787 (13)  | 0.0512 (5)*                      |           |
| C25 | 0.2213 (4)    | 0.2209 (5)    | −0.1312 (3)   | 0.0524 (12)                      | 0.733 (5) |
| H22 | 0.1989 (4)    | 0.2471 (5)    | −0.0736 (3)   | 0.0629 (15)*                     | 0.733 (5) |
| C6  | 0.38189 (13)  | 0.43908 (19)  | −0.05867 (12) | 0.0445 (4)                       |           |
| H4  | 0.34919 (13)  | 0.50591 (19)  | −0.10860 (12) | 0.0533 (5)*                      |           |

|      |              |             |               |              |           |
|------|--------------|-------------|---------------|--------------|-----------|
| C26  | 0.1922 (3)   | 0.2805 (3)  | −0.2117 (3)   | 0.0538 (9)   | 0.733 (5) |
| H23  | 0.1450 (3)   | 0.3525 (3)  | −0.2198 (3)   | 0.0646 (10)* | 0.733 (5) |
| C4   | 0.49079 (15) | 0.2890 (2)  | −0.00654 (15) | 0.0559 (6)   |           |
| H2   | 0.54597 (15) | 0.2343 (2)  | −0.01409 (15) | 0.0671 (7)*  |           |
| C5   | 0.45135 (15) | 0.3726 (2)  | −0.07029 (14) | 0.0575 (6)   |           |
| H3   | 0.47497 (15) | 0.3859 (2)  | −0.12941 (14) | 0.0690 (7)*  |           |
| C24  | 0.2779 (4)   | 0.1289 (5)  | −0.1206 (4)   | 0.0822 (15)  | 0.733 (5) |
| H21  | 0.2999 (4)   | 0.0800 (5)  | −0.0561 (4)   | 0.0987 (18)* | 0.733 (5) |
| C21  | 0.2220 (3)   | 0.2501 (4)  | −0.2814 (2)   | 0.0750 (12)  | 0.733 (5) |
| F1   | 0.1971 (3)   | 0.3011 (4)  | −0.35826 (18) | 0.152 (2)    | 0.733 (5) |
| C22  | 0.2792 (4)   | 0.1599 (5)  | −0.2742 (4)   | 0.103 (2)    | 0.733 (5) |
| H19  | 0.3027 (4)   | 0.1366 (5)  | −0.3317 (4)   | 0.124 (2)*   | 0.733 (5) |
| C23  | 0.3066 (3)   | 0.0992 (4)  | −0.1941 (6)   | 0.112 (2)    | 0.733 (5) |
| H20  | 0.3521 (3)   | 0.0257 (4)  | −0.1875 (6)   | 0.135 (3)*   | 0.733 (5) |
| F1a  | 0.2890 (10)  | 0.1513 (11) | −0.3581 (8)   | 0.092 (5)    | 0.147 (5) |
| C21a | 0.2792 (4)   | 0.1599 (5)  | −0.2742 (4)   | 0.103 (2)    | 0.147 (5) |
| C26a | 0.2220 (3)   | 0.2501 (4)  | −0.2814 (2)   | 0.0750 (12)  | 0.147 (5) |
| H23a | 0.1995 (3)   | 0.2995 (4)  | −0.3458 (2)   | 0.0899 (14)* | 0.147 (5) |
| C25a | 0.1922 (3)   | 0.2805 (3)  | −0.2117 (3)   | 0.0538 (9)   | 0.147 (5) |
| H22a | 0.1450 (3)   | 0.3525 (3)  | −0.2198 (3)   | 0.0646 (10)* | 0.147 (5) |
| C24a | 0.2213 (4)   | 0.2209 (5)  | −0.1312 (3)   | 0.0524 (12)  | 0.147 (5) |
| H21a | 0.1989 (4)   | 0.2471 (5)  | −0.0736 (3)   | 0.0629 (15)* | 0.147 (5) |
| C23a | 0.2779 (4)   | 0.1289 (5)  | −0.1206 (4)   | 0.0822 (15)  | 0.147 (5) |
| H20a | 0.2999 (4)   | 0.0800 (5)  | −0.0561 (4)   | 0.0987 (18)* | 0.147 (5) |
| C22a | 0.3066 (3)   | 0.0992 (4)  | −0.1941 (6)   | 0.112 (2)    | 0.147 (5) |
| H19a | 0.3521 (3)   | 0.0257 (4)  | −0.1875 (6)   | 0.135 (3)*   | 0.147 (5) |
| F1b  | 0.3768 (10)  | 0.0047 (12) | −0.1199 (9)   | 0.080 (4)    | 0.12      |
| C21b | 0.3117 (16)  | 0.0847 (18) | −0.1543 (12)  | 0.053 (6)    | 0.12      |
| C26b | 0.2978 (17)  | 0.1129 (18) | −0.2443 (12)  | 0.074 (8)    | 0.12      |
| H23b | 0.3337 (17)  | 0.0677 (18) | −0.2845 (12)  | 0.089 (10)*  | 0.12      |
| C25b | 0.2375 (16)  | 0.1995 (17) | −0.2827 (13)  | 0.041 (5)    | 0.12      |
| H22b | 0.2230 (16)  | 0.2235 (17) | −0.3553 (13)  | 0.049 (6)*   | 0.12      |
| C24b | 0.1954 (19)  | 0.256 (2)   | −0.2305 (16)  | 0.042 (5)    | 0.12      |
| H21b | 0.1510 (19)  | 0.328 (2)   | −0.2607 (16)  | 0.050 (6)*   | 0.12      |
| C23b | 0.207 (2)    | 0.224 (3)   | −0.1410 (18)  | 0.035 (6)    | 0.12      |
| H20b | 0.167 (2)    | 0.265 (3)   | −0.1023 (18)  | 0.042 (7)*   | 0.12      |
| C22b | 0.270 (2)    | 0.138 (2)   | −0.1014 (14)  | 0.035 (4)    | 0.12      |
| H19b | 0.286 (2)    | 0.115 (2)   | −0.0284 (14)  | 0.042 (5)*   | 0.12      |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1 | 0.02883 (12) | 0.02502 (12) | 0.02396 (11) | 0.00057 (8)  | 0.00883 (8) | −0.00293 (8) |
| S2  | 0.0394 (2)   | 0.0402 (3)   | 0.0629 (3)   | 0.01029 (19) | 0.0220 (2)  | 0.0087 (2)   |
| S1  | 0.0963 (5)   | 0.0653 (4)   | 0.0439 (3)   | −0.0443 (3)  | 0.0415 (3)  | −0.0246 (3)  |
| N4  | 0.0267 (6)   | 0.0214 (6)   | 0.0254 (6)   | 0.0030 (5)   | 0.0077 (5)  | −0.0008 (5)  |
| N5  | 0.0309 (6)   | 0.0231 (6)   | 0.0234 (5)   | 0.0027 (5)   | 0.0072 (5)  | −0.0004 (5)  |
| N6  | 0.0302 (6)   | 0.0238 (6)   | 0.0241 (6)   | 0.0012 (5)   | 0.0070 (5)  | 0.0013 (5)   |

|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| N3   | 0.0300 (6)  | 0.0377 (7)  | 0.0234 (6)  | 0.0036 (5)   | 0.0099 (5)  | −0.0016 (5)  |
| N1   | 0.0467 (8)  | 0.0330 (7)  | 0.0359 (7)  | −0.0065 (6)  | 0.0191 (6)  | −0.0050 (6)  |
| N2   | 0.0434 (8)  | 0.0414 (8)  | 0.0395 (8)  | 0.0059 (7)   | 0.0095 (7)  | 0.0038 (7)   |
| C19  | 0.0313 (7)  | 0.0225 (7)  | 0.0246 (6)  | −0.0001 (6)  | 0.0088 (6)  | −0.0014 (5)  |
| C13  | 0.0272 (7)  | 0.0231 (7)  | 0.0267 (7)  | 0.0014 (5)   | 0.0080 (6)  | 0.0001 (5)   |
| C18  | 0.0380 (9)  | 0.0258 (8)  | 0.0341 (8)  | −0.0038 (6)  | 0.0106 (7)  | −0.0042 (6)  |
| C12  | 0.0273 (7)  | 0.0303 (8)  | 0.0347 (8)  | −0.0016 (6)  | 0.0066 (6)  | 0.0008 (6)   |
| C8   | 0.0370 (8)  | 0.0361 (8)  | 0.0257 (7)  | 0.0051 (7)   | 0.0089 (6)  | 0.0070 (6)   |
| C7   | 0.0291 (7)  | 0.0386 (8)  | 0.0209 (6)  | −0.0034 (6)  | 0.0079 (5)  | −0.0012 (6)  |
| C14  | 0.0262 (7)  | 0.0246 (7)  | 0.0381 (8)  | 0.0039 (6)   | 0.0092 (6)  | −0.0023 (6)  |
| C9   | 0.0408 (9)  | 0.0227 (7)  | 0.0274 (7)  | 0.0026 (6)   | 0.0087 (6)  | 0.0003 (6)   |
| C20  | 0.0317 (7)  | 0.0217 (7)  | 0.0366 (8)  | 0.0035 (6)   | 0.0086 (6)  | −0.0045 (6)  |
| C10  | 0.0475 (10) | 0.0253 (8)  | 0.0330 (8)  | −0.0055 (7)  | 0.0102 (7)  | −0.0007 (6)  |
| C15  | 0.0323 (8)  | 0.0349 (9)  | 0.0315 (7)  | 0.0030 (7)   | 0.0052 (6)  | 0.0033 (6)   |
| C2   | 0.0316 (8)  | 0.0333 (8)  | 0.0353 (8)  | 0.0057 (6)   | 0.0110 (6)  | 0.0046 (7)   |
| C11  | 0.0357 (8)  | 0.0332 (9)  | 0.0355 (8)  | −0.0086 (7)  | 0.0075 (7)  | −0.0020 (7)  |
| C1   | 0.0463 (9)  | 0.0340 (8)  | 0.0301 (8)  | −0.0138 (7)  | 0.0165 (7)  | −0.0083 (6)  |
| C17  | 0.0384 (9)  | 0.0368 (9)  | 0.0378 (9)  | −0.0105 (7)  | 0.0094 (7)  | −0.0078 (7)  |
| C16  | 0.0321 (8)  | 0.0429 (10) | 0.0353 (8)  | −0.0045 (7)  | 0.0037 (7)  | −0.0010 (7)  |
| C3   | 0.0375 (9)  | 0.0566 (11) | 0.0348 (9)  | 0.0132 (8)   | 0.0138 (7)  | −0.0046 (8)  |
| C25  | 0.051 (2)   | 0.063 (3)   | 0.0489 (18) | −0.0194 (16) | 0.0247 (17) | −0.0196 (16) |
| C6   | 0.0415 (9)  | 0.0667 (13) | 0.0271 (8)  | −0.0043 (9)  | 0.0145 (7)  | 0.0056 (8)   |
| C26  | 0.0406 (14) | 0.062 (2)   | 0.063 (2)   | −0.0098 (13) | 0.0237 (14) | −0.0193 (16) |
| C4   | 0.0416 (10) | 0.0895 (17) | 0.0420 (10) | 0.0129 (11)  | 0.0215 (8)  | −0.0117 (11) |
| C5   | 0.0442 (10) | 0.1015 (19) | 0.0343 (9)  | −0.0005 (11) | 0.0229 (8)  | −0.0036 (11) |
| C24  | 0.062 (2)   | 0.061 (2)   | 0.117 (4)   | −0.0148 (18) | 0.024 (3)   | −0.012 (3)   |
| C21  | 0.068 (2)   | 0.115 (4)   | 0.0498 (17) | −0.048 (2)   | 0.0300 (15) | −0.034 (2)   |
| F1   | 0.141 (3)   | 0.254 (5)   | 0.0544 (15) | −0.108 (3)   | 0.0239 (16) | 0.010 (2)    |
| C22  | 0.093 (3)   | 0.115 (4)   | 0.142 (5)   | −0.057 (3)   | 0.091 (3)   | −0.090 (4)   |
| C23  | 0.076 (3)   | 0.056 (2)   | 0.228 (8)   | −0.016 (2)   | 0.082 (4)   | −0.055 (4)   |
| F1a  | 0.105 (10)  | 0.111 (11)  | 0.077 (8)   | −0.025 (8)   | 0.052 (7)   | −0.044 (7)   |
| C21a | 0.093 (3)   | 0.115 (4)   | 0.142 (5)   | −0.057 (3)   | 0.091 (3)   | −0.090 (4)   |
| C26a | 0.068 (2)   | 0.115 (4)   | 0.0498 (17) | −0.048 (2)   | 0.0300 (15) | −0.034 (2)   |
| C25a | 0.0406 (14) | 0.062 (2)   | 0.063 (2)   | −0.0098 (13) | 0.0237 (14) | −0.0193 (16) |
| C24a | 0.051 (2)   | 0.063 (3)   | 0.0489 (18) | −0.0194 (16) | 0.0247 (17) | −0.0196 (16) |
| C23a | 0.062 (2)   | 0.061 (2)   | 0.117 (4)   | −0.0148 (18) | 0.024 (3)   | −0.012 (3)   |
| C22a | 0.076 (3)   | 0.056 (2)   | 0.228 (8)   | −0.016 (2)   | 0.082 (4)   | −0.055 (4)   |
| F1b  | 0.090 (8)   | 0.083 (8)   | 0.075 (7)   | 0.037 (6)    | 0.038 (5)   | 0.010 (5)    |
| C21b | 0.062 (10)  | 0.063 (10)  | 0.041 (6)   | 0.016 (6)    | 0.027 (4)   | −0.000 (4)   |
| C26b | 0.105 (15)  | 0.078 (13)  | 0.052 (7)   | 0.043 (8)    | 0.043 (5)   | 0.011 (5)    |
| C25b | 0.050 (9)   | 0.043 (9)   | 0.036 (7)   | 0.000 (6)    | 0.022 (4)   | −0.005 (5)   |
| C24b | 0.045 (9)   | 0.048 (10)  | 0.036 (8)   | −0.004 (5)   | 0.018 (5)   | −0.008 (5)   |
| C23b | 0.031 (9)   | 0.043 (10)  | 0.033 (8)   | −0.005 (5)   | 0.015 (5)   | −0.010 (5)   |
| C22b | 0.032 (7)   | 0.044 (9)   | 0.030 (6)   | −0.004 (5)   | 0.013 (4)   | −0.008 (4)   |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N4    | 2.2338 (12) | C16—H8      | 1.1030      |
| Fe1—N5    | 2.1912 (13) | C3—H1       | 1.1030      |
| Fe1—N6    | 2.1860 (13) | C3—C4       | 1.383 (3)   |
| Fe1—N3    | 2.1846 (13) | C25—H22     | 1.1030      |
| Fe1—N1    | 2.1021 (15) | C25—C26     | 1.366 (5)   |
| Fe1—N2    | 2.0532 (17) | C25—C24     | 1.374 (6)   |
| S2—C2     | 1.6210 (17) | C6—H4       | 1.1030      |
| S1—C1     | 1.615 (2)   | C6—C5       | 1.385 (3)   |
| N4—C8     | 1.4802 (19) | C26—H23     | 1.1030      |
| N4—C14    | 1.4738 (19) | C26—C21     | 1.356 (4)   |
| N4—C20    | 1.4777 (19) | C4—H2       | 1.1030      |
| N5—C13    | 1.3426 (19) | C4—C5       | 1.387 (4)   |
| N5—C9     | 1.342 (2)   | C5—H3       | 1.1030      |
| N6—C19    | 1.3392 (19) | C24—H21     | 1.1030      |
| N6—C15    | 1.340 (2)   | C24—C23     | 1.397 (8)   |
| N3—C7     | 1.336 (2)   | C21—F1      | 1.269 (5)   |
| N3—C3     | 1.344 (2)   | C21—C22     | 1.369 (7)   |
| N1—C1     | 1.160 (2)   | C22—H19     | 1.1030      |
| N2—C2     | 1.168 (2)   | C22—C23     | 1.368 (8)   |
| C19—C18   | 1.394 (2)   | C23—H20     | 1.1030      |
| C19—C20   | 1.505 (2)   | F1a—C21a    | 1.359 (11)  |
| C13—C12   | 1.392 (2)   | C21a—C26a   | 1.369 (7)   |
| C13—C14   | 1.506 (2)   | C21a—C22a   | 1.368 (8)   |
| C18—H10   | 1.1030      | C26a—H23a   | 1.1030      |
| C18—C17   | 1.385 (2)   | C26a—C25a   | 1.356 (4)   |
| C12—H16   | 1.1030      | C25a—H22a   | 1.1030      |
| C12—C11   | 1.388 (2)   | C25a—C24a   | 1.366 (5)   |
| C8—H5     | 1.1110      | C24a—H21a   | 1.1030      |
| C8—H6     | 1.1110      | C24a—C23a   | 1.374 (6)   |
| C8—C7     | 1.506 (2)   | C23a—H20a   | 1.1030      |
| C7—C6     | 1.392 (2)   | C23a—C22a   | 1.397 (8)   |
| C14—H18   | 1.1110      | C22a—H19a   | 1.1030      |
| C14—H17   | 1.1110      | F1b—C21b    | 1.348 (15)  |
| C9—H13    | 1.1030      | C21b—C26b   | 1.372 (13)  |
| C9—C10    | 1.389 (2)   | C21b—C22b   | 1.361 (13)  |
| C20—H11   | 1.1110      | C26b—H23b   | 1.1030      |
| C20—H12   | 1.1110      | C26b—C25b   | 1.375 (13)  |
| C10—H14   | 1.1030      | C25b—H22b   | 1.1030      |
| C10—C11   | 1.387 (3)   | C25b—C24b   | 1.367 (13)  |
| C15—H7    | 1.1030      | C24b—H21b   | 1.1030      |
| C15—C16   | 1.387 (2)   | C24b—C23b   | 1.384 (14)  |
| C11—H15   | 1.1030      | C23b—H20b   | 1.1030      |
| C17—H9    | 1.1030      | C23b—C22b   | 1.387 (14)  |
| C17—C16   | 1.391 (3)   | C22b—H19b   | 1.1030      |
| N5—Fe1—N4 | 76.19 (5)   | H15—C11—C12 | 120.65 (10) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N6—Fe1—N4   | 75.89 (5)   | H15—C11—C10    | 120.65 (10) |
| N6—Fe1—N5   | 152.07 (5)  | N1—C1—S1       | 178.52 (18) |
| N3—Fe1—N4   | 78.30 (5)   | H9—C17—C18     | 120.48 (10) |
| N3—Fe1—N5   | 89.68 (5)   | C16—C17—C18    | 119.04 (16) |
| N3—Fe1—N6   | 84.08 (5)   | C16—C17—H9     | 120.48 (10) |
| N1—Fe1—N4   | 91.51 (5)   | C17—C16—C15    | 118.49 (16) |
| N1—Fe1—N5   | 91.81 (5)   | H8—C16—C15     | 120.75 (10) |
| N1—Fe1—N6   | 89.57 (5)   | H8—C16—C17     | 120.75 (10) |
| N1—Fe1—N3   | 169.04 (6)  | H1—C3—N3       | 118.63 (10) |
| N2—Fe1—N4   | 171.98 (6)  | C4—C3—N3       | 122.74 (19) |
| N2—Fe1—N5   | 100.43 (6)  | C4—C3—H1       | 118.63 (13) |
| N2—Fe1—N6   | 107.17 (6)  | C26—C25—H22    | 119.4 (3)   |
| N2—Fe1—N3   | 94.51 (6)   | C24—C25—H22    | 119.4 (3)   |
| N2—Fe1—N1   | 95.89 (6)   | C24—C25—C26    | 121.2 (4)   |
| C8—N4—Fe1   | 111.09 (9)  | H4—C6—C7       | 120.44 (11) |
| C14—N4—Fe1  | 105.81 (9)  | C5—C6—C7       | 119.12 (19) |
| C14—N4—C8   | 111.27 (12) | C5—C6—H4       | 120.44 (12) |
| C20—N4—Fe1  | 105.82 (9)  | H23—C26—C25    | 120.3 (3)   |
| C20—N4—C8   | 111.95 (13) | C21—C26—C25    | 119.4 (4)   |
| C20—N4—C14  | 110.59 (12) | C21—C26—H23    | 120.3 (3)   |
| C13—N5—Fe1  | 114.73 (10) | H2—C4—C3       | 120.78 (13) |
| C9—N5—Fe1   | 126.77 (11) | C5—C4—C3       | 118.45 (19) |
| C9—N5—C13   | 118.50 (14) | C5—C4—H2       | 120.78 (12) |
| C19—N6—Fe1  | 115.25 (10) | C4—C5—C6       | 119.05 (18) |
| C15—N6—Fe1  | 126.08 (11) | H3—C5—C6       | 120.48 (12) |
| C15—N6—C19  | 118.66 (14) | H3—C5—C4       | 120.48 (11) |
| C7—N3—Fe1   | 116.15 (10) | H21—C24—C25    | 121.0 (3)   |
| C3—N3—Fe1   | 124.95 (12) | C23—C24—C25    | 118.0 (5)   |
| C3—N3—C7    | 118.76 (15) | C23—C24—H21    | 121.0 (3)   |
| C1—N1—Fe1   | 169.59 (15) | F1—C21—C26     | 122.6 (5)   |
| C2—N2—Fe1   | 178.01 (15) | C22—C21—C26    | 121.8 (4)   |
| C18—C19—N6  | 122.22 (14) | C22—C21—F1     | 115.6 (5)   |
| C20—C19—N6  | 115.93 (13) | H19—C22—C21    | 120.7 (3)   |
| C20—C19—C18 | 121.78 (14) | C23—C22—C21    | 118.6 (4)   |
| C12—C13—N5  | 122.08 (14) | C23—C22—H19    | 120.7 (3)   |
| C14—C13—N5  | 116.13 (13) | C22—C23—C24    | 121.0 (4)   |
| C14—C13—C12 | 121.74 (14) | H20—C23—C24    | 119.5 (3)   |
| H10—C18—C19 | 120.59 (9)  | H20—C23—C22    | 119.5 (3)   |
| C17—C18—C19 | 118.83 (16) | C26a—C21a—F1a  | 105.1 (9)   |
| C17—C18—H10 | 120.59 (10) | C22a—C21a—F1a  | 136.1 (9)   |
| H16—C12—C13 | 120.38 (9)  | C22a—C21a—C26a | 118.6 (4)   |
| C11—C12—C13 | 119.23 (15) | H23a—C26a—C21a | 119.1 (3)   |
| C11—C12—H16 | 120.38 (10) | C25a—C26a—C21a | 121.8 (4)   |
| H5—C8—N4    | 108.46 (8)  | C25a—C26a—H23a | 119.1 (3)   |
| H6—C8—N4    | 108.46 (8)  | H22a—C25a—C26a | 120.3 (3)   |
| H6—C8—H5    | 107.5       | C24a—C25a—C26a | 119.4 (4)   |
| C7—C8—N4    | 115.26 (12) | C24a—C25a—H22a | 120.3 (3)   |
| C7—C8—H5    | 108.46 (8)  | H21a—C24a—C25a | 119.4 (3)   |



|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C7—C8—H6       | 108.46 (9)   | C23a—C24a—C25a  | 121.2 (4)    |
| C8—C7—N3       | 118.62 (13)  | C23a—C24a—H21a  | 119.4 (3)    |
| C6—C7—N3       | 121.88 (16)  | H20a—C23a—C24a  | 121.0 (3)    |
| C6—C7—C8       | 119.46 (15)  | C22a—C23a—C24a  | 118.0 (5)    |
| C13—C14—N4     | 111.17 (12)  | C22a—C23a—H20a  | 121.0 (3)    |
| H18—C14—N4     | 109.40 (8)   | C23a—C22a—C21a  | 121.0 (4)    |
| H18—C14—C13    | 109.40 (9)   | H19a—C22a—C21a  | 119.5 (3)    |
| H17—C14—N4     | 109.40 (8)   | H19a—C22a—C23a  | 119.5 (3)    |
| H17—C14—C13    | 109.40 (8)   | C26b—C21b—F1b   | 114.0 (15)   |
| H17—C14—H18    | 108.0        | C22b—C21b—F1b   | 122.3 (15)   |
| H13—C9—N5      | 118.64 (9)   | C22b—C21b—C26b  | 123.4 (16)   |
| C10—C9—N5      | 122.72 (15)  | H23b—C26b—C21b  | 121.1 (11)   |
| C10—C9—H13     | 118.64 (10)  | C25b—C26b—C21b  | 117.8 (16)   |
| C19—C20—N4     | 111.25 (12)  | C25b—C26b—H23b  | 121.1 (10)   |
| H11—C20—N4     | 109.38 (8)   | H22b—C25b—C26b  | 120.1 (10)   |
| H11—C20—C19    | 109.38 (8)   | C24b—C25b—C26b  | 119.8 (15)   |
| H12—C20—N4     | 109.38 (8)   | C24b—C25b—H22b  | 120.1 (10)   |
| H12—C20—C19    | 109.38 (9)   | H21b—C24b—C25b  | 119.0 (10)   |
| H12—C20—H11    | 108.0        | C23b—C24b—C25b  | 121.9 (16)   |
| H14—C10—C9     | 120.62 (10)  | C23b—C24b—H21b  | 119.0 (11)   |
| C11—C10—C9     | 118.75 (15)  | H20b—C23b—C24b  | 120.9 (11)   |
| C11—C10—H14    | 120.62 (10)  | C22b—C23b—C24b  | 118.2 (17)   |
| H7—C15—N6      | 118.64 (9)   | C22b—C23b—H20b  | 120.9 (11)   |
| C16—C15—N6     | 122.71 (16)  | C23b—C22b—C21b  | 118.6 (16)   |
| C16—C15—H7     | 118.64 (10)  | H19b—C22b—C21b  | 120.7 (11)   |
| N2—C2—S2       | 179.63 (17)  | H19b—C22b—C23b  | 120.7 (11)   |
| C10—C11—C12    | 118.71 (15)  |                 |              |
| Fe1—N4—C8—C7   | −7.04 (10)   | C8—C7—N3—C3     | 177.93 (16)  |
| Fe1—N4—C14—C13 | −42.89 (9)   | C8—C7—C6—C5     | −177.85 (18) |
| Fe1—N4—C20—C19 | 42.51 (9)    | C7—N3—C3—C4     | −0.4 (2)     |
| Fe1—N5—C13—C12 | 179.35 (10)  | C7—C8—N4—C14    | −124.65 (14) |
| Fe1—N5—C13—C14 | −3.02 (11)   | C7—C8—N4—C20    | 111.03 (14)  |
| Fe1—N5—C9—C10  | −179.92 (13) | C7—C6—C5—C4     | 0.3 (2)      |
| Fe1—N6—C19—C18 | 177.89 (10)  | C14—C13—N5—C9   | 177.57 (13)  |
| Fe1—N6—C19—C20 | 0.93 (11)    | C14—C13—C12—C11 | −176.88 (16) |
| Fe1—N6—C15—C16 | −176.85 (13) | C20—C19—N6—C15  | −178.15 (14) |
| Fe1—N3—C7—C8   | −6.11 (12)   | C20—C19—C18—C17 | 175.93 (16)  |
| Fe1—N3—C7—C6   | 176.29 (12)  | C3—N3—C7—C6     | 0.33 (19)    |
| Fe1—N3—C3—C4   | −175.98 (17) | C3—C4—C5—C6     | −0.3 (3)     |
| N4—C8—C7—N3    | 9.00 (17)    | C25—C26—C21—F1  | 179.4 (4)    |
| N4—C8—C7—C6    | −173.34 (15) | C25—C26—C21—C22 | 1.2 (5)      |
| N4—C14—C13—N5  | 32.30 (15)   | C25—C24—C23—C22 | −0.0 (5)     |
| N4—C14—C13—C12 | −150.07 (13) | C26—C25—C24—C23 | 1.3 (6)      |
| N4—C20—C19—N6  | −30.60 (15)  | C26—C21—C22—C23 | 0.0 (4)      |
| N4—C20—C19—C18 | 152.43 (13)  | C24—C25—C26—C21 | −1.9 (5)     |
| N5—C13—C12—C11 | 0.62 (18)    | C24—C23—C22—C21 | −0.6 (5)     |
| N5—C9—C10—C11  | 0.67 (19)    | F1—C21—C22—C23  | −178.2 (3)   |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N6—C19—C18—C17  | −0.84 (18)   | F1a—C21a—C26a—C25a  | 176.2 (6)   |
| N6—C15—C16—C17  | −1.0 (2)     | F1a—C21a—C22a—C23a  | −175.3 (9)  |
| N3—C7—C6—C5     | −0.3 (2)     | C21a—C26a—C25a—C24a | 1.2 (5)     |
| N3—C3—C4—C5     | 0.4 (2)      | C21a—C22a—C23a—C24a | −0.0 (5)    |
| C19—N6—C15—C16  | 2.13 (17)    | C26a—C21a—C22a—C23a | −0.6 (5)    |
| C19—C18—C17—C16 | 1.98 (19)    | C26a—C25a—C24a—C23a | −1.9 (5)    |
| C19—C20—N4—C8   | −78.66 (14)  | C25a—C26a—C21a—C22a | 0.0 (4)     |
| C19—C20—N4—C14  | 156.65 (13)  | C25a—C24a—C23a—C22a | 1.3 (6)     |
| C13—N5—C9—C10   | −0.60 (16)   | F1b—C21b—C26b—C25b  | −175.4 (15) |
| C13—C12—C11—C10 | −0.53 (18)   | F1b—C21b—C22b—C23b  | 176.8 (19)  |
| C13—C14—N4—C8   | 77.88 (14)   | C21b—C26b—C25b—C24b | 1 (3)       |
| C13—C14—N4—C20  | −157.04 (13) | C21b—C22b—C23b—C24b | −5 (3)      |
| C18—C19—N6—C15  | −1.20 (18)   | C26b—C21b—C22b—C23b | 3 (2)       |
| C18—C17—C16—C15 | −1.1 (2)     | C26b—C25b—C24b—C23b | −4 (3)      |
| C12—C13—N5—C9   | −0.06 (18)   | C25b—C26b—C21b—C22b | −1 (3)      |
| C12—C11—C10—C9  | −0.1 (2)     | C25b—C24b—C23b—C22b | 6 (3)       |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_175K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.2396$  (3) Å

$b = 12.0011$  (2) Å

$c = 15.4444$  (3) Å

$\beta = 110.445$  (2)°

$V = 2646.73$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.402$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 57911 reflections

$\theta = 2.1$ – $32.5^\circ$

$\mu = 0.76$  mm<sup>−1</sup>

$T = 175$  K

Block, yellow

$0.31 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.664$ ,  $T_{\max} = 1.000$

76659 measured reflections

6558 independent reflections

6122 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 17$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.091$

$S = 1.05$

6558 reflections

408 parameters

149 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 1.3085P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.0003$

$\Delta\rho_{\max} = 0.80$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.52$  e Å<sup>−3</sup>

*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 | 0.655545 (14) | 0.330477 (16) | 0.803114 (13) | 0.02628 (7)                      |           |
| S2  | 0.47137 (3)   | 0.00242 (4)   | 0.67458 (4)   | 0.04743 (12)                     |           |
| S1  | 0.76522 (11)  | 0.34385 (11)  | 0.54334 (8)   | 0.06691 (19)                     |           |
| N4  | 0.74106 (8)   | 0.47209 (9)   | 0.88397 (8)   | 0.0258 (2)                       |           |
| N6  | 0.56559 (8)   | 0.47678 (10)  | 0.75510 (8)   | 0.0276 (2)                       |           |
| N5  | 0.78608 (8)   | 0.25113 (10)  | 0.89134 (8)   | 0.0273 (2)                       |           |
| N3  | 0.60836 (9)   | 0.33916 (11)  | 0.92164 (8)   | 0.0315 (3)                       |           |
| N1  | 0.70882 (10)  | 0.35419 (11)  | 0.69559 (10)  | 0.0384 (3)                       |           |
| N2  | 0.57641 (11)  | 0.19263 (13)  | 0.74660 (10)  | 0.0432 (3)                       |           |
| C19 | 0.60964 (10)  | 0.57513 (11)  | 0.77372 (9)   | 0.0275 (3)                       |           |
| C13 | 0.85777 (10)  | 0.32155 (11)  | 0.92874 (9)   | 0.0272 (3)                       |           |
| C18 | 0.56279 (11)  | 0.67577 (12)  | 0.74522 (11)  | 0.0344 (3)                       |           |
| H10 | 0.60078 (11)  | 0.75562 (12)  | 0.76238 (11)  | 0.0412 (4)*                      |           |
| C12 | 0.94515 (10)  | 0.28524 (13)  | 0.98701 (10)  | 0.0335 (3)                       |           |
| H16 | 1.00250 (10)  | 0.34533 (13)  | 1.01723 (10)  | 0.0402 (4)*                      |           |
| C7  | 0.64612 (10)  | 0.41984 (13)  | 0.98291 (9)   | 0.0310 (3)                       |           |
| C8  | 0.72406 (11)  | 0.48886 (13)  | 0.97166 (10)  | 0.0348 (3)                       |           |
| H5  | 0.78974 (11)  | 0.46966 (13)  | 1.02987 (10)  | 0.0418 (4)*                      |           |
| H6  | 0.70781 (11)  | 0.57826 (13)  | 0.97715 (10)  | 0.0418 (4)*                      |           |
| C9  | 0.79982 (11)  | 0.14221 (12)  | 0.91115 (10)  | 0.0320 (3)                       |           |
| H13 | 0.74082 (11)  | 0.08422 (12)  | 0.88114 (10)  | 0.0384 (4)*                      |           |
| C10 | 0.88520 (12)  | 0.09954 (13)  | 0.96782 (11)  | 0.0374 (3)                       |           |
| H14 | 0.89403 (12)  | 0.00932 (13)  | 0.98174 (11)  | 0.0448 (4)*                      |           |
| C14 | 0.83948 (10)  | 0.44220 (12)  | 0.90113 (11)  | 0.0316 (3)                       |           |
| H18 | 0.85607 (10)  | 0.45754 (12)  | 0.83759 (11)  | 0.0379 (3)*                      |           |
| H17 | 0.88602 (10)  | 0.49586 (12)  | 0.95699 (11)  | 0.0379 (3)*                      |           |
| C2  | 0.53256 (10)  | 0.11289 (13)  | 0.71685 (10)  | 0.0340 (3)                       |           |
| C11 | 0.95920 (12)  | 0.17232 (13)  | 1.00662 (11)  | 0.0376 (3)                       |           |
| H15 | 1.02790 (12)  | 0.14115 (13)  | 1.05219 (11)  | 0.0451 (4)*                      |           |
| C20 | 0.71393 (10)  | 0.57052 (12)  | 0.82306 (11)  | 0.0322 (3)                       |           |
| H11 | 0.73732 (10)  | 0.64734 (12)  | 0.86493 (11)  | 0.0386 (4)*                      |           |
| H12 | 0.74951 (10)  | 0.56731 (12)  | 0.77140 (11)  | 0.0386 (4)*                      |           |
| C15 | 0.47301 (11)  | 0.47561 (14)  | 0.70888 (10)  | 0.0354 (3)                       |           |
| H7  | 0.43629 (11)  | 0.39476 (14)  | 0.69564 (10)  | 0.0425 (4)*                      |           |
| C17 | 0.46770 (12)  | 0.67369 (14)  | 0.69498 (12)  | 0.0399 (4)                       |           |
| H9  | 0.42915 (12)  | 0.75179 (14)  | 0.66957 (12)  | 0.0478 (4)*                      |           |
| C1  | 0.73265 (12)  | 0.35134 (13)  | 0.63204 (11)  | 0.0364 (3)                       |           |
| C16 | 0.42152 (11)  | 0.57183 (15)  | 0.67705 (11)  | 0.0406 (4)                       |           |
| H8  | 0.34556 (11)  | 0.56762 (15)  | 0.63832 (11)  | 0.0487 (4)*                      |           |
| C3  | 0.54099 (12)  | 0.27515 (17)  | 0.93321 (12)  | 0.0446 (4)                       |           |
| H1  | 0.50948 (12)  | 0.20899 (17)  | 0.88224 (12)  | 0.0535 (5)*                      |           |
| C25 | 0.7789 (4)    | 0.2207 (5)    | 1.1315 (3)    | 0.0575 (13)                      | 0.712 (5) |
| H22 | 0.8013 (4)    | 0.2463 (5)    | 1.0738 (3)    | 0.0690 (16)*                     | 0.712 (5) |
| C6  | 0.61805 (12)  | 0.43886 (18)  | 1.05821 (11)  | 0.0470 (4)                       |           |
| H4  | 0.65054 (12)  | 0.50596 (18)  | 1.10787 (11)  | 0.0564 (5)*                      |           |

|      |              |             |              |              |           |
|------|--------------|-------------|--------------|--------------|-----------|
| C26  | 0.8082 (3)   | 0.2808 (3)  | 1.2118 (3)   | 0.0579 (9)   | 0.712 (5) |
| H23  | 0.8553 (3)   | 0.3528 (3)  | 1.2195 (3)   | 0.0695 (10)* | 0.712 (5) |
| C4   | 0.50947 (14) | 0.2888 (2)  | 1.00646 (14) | 0.0591 (6)   |           |
| H2   | 0.45434 (14) | 0.2343 (2)  | 1.01407 (14) | 0.0709 (7)*  |           |
| C5   | 0.54882 (14) | 0.3725 (2)  | 1.06997 (13) | 0.0610 (6)   |           |
| H3   | 0.52529 (14) | 0.3860 (2)  | 1.12906 (13) | 0.0732 (7)*  |           |
| C21  | 0.7789 (3)   | 0.2509 (4)  | 1.2817 (2)   | 0.0784 (11)  | 0.712 (5) |
| C24  | 0.7223 (4)   | 0.1291 (4)  | 1.1214 (4)   | 0.0880 (15)  | 0.712 (5) |
| H21  | 0.6997 (4)   | 0.0801 (4)  | 1.0569 (4)   | 0.1056 (18)* | 0.712 (5) |
| C22  | 0.7219 (4)   | 0.1611 (5)  | 1.2751 (4)   | 0.107 (2)    | 0.712 (5) |
| H19  | 0.6988 (4)   | 0.1383 (5)  | 1.3330 (4)   | 0.128 (2)*   | 0.712 (5) |
| F1   | 0.8038 (3)   | 0.3023 (4)  | 1.35794 (19) | 0.166 (2)    | 0.712 (5) |
| C23  | 0.6941 (3)   | 0.1000 (4)  | 1.1953 (6)   | 0.120 (2)    | 0.712 (5) |
| H20  | 0.6487 (3)   | 0.0266 (4)  | 1.1892 (6)   | 0.144 (3)*   | 0.712 (5) |
| F1a  | 0.7111 (9)   | 0.1503 (11) | 1.3572 (7)   | 0.104 (5)    | 0.168 (5) |
| C21a | 0.7219 (4)   | 0.1611 (5)  | 1.2751 (4)   | 0.107 (2)    | 0.168 (5) |
| C22a | 0.6941 (3)   | 0.1000 (4)  | 1.1953 (6)   | 0.120 (2)    | 0.168 (5) |
| H19a | 0.6487 (3)   | 0.0266 (4)  | 1.1892 (6)   | 0.144 (3)*   | 0.168 (5) |
| C23a | 0.7223 (4)   | 0.1291 (4)  | 1.1214 (4)   | 0.0880 (15)  | 0.168 (5) |
| H20a | 0.6997 (4)   | 0.0801 (4)  | 1.0569 (4)   | 0.1056 (18)* | 0.168 (5) |
| C24a | 0.7789 (4)   | 0.2207 (5)  | 1.1315 (3)   | 0.0575 (13)  | 0.168 (5) |
| H21a | 0.8013 (4)   | 0.2463 (5)  | 1.0738 (3)   | 0.0690 (16)* | 0.168 (5) |
| C25a | 0.8082 (3)   | 0.2808 (3)  | 1.2118 (3)   | 0.0579 (9)   | 0.168 (5) |
| H22a | 0.8553 (3)   | 0.3528 (3)  | 1.2195 (3)   | 0.0695 (10)* | 0.168 (5) |
| C26a | 0.7789 (3)   | 0.2509 (4)  | 1.2817 (2)   | 0.0784 (11)  | 0.168 (5) |
| H23a | 0.8017 (3)   | 0.3006 (4)  | 1.3458 (2)   | 0.0941 (14)* | 0.168 (5) |
| F1b  | 0.6237 (9)   | 0.0050 (10) | 1.1198 (9)   | 0.079 (3)    | 0.12      |
| C21b | 0.6883 (15)  | 0.0850 (17) | 1.1536 (11)  | 0.054 (5)    | 0.12      |
| C22b | 0.7327 (18)  | 0.138 (2)   | 1.1023 (13)  | 0.037 (4)    | 0.12      |
| H19b | 0.7208 (18)  | 0.112 (2)   | 1.0306 (13)  | 0.044 (5)*   | 0.12      |
| C23b | 0.793 (2)    | 0.225 (3)   | 1.1420 (18)  | 0.033 (5)    | 0.12      |
| H20b | 0.831 (2)    | 0.268 (3)   | 1.1029 (18)  | 0.040 (6)*   | 0.12      |
| C24b | 0.8048 (17)  | 0.2566 (18) | 1.2316 (14)  | 0.041 (5)    | 0.12      |
| H21b | 0.8501 (17)  | 0.3283 (18) | 1.2627 (14)  | 0.049 (6)*   | 0.12      |
| C25b | 0.7612 (14)  | 0.2006 (15) | 1.2822 (13)  | 0.042 (4)    | 0.12      |
| H22b | 0.7755 (14)  | 0.2233 (15) | 1.3551 (13)  | 0.050 (5)*   | 0.12      |
| C26b | 0.6994 (14)  | 0.1157 (15) | 1.2424 (11)  | 0.074 (8)    | 0.12      |
| H23b | 0.6599 (14)  | 0.0737 (15) | 1.2808 (11)  | 0.088 (9)*   | 0.12      |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$    | $U^{23}$    |
|-----|--------------|--------------|--------------|---------------|-------------|-------------|
| Fe1 | 0.02955 (11) | 0.02456 (11) | 0.02401 (11) | −0.00140 (7)  | 0.00844 (8) | 0.00171 (7) |
| S2  | 0.0409 (2)   | 0.0392 (2)   | 0.0655 (3)   | −0.01192 (17) | 0.0226 (2)  | −0.0106 (2) |
| S1  | 0.1014 (5)   | 0.0691 (4)   | 0.0452 (3)   | 0.0471 (3)    | 0.0444 (3)  | 0.0260 (2)  |
| N4  | 0.0275 (5)   | 0.0219 (5)   | 0.0267 (5)   | −0.0036 (4)   | 0.0078 (4)  | 0.0005 (4)  |
| N6  | 0.0308 (6)   | 0.0247 (5)   | 0.0250 (5)   | −0.0009 (4)   | 0.0068 (4)  | −0.0011 (4) |
| N5  | 0.0317 (6)   | 0.0235 (5)   | 0.0246 (5)   | −0.0028 (4)   | 0.0073 (4)  | 0.0006 (4)  |

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|      |             |             |             |              |             |             |
|------|-------------|-------------|-------------|--------------|-------------|-------------|
| N3   | 0.0311 (6)  | 0.0392 (7)  | 0.0249 (5)  | −0.0041 (5)  | 0.0104 (5)  | 0.0019 (5)  |
| N1   | 0.0495 (8)  | 0.0344 (7)  | 0.0352 (7)  | 0.0062 (6)   | 0.0198 (6)  | 0.0045 (5)  |
| N2   | 0.0448 (8)  | 0.0395 (7)  | 0.0408 (7)  | −0.0083 (6)  | 0.0091 (6)  | −0.0058 (6) |
| C19  | 0.0324 (7)  | 0.0235 (6)  | 0.0256 (6)  | 0.0003 (5)   | 0.0089 (5)  | 0.0013 (5)  |
| C13  | 0.0277 (6)  | 0.0243 (6)  | 0.0285 (6)  | −0.0011 (5)  | 0.0085 (5)  | 0.0002 (5)  |
| C18  | 0.0389 (8)  | 0.0270 (7)  | 0.0353 (7)  | 0.0039 (6)   | 0.0106 (6)  | 0.0041 (6)  |
| C12  | 0.0283 (7)  | 0.0320 (7)  | 0.0363 (7)  | 0.0015 (6)   | 0.0065 (6)  | −0.0014 (6) |
| C7   | 0.0300 (6)  | 0.0405 (8)  | 0.0220 (6)  | 0.0034 (6)   | 0.0083 (5)  | 0.0007 (5)  |
| C8   | 0.0390 (8)  | 0.0364 (8)  | 0.0274 (6)  | −0.0056 (6)  | 0.0093 (6)  | −0.0070 (6) |
| C9   | 0.0408 (8)  | 0.0237 (6)  | 0.0284 (6)  | −0.0020 (6)  | 0.0082 (6)  | −0.0006 (5) |
| C10  | 0.0474 (9)  | 0.0264 (7)  | 0.0346 (7)  | 0.0059 (6)   | 0.0097 (6)  | 0.0010 (6)  |
| C14  | 0.0271 (6)  | 0.0258 (7)  | 0.0400 (8)  | −0.0039 (5)  | 0.0094 (6)  | 0.0026 (6)  |
| C2   | 0.0321 (7)  | 0.0330 (8)  | 0.0363 (7)  | −0.0059 (6)  | 0.0111 (6)  | −0.0051 (6) |
| C11  | 0.0364 (8)  | 0.0344 (8)  | 0.0372 (8)  | 0.0088 (6)   | 0.0069 (6)  | 0.0021 (6)  |
| C20  | 0.0329 (7)  | 0.0221 (6)  | 0.0385 (7)  | −0.0036 (5)  | 0.0086 (6)  | 0.0049 (5)  |
| C15  | 0.0329 (7)  | 0.0353 (8)  | 0.0327 (7)  | −0.0031 (6)  | 0.0046 (6)  | −0.0033 (6) |
| C17  | 0.0393 (8)  | 0.0371 (8)  | 0.0396 (8)  | 0.0109 (7)   | 0.0094 (7)  | 0.0074 (6)  |
| C1   | 0.0475 (8)  | 0.0347 (7)  | 0.0297 (7)  | 0.0140 (6)   | 0.0169 (6)  | 0.0077 (6)  |
| C16  | 0.0334 (7)  | 0.0444 (9)  | 0.0370 (8)  | 0.0053 (7)   | 0.0036 (6)  | 0.0012 (7)  |
| C3   | 0.0384 (8)  | 0.0598 (11) | 0.0367 (8)  | −0.0148 (8)  | 0.0145 (7)  | 0.0051 (8)  |
| C25  | 0.057 (3)   | 0.070 (3)   | 0.0521 (18) | 0.0215 (17)  | 0.0268 (18) | 0.0211 (16) |
| C6   | 0.0430 (9)  | 0.0722 (13) | 0.0283 (7)  | 0.0047 (8)   | 0.0157 (7)  | −0.0054 (8) |
| C26  | 0.0427 (13) | 0.067 (2)   | 0.069 (2)   | 0.0097 (13)  | 0.0255 (14) | 0.0215 (17) |
| C4   | 0.0424 (9)  | 0.0962 (17) | 0.0444 (10) | −0.0131 (10) | 0.0225 (8)  | 0.0129 (11) |
| C5   | 0.0474 (10) | 0.1080 (18) | 0.0357 (9)  | 0.0003 (11)  | 0.0247 (8)  | 0.0038 (10) |
| C21  | 0.071 (2)   | 0.118 (3)   | 0.0531 (17) | 0.048 (2)    | 0.0308 (15) | 0.034 (2)   |
| C24  | 0.063 (2)   | 0.066 (2)   | 0.128 (4)   | 0.0159 (18)  | 0.026 (3)   | 0.011 (3)   |
| C22  | 0.096 (3)   | 0.117 (4)   | 0.148 (5)   | 0.057 (3)    | 0.094 (3)   | 0.093 (4)   |
| F1   | 0.155 (3)   | 0.276 (5)   | 0.0592 (15) | 0.118 (3)    | 0.0256 (17) | −0.011 (2)  |
| C23  | 0.078 (3)   | 0.059 (2)   | 0.246 (8)   | 0.0163 (19)  | 0.085 (5)   | 0.056 (4)   |
| F1a  | 0.115 (9)   | 0.129 (10)  | 0.086 (7)   | 0.020 (7)    | 0.057 (7)   | 0.048 (7)   |
| C21a | 0.096 (3)   | 0.117 (4)   | 0.148 (5)   | 0.057 (3)    | 0.094 (3)   | 0.093 (4)   |
| C22a | 0.078 (3)   | 0.059 (2)   | 0.246 (8)   | 0.0163 (19)  | 0.085 (5)   | 0.056 (4)   |
| C23a | 0.063 (2)   | 0.066 (2)   | 0.128 (4)   | 0.0159 (18)  | 0.026 (3)   | 0.011 (3)   |
| C24a | 0.057 (3)   | 0.070 (3)   | 0.0521 (18) | 0.0215 (17)  | 0.0268 (18) | 0.0211 (16) |
| C25a | 0.0427 (13) | 0.067 (2)   | 0.069 (2)   | 0.0097 (13)  | 0.0255 (14) | 0.0215 (17) |
| C26a | 0.071 (2)   | 0.118 (3)   | 0.0531 (17) | 0.048 (2)    | 0.0308 (15) | 0.034 (2)   |
| F1b  | 0.090 (7)   | 0.076 (7)   | 0.080 (7)   | −0.035 (5)   | 0.042 (5)   | −0.008 (5)  |
| C21b | 0.066 (10)  | 0.062 (9)   | 0.045 (6)   | −0.019 (5)   | 0.033 (4)   | −0.000 (4)  |
| C22b | 0.035 (7)   | 0.045 (8)   | 0.033 (5)   | 0.002 (4)    | 0.015 (4)   | 0.013 (4)   |
| C23b | 0.027 (7)   | 0.039 (9)   | 0.039 (8)   | 0.007 (5)    | 0.018 (4)   | 0.015 (4)   |
| C24b | 0.038 (7)   | 0.047 (9)   | 0.042 (7)   | 0.006 (5)    | 0.021 (4)   | 0.013 (4)   |
| C25b | 0.055 (9)   | 0.039 (8)   | 0.042 (7)   | −0.005 (5)   | 0.030 (4)   | 0.002 (4)   |
| C26b | 0.103 (13)  | 0.076 (12)  | 0.058 (7)   | −0.045 (7)   | 0.048 (5)   | −0.012 (5)  |

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*Geometric parameters (Å, °)*

|           |             |            |             |
|-----------|-------------|------------|-------------|
| Fe1—N4    | 2.2382 (11) | C16—H8     | 1.1030      |
| Fe1—N6    | 2.1913 (12) | C3—H1      | 1.1030      |
| Fe1—N5    | 2.1970 (12) | C3—C4      | 1.384 (3)   |
| Fe1—N3    | 2.1906 (12) | C25—H22    | 1.1030      |
| Fe1—N1    | 2.1071 (14) | C25—C26    | 1.367 (5)   |
| Fe1—N2    | 2.0537 (15) | C25—C24    | 1.372 (6)   |
| S2—C2     | 1.6211 (16) | C6—H4      | 1.1030      |
| S1—C1     | 1.6142 (19) | C6—C5      | 1.384 (3)   |
| N4—C8     | 1.4788 (18) | C26—H23    | 1.1030      |
| N4—C14    | 1.4733 (18) | C26—C21    | 1.352 (4)   |
| N4—C20    | 1.4763 (17) | C4—H2      | 1.1030      |
| N6—C19    | 1.3386 (18) | C4—C5      | 1.386 (3)   |
| N6—C15    | 1.3404 (18) | C5—H3      | 1.1030      |
| N5—C13    | 1.3419 (17) | C21—C22    | 1.366 (7)   |
| N5—C9     | 1.3421 (18) | C21—F1     | 1.264 (5)   |
| N3—C7     | 1.3356 (19) | C24—H21    | 1.1030      |
| N3—C3     | 1.343 (2)   | C24—C23    | 1.397 (8)   |
| N1—C1     | 1.160 (2)   | C22—H19    | 1.1030      |
| N2—C2     | 1.166 (2)   | C22—C23    | 1.368 (8)   |
| C19—C18   | 1.3937 (19) | C23—H20    | 1.1030      |
| C19—C20   | 1.505 (2)   | F1a—C21a   | 1.340 (10)  |
| C13—C12   | 1.3913 (19) | C21a—C22a  | 1.368 (8)   |
| C13—C14   | 1.5077 (19) | C21a—C26a  | 1.366 (7)   |
| C18—H10   | 1.1030      | C22a—H19a  | 1.1030      |
| C18—C17   | 1.385 (2)   | C22a—C23a  | 1.397 (8)   |
| C12—H16   | 1.1030      | C23a—H20a  | 1.1030      |
| C12—C11   | 1.389 (2)   | C23a—C24a  | 1.372 (6)   |
| C7—C8     | 1.507 (2)   | C24a—H21a  | 1.1030      |
| C7—C6     | 1.391 (2)   | C24a—C25a  | 1.367 (5)   |
| C8—H5     | 1.1110      | C25a—H22a  | 1.1030      |
| C8—H6     | 1.1110      | C25a—C26a  | 1.352 (4)   |
| C9—H13    | 1.1030      | C26a—H23a  | 1.1030      |
| C9—C10    | 1.388 (2)   | F1b—C21b   | 1.344 (15)  |
| C10—H14   | 1.1030      | C21b—C22b  | 1.364 (13)  |
| C10—C11   | 1.386 (2)   | C21b—C26b  | 1.373 (13)  |
| C14—H18   | 1.1110      | C22b—H19b  | 1.1030      |
| C14—H17   | 1.1110      | C22b—C23b  | 1.388 (14)  |
| C11—H15   | 1.1030      | C23b—H20b  | 1.1030      |
| C20—H11   | 1.1110      | C23b—C24b  | 1.386 (14)  |
| C20—H12   | 1.1110      | C24b—H21b  | 1.1030      |
| C15—H7    | 1.1030      | C24b—C25b  | 1.367 (13)  |
| C15—C16   | 1.386 (2)   | C25b—H22b  | 1.1030      |
| C17—H9    | 1.1030      | C25b—C26b  | 1.377 (13)  |
| C17—C16   | 1.389 (2)   | C26b—H23b  | 1.1030      |
| N6—Fe1—N4 | 75.75 (4)   | C16—C15—N6 | 122.71 (15) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N5—Fe1—N4   | 76.02 (4)   | C16—C15—H7     | 118.65 (9)  |
| N5—Fe1—N6   | 151.77 (4)  | H9—C17—C18     | 120.47 (9)  |
| N3—Fe1—N4   | 78.16 (4)   | C16—C17—C18    | 119.05 (14) |
| N3—Fe1—N6   | 84.00 (5)   | C16—C17—H9     | 120.47 (9)  |
| N3—Fe1—N5   | 89.58 (4)   | N1—C1—S1       | 178.47 (17) |
| N1—Fe1—N4   | 91.58 (5)   | C17—C16—C15    | 118.54 (14) |
| N1—Fe1—N6   | 89.59 (5)   | H8—C16—C15     | 120.73 (9)  |
| N1—Fe1—N5   | 91.87 (5)   | H8—C16—C17     | 120.73 (9)  |
| N1—Fe1—N3   | 168.97 (5)  | H1—C3—N3       | 118.63 (10) |
| N2—Fe1—N4   | 171.83 (5)  | C4—C3—N3       | 122.73 (18) |
| N2—Fe1—N6   | 107.25 (5)  | C4—C3—H1       | 118.63 (12) |
| N2—Fe1—N5   | 100.63 (5)  | C26—C25—H22    | 119.4 (3)   |
| N2—Fe1—N3   | 94.47 (5)   | C24—C25—H22    | 119.4 (3)   |
| N2—Fe1—N1   | 96.00 (6)   | C24—C25—C26    | 121.2 (4)   |
| C8—N4—Fe1   | 111.11 (8)  | H4—C6—C7       | 120.44 (11) |
| C14—N4—Fe1  | 105.86 (8)  | C5—C6—C7       | 119.11 (18) |
| C14—N4—C8   | 111.18 (11) | C5—C6—H4       | 120.44 (12) |
| C20—N4—Fe1  | 105.85 (8)  | H23—C26—C25    | 120.3 (3)   |
| C20—N4—C8   | 112.02 (12) | C21—C26—C25    | 119.3 (4)   |
| C20—N4—C14  | 110.51 (11) | C21—C26—H23    | 120.3 (3)   |
| C19—N6—Fe1  | 115.23 (9)  | H2—C4—C3       | 120.79 (12) |
| C15—N6—Fe1  | 126.13 (10) | C5—C4—C3       | 118.43 (18) |
| C15—N6—C19  | 118.63 (13) | C5—C4—H2       | 120.79 (11) |
| C13—N5—Fe1  | 114.75 (9)  | C4—C5—C6       | 119.06 (17) |
| C9—N5—Fe1   | 126.79 (10) | H3—C5—C6       | 120.47 (12) |
| C9—N5—C13   | 118.45 (13) | H3—C5—C4       | 120.47 (11) |
| C7—N3—Fe1   | 116.14 (10) | C22—C21—C26    | 121.9 (4)   |
| C3—N3—Fe1   | 124.98 (11) | F1—C21—C26     | 122.6 (5)   |
| C3—N3—C7    | 118.73 (14) | F1—C21—C22     | 115.5 (5)   |
| C1—N1—Fe1   | 169.62 (14) | H21—C24—C25    | 121.0 (3)   |
| C2—N2—Fe1   | 178.11 (14) | C23—C24—C25    | 117.9 (5)   |
| C18—C19—N6  | 122.22 (13) | C23—C24—H21    | 121.0 (3)   |
| C20—C19—N6  | 115.99 (12) | H19—C22—C21    | 120.7 (3)   |
| C20—C19—C18 | 121.73 (13) | C23—C22—C21    | 118.7 (4)   |
| C12—C13—N5  | 122.18 (13) | C23—C22—H19    | 120.7 (3)   |
| C14—C13—N5  | 116.15 (12) | C22—C23—C24    | 120.9 (4)   |
| C14—C13—C12 | 121.62 (13) | H20—C23—C24    | 119.5 (3)   |
| H10—C18—C19 | 120.60 (9)  | H20—C23—C22    | 119.5 (3)   |
| C17—C18—C19 | 118.80 (14) | C22a—C21a—F1a  | 134.3 (8)   |
| C17—C18—H10 | 120.60 (9)  | C26a—C21a—F1a  | 106.9 (8)   |
| H16—C12—C13 | 120.45 (9)  | C26a—C21a—C22a | 118.7 (4)   |
| C11—C12—C13 | 119.11 (14) | H19a—C22a—C21a | 119.5 (3)   |
| C11—C12—H16 | 120.45 (9)  | C23a—C22a—C21a | 120.9 (4)   |
| C8—C7—N3    | 118.61 (12) | C23a—C22a—H19a | 119.5 (3)   |
| C6—C7—N3    | 121.94 (15) | H20a—C23a—C22a | 121.0 (3)   |
| C6—C7—C8    | 119.41 (14) | C24a—C23a—C22a | 117.9 (5)   |
| C7—C8—N4    | 115.34 (12) | C24a—C23a—H20a | 121.0 (3)   |
| H5—C8—N4    | 108.44 (8)  | H21a—C24a—C23a | 119.4 (3)   |



|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| H5—C8—C7       | 108.44 (8)   | C25a—C24a—C23a  | 121.2 (4)    |
| H6—C8—N4       | 108.44 (8)   | C25a—C24a—H21a  | 119.4 (3)    |
| H6—C8—C7       | 108.44 (8)   | H22a—C25a—C24a  | 120.3 (3)    |
| H6—C8—H5       | 107.5        | C26a—C25a—C24a  | 119.3 (4)    |
| H13—C9—N5      | 118.64 (8)   | C26a—C25a—H22a  | 120.3 (3)    |
| C10—C9—N5      | 122.72 (14)  | C25a—C26a—C21a  | 121.9 (4)    |
| C10—C9—H13     | 118.64 (9)   | H23a—C26a—C21a  | 119.1 (3)    |
| H14—C10—C9     | 120.59 (9)   | H23a—C26a—C25a  | 119.1 (3)    |
| C11—C10—C9     | 118.82 (14)  | C22b—C21b—F1b   | 123.5 (14)   |
| C11—C10—H14    | 120.59 (9)   | C26b—C21b—F1b   | 113.4 (14)   |
| C13—C14—N4     | 111.15 (11)  | C26b—C21b—C22b  | 122.9 (15)   |
| H18—C14—N4     | 109.40 (7)   | H19b—C22b—C21b  | 120.6 (10)   |
| H18—C14—C13    | 109.40 (8)   | C23b—C22b—C21b  | 118.7 (15)   |
| H17—C14—N4     | 109.40 (7)   | C23b—C22b—H19b  | 120.6 (10)   |
| H17—C14—C13    | 109.40 (8)   | H20b—C23b—C22b  | 120.6 (10)   |
| H17—C14—H18    | 108.0        | C24b—C23b—C22b  | 118.8 (16)   |
| N2—C2—S2       | 179.50 (15)  | C24b—C23b—H20b  | 120.6 (10)   |
| C10—C11—C12    | 118.71 (14)  | H21b—C24b—C23b  | 119.4 (10)   |
| H15—C11—C12    | 120.64 (9)   | C25b—C24b—C23b  | 121.3 (15)   |
| H15—C11—C10    | 120.64 (9)   | C25b—C24b—H21b  | 119.4 (10)   |
| C19—C20—N4     | 111.31 (11)  | H22b—C25b—C24b  | 119.9 (10)   |
| H11—C20—N4     | 109.37 (7)   | C26b—C25b—C24b  | 120.1 (14)   |
| H11—C20—C19    | 109.37 (8)   | C26b—C25b—H22b  | 119.9 (10)   |
| H12—C20—N4     | 109.37 (8)   | C25b—C26b—C21b  | 118.1 (15)   |
| H12—C20—C19    | 109.37 (8)   | H23b—C26b—C21b  | 121.0 (10)   |
| H12—C20—H11    | 108.0        | H23b—C26b—C25b  | 121.0 (10)   |
| H7—C15—N6      | 118.65 (9)   |                 |              |
| Fe1—N4—C8—C7   | −7.47 (9)    | C7—N3—C3—C4     | −0.49 (18)   |
| Fe1—N4—C14—C13 | −43.02 (8)   | C7—C8—N4—C14    | −125.10 (13) |
| Fe1—N4—C20—C19 | 42.56 (8)    | C7—C8—N4—C20    | 110.69 (13)  |
| Fe1—N6—C19—C18 | 177.93 (10)  | C7—C6—C5—C4     | 0.1 (2)      |
| Fe1—N6—C19—C20 | 1.01 (10)    | C8—C7—N3—C3     | 177.85 (15)  |
| Fe1—N6—C15—C16 | −176.94 (12) | C8—C7—C6—C5     | −177.65 (17) |
| Fe1—N5—C13—C12 | 179.40 (10)  | C9—N5—C13—C14   | 177.67 (13)  |
| Fe1—N5—C13—C14 | −2.87 (10)   | C14—C13—C12—C11 | −176.99 (15) |
| Fe1—N5—C9—C10  | 179.95 (12)  | C20—C19—N6—C15  | −178.12 (13) |
| Fe1—N3—C7—C8   | −6.52 (12)   | C20—C19—C18—C17 | 175.93 (15)  |
| Fe1—N3—C7—C6   | 175.97 (11)  | C3—N3—C7—C6     | 0.34 (18)    |
| Fe1—N3—C3—C4   | −175.70 (16) | C3—C4—C5—C6     | −0.2 (2)     |
| N4—C8—C7—N3    | 9.57 (16)    | C25—C26—C21—C22 | 1.1 (5)      |
| N4—C8—C7—C6    | −172.85 (14) | C25—C26—C21—F1  | 179.5 (4)    |
| N4—C14—C13—N5  | 32.28 (14)   | C25—C24—C23—C22 | 0.2 (5)      |
| N4—C14—C13—C12 | −149.98 (12) | C26—C25—C24—C23 | 1.0 (7)      |
| N4—C20—C19—N6  | −30.70 (14)  | C26—C21—C22—C23 | 0.0 (4)      |
| N4—C20—C19—C18 | 152.36 (12)  | C21—C26—C25—C24 | −1.6 (4)     |
| N6—C19—C18—C17 | −0.81 (16)   | C21—C22—C23—C24 | −0.7 (4)     |
| N6—C15—C16—C17 | −0.92 (19)   | F1—C21—C22—C23  | −178.4 (3)   |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N5—C13—C12—C11  | 0.62 (17)    | F1a—C21a—C22a—C23a  | −175.4 (8)  |
| N5—C9—C10—C11   | 0.82 (18)    | F1a—C21a—C26a—C25a  | 176.1 (6)   |
| N3—C7—C6—C5     | −0.15 (19)   | C21a—C22a—C23a—C24a | 0.2 (5)     |
| N3—C3—C4—C5     | 0.4 (2)      | C21a—C26a—C25a—C24a | 1.1 (5)     |
| C19—N6—C15—C16  | 2.08 (15)    | C22a—C21a—C26a—C25a | 0.0 (4)     |
| C19—C18—C17—C16 | 1.95 (18)    | C22a—C23a—C24a—C25a | 1.0 (5)     |
| C19—C20—N4—C8   | −78.69 (13)  | C23a—C22a—C21a—C26a | −0.7 (4)    |
| C19—C20—N4—C14  | 156.73 (12)  | C23a—C24a—C25a—C26a | −1.6 (6)    |
| C13—N5—C9—C10   | −0.66 (15)   | F1b—C21b—C22b—C23b  | 175.2 (16)  |
| C13—C12—C11—C10 | −0.44 (17)   | F1b—C21b—C26b—C25b  | −177.3 (15) |
| C13—C14—N4—C8   | 77.76 (13)   | C21b—C22b—C23b—C24b | −1 (3)      |
| C13—C14—N4—C20  | −157.19 (12) | C21b—C26b—C25b—C24b | 4 (3)       |
| C18—C19—N6—C15  | −1.19 (17)   | C22b—C21b—C26b—C25b | −3 (2)      |
| C18—C17—C16—C15 | −1.13 (19)   | C22b—C23b—C24b—C25b | 3 (3)       |
| C12—C13—N5—C9   | −0.07 (17)   | C23b—C22b—C21b—C26b | 1 (3)       |
| C12—C11—C10—C9  | −0.24 (19)   | C23b—C24b—C25b—C26b | −4 (3)      |

### Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) fluorobenzene monosolvate (f\_180K)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.2539$  (3) Å

$b = 12.0022$  (2) Å

$c = 15.4464$  (3) Å

$\beta = 110.437$  (2)°

$V = 2649.93$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.400$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 57588 reflections

$\theta = 2.1$ – $32.5^\circ$

$\mu = 0.76$  mm<sup>−1</sup>

$T = 180$  K

Block, yellow

$0.31 \times 0.2 \times 0.12$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.663$ ,  $T_{\max} = 1.000$

76682 measured reflections

6565 independent reflections

6101 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.090$

$S = 1.05$

6565 reflections

408 parameters

149 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 1.190P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.77$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.52$  e Å<sup>−3</sup>

*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  | 0.344402 (13) | 0.330203 (16) | 0.197054 (13) | 0.02662 (7)                      |           |
| S2   | 0.52859 (3)   | 0.00241 (4)   | 0.32553 (4)   | 0.04820 (12)                     |           |
| S1   | 0.23493 (11)  | 0.34381 (11)  | 0.45688 (8)   | 0.06839 (19)                     |           |
| N4   | 0.25895 (8)   | 0.47190 (9)   | 0.11609 (8)   | 0.0266 (2)                       |           |
| N5   | 0.21379 (8)   | 0.25094 (9)   | 0.10876 (7)   | 0.0276 (2)                       |           |
| N6   | 0.43439 (8)   | 0.47672 (10)  | 0.24490 (8)   | 0.0282 (2)                       |           |
| N3   | 0.39151 (9)   | 0.33890 (10)  | 0.07835 (8)   | 0.0321 (3)                       |           |
| N1   | 0.29128 (10)  | 0.35390 (11)  | 0.30470 (9)   | 0.0391 (3)                       |           |
| N2   | 0.42347 (10)  | 0.19234 (12)  | 0.25349 (10)  | 0.0439 (3)                       |           |
| C19  | 0.39026 (10)  | 0.57505 (11)  | 0.22613 (9)   | 0.0279 (3)                       |           |
| C18  | 0.43707 (11)  | 0.67559 (12)  | 0.25449 (11)  | 0.0351 (3)                       |           |
| H10  | 0.39911 (11)  | 0.75542 (12)  | 0.23724 (11)  | 0.0421 (4)*                      |           |
| C13  | 0.14225 (9)   | 0.32159 (11)  | 0.07142 (9)   | 0.0279 (3)                       |           |
| C12  | 0.05488 (10)  | 0.28529 (13)  | 0.01321 (10)  | 0.0343 (3)                       |           |
| H16  | −0.00240 (10) | 0.34544 (13)  | −0.01682 (10) | 0.0412 (4)*                      |           |
| C9   | 0.20000 (11)  | 0.14215 (12)  | 0.08890 (10)  | 0.0326 (3)                       |           |
| H13  | 0.25893 (11)  | 0.08412 (12)  | 0.11884 (10)  | 0.0391 (3)*                      |           |
| C8   | 0.27589 (11)  | 0.48859 (13)  | 0.02837 (10)  | 0.0354 (3)                       |           |
| H5   | 0.21030 (11)  | 0.46921 (13)  | −0.02979 (10) | 0.0425 (4)*                      |           |
| H6   | 0.29199 (11)  | 0.57801 (13)  | 0.02277 (10)  | 0.0425 (4)*                      |           |
| C7   | 0.35385 (10)  | 0.41969 (13)  | 0.01720 (9)   | 0.0319 (3)                       |           |
| C10  | 0.11462 (12)  | 0.09951 (13)  | 0.03221 (10)  | 0.0383 (3)                       |           |
| H14  | 0.10572 (12)  | 0.00930 (13)  | 0.01827 (10)  | 0.0459 (4)*                      |           |
| C14  | 0.16073 (9)   | 0.44208 (12)  | 0.09914 (11)  | 0.0324 (3)                       |           |
| H18  | 0.14434 (9)   | 0.45734 (12)  | 0.16279 (11)  | 0.0389 (3)*                      |           |
| H17  | 0.11419 (9)   | 0.49586 (12)  | 0.04343 (11)  | 0.0389 (3)*                      |           |
| C2   | 0.46731 (10)  | 0.11268 (13)  | 0.28326 (10)  | 0.0343 (3)                       |           |
| C20  | 0.28613 (10)  | 0.57046 (11)  | 0.17695 (10)  | 0.0330 (3)                       |           |
| H11  | 0.26273 (10)  | 0.64724 (11)  | 0.13504 (10)  | 0.0396 (3)*                      |           |
| H12  | 0.25066 (10)  | 0.56734 (11)  | 0.22868 (10)  | 0.0396 (3)*                      |           |
| C11  | 0.04070 (11)  | 0.17252 (13)  | −0.00659 (11) | 0.0385 (3)                       |           |
| H15  | −0.02795 (11) | 0.14152 (13)  | −0.05224 (11) | 0.0462 (4)*                      |           |
| C15  | 0.52681 (10)  | 0.47566 (13)  | 0.29112 (10)  | 0.0363 (3)                       |           |
| H7   | 0.56351 (10)  | 0.39482 (13)  | 0.30452 (10)  | 0.0435 (4)*                      |           |
| C17  | 0.53205 (12)  | 0.67363 (14)  | 0.30471 (12)  | 0.0411 (4)                       |           |
| H9   | 0.57051 (12)  | 0.75178 (14)  | 0.33002 (12)  | 0.0494 (4)*                      |           |
| C1   | 0.26752 (12)  | 0.35115 (13)  | 0.36824 (10)  | 0.0370 (3)                       |           |
| C16  | 0.57822 (11)  | 0.57181 (15)  | 0.32277 (11)  | 0.0414 (3)                       |           |
| H8   | 0.65411 (11)  | 0.56764 (15)  | 0.36144 (11)  | 0.0497 (4)*                      |           |
| C3   | 0.45884 (12)  | 0.27502 (17)  | 0.06671 (12)  | 0.0460 (4)                       |           |
| H1   | 0.49035 (12)  | 0.20884 (17)  | 0.11764 (12)  | 0.0552 (5)*                      |           |
| C6   | 0.38185 (12)  | 0.43875 (18)  | −0.05810 (11) | 0.0483 (4)                       |           |
| H4   | 0.34931 (12)  | 0.50577 (18)  | −0.10779 (11) | 0.0579 (5)*                      |           |
| C24a | 0.2208 (4)    | 0.2207 (5)    | −0.1316 (3)   | 0.0587 (12)                      | 0.171 (5) |
| H21a | 0.1985 (4)    | 0.2464 (5)    | −0.0739 (3)   | 0.0705 (15)*                     | 0.171 (5) |

|      |              |             |               |              |           |
|------|--------------|-------------|---------------|--------------|-----------|
| C26  | 0.1917 (2)   | 0.2804 (3)  | −0.2119 (3)   | 0.0593 (8)   | 0.709 (5) |
| H23  | 0.1447 (2)   | 0.3524 (3)  | −0.2196 (3)   | 0.0712 (10)* | 0.709 (5) |
| C4   | 0.49033 (14) | 0.2887 (2)  | −0.00638 (14) | 0.0610 (6)   |           |
| H2   | 0.54538 (14) | 0.2342 (2)  | −0.01403 (14) | 0.0732 (7)*  |           |
| C5   | 0.45107 (14) | 0.3725 (2)  | −0.06978 (13) | 0.0625 (6)   |           |
| H3   | 0.47469 (14) | 0.3861 (2)  | −0.12878 (13) | 0.0750 (7)*  |           |
| C26a | 0.2209 (3)   | 0.2509 (4)  | −0.2818 (2)   | 0.0809 (11)  | 0.171 (5) |
| H23a | 0.1978 (3)   | 0.3005 (4)  | −0.3459 (2)   | 0.0971 (14)* | 0.171 (5) |
| C23a | 0.2772 (4)   | 0.1291 (4)  | −0.1217 (4)   | 0.0918 (15)  | 0.171 (5) |
| H20a | 0.2995 (4)   | 0.0797 (4)  | −0.0575 (4)   | 0.1102 (18)* | 0.171 (5) |
| C21a | 0.2779 (4)   | 0.1615 (5)  | −0.2754 (4)   | 0.110 (2)    | 0.171 (5) |
| F1   | 0.1961 (3)   | 0.3022 (4)  | −0.35779 (19) | 0.175 (2)    | 0.709 (5) |
| C22a | 0.3056 (3)   | 0.1002 (4)  | −0.1957 (6)   | 0.123 (2)    | 0.171 (5) |
| H19a | 0.3510 (3)   | 0.0270 (4)  | −0.1896 (6)   | 0.147 (3)*   | 0.171 (5) |
| C21  | 0.2209 (3)   | 0.2509 (4)  | −0.2818 (2)   | 0.0809 (11)  | 0.709 (5) |
| C25a | 0.1917 (2)   | 0.2804 (3)  | −0.2119 (3)   | 0.0593 (8)   | 0.171 (5) |
| H22a | 0.1447 (2)   | 0.3524 (3)  | −0.2196 (3)   | 0.0712 (10)* | 0.171 (5) |
| C25  | 0.2208 (4)   | 0.2207 (5)  | −0.1316 (3)   | 0.0587 (12)  | 0.709 (5) |
| H22  | 0.1985 (4)   | 0.2464 (5)  | −0.0739 (3)   | 0.0705 (15)* | 0.709 (5) |
| C22  | 0.2779 (4)   | 0.1615 (5)  | −0.2754 (4)   | 0.110 (2)    | 0.709 (5) |
| H19  | 0.3012 (4)   | 0.1391 (5)  | −0.3332 (4)   | 0.132 (3)*   | 0.709 (5) |
| C24  | 0.2772 (4)   | 0.1291 (4)  | −0.1217 (4)   | 0.0918 (15)  | 0.709 (5) |
| H21  | 0.2995 (4)   | 0.0797 (4)  | −0.0575 (4)   | 0.1102 (18)* | 0.709 (5) |
| C23  | 0.3056 (3)   | 0.1002 (4)  | −0.1957 (6)   | 0.123 (2)    | 0.709 (5) |
| H20  | 0.3510 (3)   | 0.0270 (4)  | −0.1896 (6)   | 0.147 (3)*   | 0.709 (5) |
| F1a  | 0.2888 (8)   | 0.1503 (10) | −0.3577 (7)   | 0.103 (5)    | 0.171 (5) |
| F1b  | 0.3763 (8)   | 0.0055 (10) | −0.1199 (8)   | 0.076 (3)    | 0.12      |
| C21b | 0.3116 (15)  | 0.0858 (17) | −0.1527 (11)  | 0.059 (6)    | 0.12      |
| C26b | 0.2997 (16)  | 0.1146 (17) | −0.2420 (12)  | 0.074 (8)    | 0.12      |
| H23b | 0.3369 (16)  | 0.0693 (17) | −0.2809 (12)  | 0.089 (9)*   | 0.12      |
| C25b | 0.2404 (15)  | 0.2011 (16) | −0.2820 (13)  | 0.045 (5)    | 0.12      |
| H22b | 0.2292 (15)  | 0.2255 (16) | −0.3539 (13)  | 0.054 (5)*   | 0.12      |
| C24b | 0.1954 (18)  | 0.2568 (19) | −0.2327 (15)  | 0.048 (6)    | 0.12      |
| H21b | 0.1493 (18)  | 0.3275 (19) | −0.2650 (15)  | 0.058 (7)*   | 0.12      |
| C23b | 0.206 (2)    | 0.226 (2)   | −0.1429 (17)  | 0.038 (5)    | 0.12      |
| H20b | 0.167 (2)    | 0.269 (2)   | −0.1049 (17)  | 0.045 (6)*   | 0.12      |
| C22b | 0.2683 (18)  | 0.141 (2)   | −0.1020 (13)  | 0.039 (4)    | 0.12      |
| H19b | 0.2822 (18)  | 0.118 (2)   | −0.0293 (13)  | 0.046 (5)*   | 0.12      |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1 | 0.03004 (11) | 0.02466 (11) | 0.02421 (10) | 0.00167 (7)  | 0.00828 (8) | −0.00133 (7) |
| S2  | 0.0417 (2)   | 0.0392 (2)   | 0.0670 (3)   | 0.01269 (17) | 0.0231 (2)  | 0.01133 (19) |
| S1  | 0.1035 (5)   | 0.0710 (4)   | 0.0463 (3)   | −0.0483 (3)  | 0.0457 (3)  | −0.0266 (2)  |
| N4  | 0.0278 (5)   | 0.0226 (5)   | 0.0278 (5)   | 0.0037 (4)   | 0.0079 (4)  | −0.0006 (4)  |
| N5  | 0.0320 (6)   | 0.0237 (5)   | 0.0249 (5)   | 0.0025 (4)   | 0.0072 (4)  | −0.0005 (4)  |
| N6  | 0.0312 (6)   | 0.0253 (5)   | 0.0257 (5)   | 0.0009 (4)   | 0.0068 (4)  | 0.0008 (4)   |

|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| N3   | 0.0316 (6)  | 0.0397 (7)  | 0.0257 (5)  | 0.0041 (5)   | 0.0109 (5)  | −0.0019 (5)  |
| N1   | 0.0510 (8)  | 0.0350 (6)  | 0.0353 (7)  | −0.0067 (6)  | 0.0203 (6)  | −0.0048 (5)  |
| N2   | 0.0460 (8)  | 0.0389 (7)  | 0.0420 (7)  | 0.0094 (6)   | 0.0091 (6)  | 0.0069 (6)   |
| C19  | 0.0323 (6)  | 0.0237 (6)  | 0.0268 (6)  | −0.0003 (5)  | 0.0092 (5)  | −0.0014 (5)  |
| C18  | 0.0399 (8)  | 0.0272 (7)  | 0.0363 (7)  | −0.0042 (6)  | 0.0109 (6)  | −0.0043 (5)  |
| C13  | 0.0277 (6)  | 0.0250 (6)  | 0.0298 (6)  | 0.0006 (5)   | 0.0085 (5)  | −0.0004 (5)  |
| C12  | 0.0287 (7)  | 0.0331 (7)  | 0.0374 (7)  | −0.0015 (6)  | 0.0068 (6)  | 0.0012 (6)   |
| C9   | 0.0411 (8)  | 0.0239 (6)  | 0.0293 (6)  | 0.0016 (6)   | 0.0081 (6)  | 0.0006 (5)   |
| C8   | 0.0394 (8)  | 0.0369 (8)  | 0.0283 (6)  | 0.0056 (6)   | 0.0096 (6)  | 0.0074 (6)   |
| C7   | 0.0309 (6)  | 0.0417 (8)  | 0.0225 (6)  | −0.0034 (6)  | 0.0084 (5)  | −0.0008 (5)  |
| C10  | 0.0486 (9)  | 0.0269 (7)  | 0.0357 (7)  | −0.0063 (6)  | 0.0101 (6)  | −0.0011 (6)  |
| C14  | 0.0279 (6)  | 0.0262 (7)  | 0.0411 (8)  | 0.0039 (5)   | 0.0096 (6)  | −0.0028 (6)  |
| C2   | 0.0330 (7)  | 0.0323 (7)  | 0.0372 (7)  | 0.0062 (6)   | 0.0116 (6)  | 0.0059 (6)   |
| C20  | 0.0329 (7)  | 0.0233 (6)  | 0.0396 (7)  | 0.0033 (5)   | 0.0087 (6)  | −0.0055 (5)  |
| C11  | 0.0375 (8)  | 0.0351 (8)  | 0.0379 (8)  | −0.0089 (6)  | 0.0068 (6)  | −0.0021 (6)  |
| C15  | 0.0333 (7)  | 0.0362 (8)  | 0.0336 (7)  | 0.0029 (6)   | 0.0044 (6)  | 0.0036 (6)   |
| C17  | 0.0406 (8)  | 0.0379 (8)  | 0.0413 (8)  | −0.0115 (6)  | 0.0099 (7)  | −0.0075 (6)  |
| C1   | 0.0481 (8)  | 0.0354 (7)  | 0.0301 (7)  | −0.0142 (6)  | 0.0169 (6)  | −0.0078 (6)  |
| C16  | 0.0337 (7)  | 0.0449 (9)  | 0.0386 (8)  | −0.0053 (7)  | 0.0037 (6)  | −0.0017 (7)  |
| C3   | 0.0391 (8)  | 0.0622 (11) | 0.0380 (8)  | 0.0152 (8)   | 0.0152 (7)  | −0.0057 (8)  |
| C6   | 0.0440 (9)  | 0.0746 (13) | 0.0288 (7)  | −0.0046 (8)  | 0.0160 (6)  | 0.0057 (8)   |
| C24a | 0.058 (2)   | 0.071 (3)   | 0.0540 (18) | −0.0212 (17) | 0.0280 (17) | −0.0221 (16) |
| C26  | 0.0423 (13) | 0.0682 (19) | 0.072 (2)   | −0.0102 (12) | 0.0260 (13) | −0.0221 (17) |
| C4   | 0.0439 (9)  | 0.0988 (17) | 0.0462 (10) | 0.0133 (10)  | 0.0234 (8)  | −0.0135 (11) |
| C5   | 0.0473 (10) | 0.1108 (18) | 0.0381 (9)  | −0.0004 (11) | 0.0259 (8)  | −0.0033 (11) |
| C26a | 0.071 (2)   | 0.123 (4)   | 0.0555 (17) | −0.048 (2)   | 0.0304 (15) | −0.035 (2)   |
| C23a | 0.067 (2)   | 0.068 (2)   | 0.134 (4)   | −0.0179 (18) | 0.026 (3)   | −0.016 (3)   |
| C21a | 0.100 (3)   | 0.121 (4)   | 0.151 (5)   | −0.057 (3)   | 0.095 (3)   | −0.094 (4)   |
| F1   | 0.163 (3)   | 0.288 (5)   | 0.0633 (16) | −0.122 (4)   | 0.0274 (18) | 0.012 (2)    |
| C22a | 0.077 (3)   | 0.060 (2)   | 0.252 (8)   | −0.0152 (19) | 0.084 (4)   | −0.057 (4)   |
| C21  | 0.071 (2)   | 0.123 (4)   | 0.0555 (17) | −0.048 (2)   | 0.0304 (15) | −0.035 (2)   |
| C25a | 0.0423 (13) | 0.0682 (19) | 0.072 (2)   | −0.0102 (12) | 0.0260 (13) | −0.0221 (17) |
| C25  | 0.058 (2)   | 0.071 (3)   | 0.0540 (18) | −0.0212 (17) | 0.0280 (17) | −0.0221 (16) |
| C22  | 0.100 (3)   | 0.121 (4)   | 0.151 (5)   | −0.057 (3)   | 0.095 (3)   | −0.094 (4)   |
| C24  | 0.067 (2)   | 0.068 (2)   | 0.134 (4)   | −0.0179 (18) | 0.026 (3)   | −0.016 (3)   |
| C23  | 0.077 (3)   | 0.060 (2)   | 0.252 (8)   | −0.0152 (19) | 0.084 (4)   | −0.057 (4)   |
| F1a  | 0.115 (9)   | 0.124 (9)   | 0.086 (7)   | −0.020 (7)   | 0.056 (6)   | −0.046 (6)   |
| F1b  | 0.086 (7)   | 0.074 (6)   | 0.076 (6)   | 0.026 (5)    | 0.038 (5)   | 0.005 (4)    |
| C21b | 0.076 (10)  | 0.069 (9)   | 0.044 (6)   | 0.020 (6)    | 0.035 (4)   | 0.002 (4)    |
| C26b | 0.108 (14)  | 0.072 (11)  | 0.057 (7)   | 0.042 (7)    | 0.048 (5)   | 0.010 (4)    |
| C25b | 0.062 (9)   | 0.042 (8)   | 0.042 (7)   | 0.006 (5)    | 0.032 (4)   | −0.001 (4)   |
| C24b | 0.053 (9)   | 0.055 (9)   | 0.044 (8)   | 0.001 (5)    | 0.025 (4)   | −0.011 (5)   |
| C23b | 0.034 (8)   | 0.045 (9)   | 0.038 (8)   | −0.010 (5)   | 0.018 (4)   | −0.015 (4)   |
| C22b | 0.038 (7)   | 0.047 (8)   | 0.034 (5)   | −0.007 (4)   | 0.017 (4)   | −0.013 (4)   |

*Geometric parameters (Å, °)*

|           |             |            |             |
|-----------|-------------|------------|-------------|
| Fe1—N4    | 2.2398 (11) | C16—H8     | 1.1030      |
| Fe1—N5    | 2.1993 (12) | C3—H1      | 1.1030      |
| Fe1—N6    | 2.1941 (12) | C3—C4      | 1.382 (3)   |
| Fe1—N3    | 2.1930 (12) | C6—H4      | 1.1030      |
| Fe1—N1    | 2.1078 (14) | C6—C5      | 1.383 (3)   |
| Fe1—N2    | 2.0538 (14) | C24a—H21a  | 1.1030      |
| S2—C2     | 1.6202 (15) | C24a—C23a  | 1.371 (6)   |
| S1—C1     | 1.6138 (19) | C24a—C25a  | 1.365 (5)   |
| N4—C8     | 1.4790 (18) | C26—H23    | 1.1030      |
| N4—C14    | 1.4721 (17) | C26—C21    | 1.351 (4)   |
| N4—C20    | 1.4774 (17) | C26—C25    | 1.365 (5)   |
| N5—C13    | 1.3427 (17) | C4—H2      | 1.1030      |
| N5—C9     | 1.3410 (18) | C4—C5      | 1.386 (3)   |
| N6—C19    | 1.3392 (17) | C5—H3      | 1.1030      |
| N6—C15    | 1.3396 (18) | C26a—H23a  | 1.1030      |
| N3—C7     | 1.3354 (19) | C26a—C21a  | 1.363 (7)   |
| N3—C3     | 1.3432 (19) | C26a—C25a  | 1.351 (4)   |
| N1—C1     | 1.160 (2)   | C23a—H20a  | 1.1030      |
| N2—C2     | 1.165 (2)   | C23a—C22a  | 1.399 (8)   |
| C19—C18   | 1.3926 (19) | C21a—C22a  | 1.369 (8)   |
| C19—C20   | 1.5036 (19) | C21a—F1a   | 1.345 (10)  |
| C18—H10   | 1.1030      | F1—C21     | 1.261 (5)   |
| C18—C17   | 1.384 (2)   | C22a—H19a  | 1.1030      |
| C13—C12   | 1.3919 (19) | C21—C22    | 1.363 (7)   |
| C13—C14   | 1.5066 (19) | C25a—H22a  | 1.1030      |
| C12—H16   | 1.1030      | C25—H22    | 1.1030      |
| C12—C11   | 1.388 (2)   | C25—C24    | 1.371 (6)   |
| C9—H13    | 1.1030      | C22—H19    | 1.1030      |
| C9—C10    | 1.389 (2)   | C22—C23    | 1.369 (8)   |
| C8—H5     | 1.1110      | C24—H21    | 1.1030      |
| C8—H6     | 1.1110      | C24—C23    | 1.399 (8)   |
| C8—C7     | 1.507 (2)   | C23—H20    | 1.1030      |
| C7—C6     | 1.391 (2)   | F1b—C21b   | 1.346 (15)  |
| C10—H14   | 1.1030      | C21b—C26b  | 1.371 (13)  |
| C10—C11   | 1.388 (2)   | C21b—C22b  | 1.360 (13)  |
| C14—H18   | 1.1110      | C26b—H23b  | 1.1030      |
| C14—H17   | 1.1110      | C26b—C25b  | 1.373 (13)  |
| C20—H11   | 1.1110      | C25b—H22b  | 1.1030      |
| C20—H12   | 1.1110      | C25b—C24b  | 1.365 (13)  |
| C11—H15   | 1.1030      | C24b—H21b  | 1.1030      |
| C15—H7    | 1.1030      | C24b—C23b  | 1.387 (14)  |
| C15—C16   | 1.385 (2)   | C23b—H20b  | 1.1030      |
| C17—H9    | 1.1030      | C23b—C22b  | 1.386 (14)  |
| C17—C16   | 1.389 (2)   | C22b—H19b  | 1.1030      |
| N5—Fe1—N4 | 75.97 (4)   | C16—C15—N6 | 122.75 (14) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N6—Fe1—N4   | 75.71 (4)   | C16—C15—H7     | 118.62 (9)  |
| N6—Fe1—N5   | 151.67 (4)  | H9—C17—C18     | 120.48 (9)  |
| N3—Fe1—N4   | 78.10 (4)   | C16—C17—C18    | 119.04 (14) |
| N3—Fe1—N5   | 89.57 (4)   | C16—C17—H9     | 120.48 (9)  |
| N3—Fe1—N6   | 83.94 (4)   | N1—C1—S1       | 178.48 (16) |
| N1—Fe1—N4   | 91.66 (5)   | C17—C16—C15    | 118.51 (14) |
| N1—Fe1—N5   | 91.89 (5)   | H8—C16—C15     | 120.75 (9)  |
| N1—Fe1—N6   | 89.64 (5)   | H8—C16—C17     | 120.75 (9)  |
| N1—Fe1—N3   | 168.98 (5)  | H1—C3—N3       | 118.61 (10) |
| N2—Fe1—N4   | 171.77 (5)  | C4—C3—N3       | 122.79 (18) |
| N2—Fe1—N5   | 100.68 (5)  | C4—C3—H1       | 118.61 (12) |
| N2—Fe1—N6   | 107.29 (5)  | H4—C6—C7       | 120.47 (11) |
| N2—Fe1—N3   | 94.47 (5)   | C5—C6—C7       | 119.07 (18) |
| N2—Fe1—N1   | 95.99 (6)   | C5—C6—H4       | 120.47 (12) |
| C8—N4—Fe1   | 111.14 (8)  | C23a—C24a—H21a | 119.5 (3)   |
| C14—N4—Fe1  | 105.81 (8)  | C25a—C24a—H21a | 119.5 (3)   |
| C14—N4—C8   | 111.25 (11) | C25a—C24a—C23a | 120.9 (4)   |
| C20—N4—Fe1  | 105.87 (8)  | C21—C26—H23    | 120.1 (3)   |
| C20—N4—C8   | 111.99 (12) | C25—C26—H23    | 120.1 (3)   |
| C20—N4—C14  | 110.48 (11) | C25—C26—C21    | 119.7 (4)   |
| C13—N5—Fe1  | 114.68 (9)  | H2—C4—C3       | 120.79 (12) |
| C9—N5—Fe1   | 126.78 (10) | C5—C4—C3       | 118.41 (18) |
| C9—N5—C13   | 118.53 (12) | C5—C4—H2       | 120.79 (11) |
| C19—N6—Fe1  | 115.17 (9)  | C4—C5—C6       | 119.10 (17) |
| C15—N6—Fe1  | 126.16 (10) | H3—C5—C6       | 120.45 (12) |
| C15—N6—C19  | 118.66 (12) | H3—C5—C4       | 120.45 (11) |
| C7—N3—Fe1   | 116.12 (9)  | C21a—C26a—H23a | 119.1 (3)   |
| C3—N3—Fe1   | 125.01 (11) | C25a—C26a—H23a | 119.1 (3)   |
| C3—N3—C7    | 118.69 (14) | C25a—C26a—C21a | 121.9 (4)   |
| C1—N1—Fe1   | 169.69 (14) | H20a—C23a—C24a | 121.0 (3)   |
| C2—N2—Fe1   | 178.18 (14) | C22a—C23a—C24a | 118.0 (5)   |
| C18—C19—N6  | 122.13 (13) | C22a—C23a—H20a | 121.0 (3)   |
| C20—C19—N6  | 116.07 (11) | C22a—C21a—C26a | 118.5 (4)   |
| C20—C19—C18 | 121.72 (12) | F1a—C21a—C26a  | 107.3 (8)   |
| H10—C18—C19 | 120.56 (9)  | F1a—C21a—C22a  | 134.0 (8)   |
| C17—C18—C19 | 118.87 (14) | C21a—C22a—C23a | 121.0 (4)   |
| C17—C18—H10 | 120.56 (9)  | H19a—C22a—C23a | 119.5 (3)   |
| C12—C13—N5  | 122.07 (13) | H19a—C22a—C21a | 119.5 (3)   |
| C14—C13—N5  | 116.15 (12) | F1—C21—C26     | 122.7 (5)   |
| C14—C13—C12 | 121.73 (12) | C22—C21—C26    | 121.9 (4)   |
| H16—C12—C13 | 120.40 (8)  | C22—C21—F1     | 115.4 (5)   |
| C11—C12—C13 | 119.21 (14) | C26a—C25a—C24a | 119.7 (4)   |
| C11—C12—H16 | 120.40 (9)  | H22a—C25a—C24a | 120.1 (3)   |
| H13—C9—N5   | 118.63 (8)  | H22a—C25a—C26a | 120.1 (3)   |
| C10—C9—N5   | 122.73 (14) | H22—C25—C26    | 119.5 (3)   |
| C10—C9—H13  | 118.63 (9)  | C24—C25—C26    | 120.9 (4)   |
| H5—C8—N4    | 108.44 (7)  | C24—C25—H22    | 119.5 (3)   |
| H6—C8—N4    | 108.44 (8)  | H19—C22—C21    | 120.8 (3)   |



|                |              |                     |              |
|----------------|--------------|---------------------|--------------|
| H6—C8—H5       | 107.5        | C23—C22—C21         | 118.5 (4)    |
| C7—C8—N4       | 115.33 (11)  | C23—C22—H19         | 120.8 (3)    |
| C7—C8—H5       | 108.44 (8)   | H21—C24—C25         | 121.0 (3)    |
| C7—C8—H6       | 108.44 (8)   | C23—C24—C25         | 118.0 (5)    |
| C8—C7—N3       | 118.64 (12)  | C23—C24—H21         | 121.0 (3)    |
| C6—C7—N3       | 121.94 (15)  | C24—C23—C22         | 121.0 (4)    |
| C6—C7—C8       | 119.37 (14)  | H20—C23—C22         | 119.5 (3)    |
| H14—C10—C9     | 120.62 (9)   | H20—C23—C24         | 119.5 (3)    |
| C11—C10—C9     | 118.77 (14)  | C26b—C21b—F1b       | 112.4 (14)   |
| C11—C10—H14    | 120.62 (9)   | C22b—C21b—F1b       | 124.9 (15)   |
| C13—C14—N4     | 111.21 (11)  | C22b—C21b—C26b      | 122.4 (15)   |
| H18—C14—N4     | 109.39 (7)   | H23b—C26b—C21b      | 120.7 (10)   |
| H18—C14—C13    | 109.39 (8)   | C25b—C26b—C21b      | 118.5 (15)   |
| H17—C14—N4     | 109.39 (7)   | C25b—C26b—H23b      | 120.7 (10)   |
| H17—C14—C13    | 109.39 (8)   | H22b—C25b—C26b      | 120.0 (10)   |
| H17—C14—H18    | 108.0        | C24b—C25b—C26b      | 120.1 (14)   |
| N2—C2—S2       | 179.51 (15)  | C24b—C25b—H22b      | 120.0 (10)   |
| C19—C20—N4     | 111.29 (11)  | H21b—C24b—C25b      | 119.4 (10)   |
| H11—C20—N4     | 109.37 (7)   | C23b—C24b—C25b      | 121.2 (16)   |
| H11—C20—C19    | 109.37 (7)   | C23b—C24b—H21b      | 119.4 (11)   |
| H12—C20—N4     | 109.37 (8)   | H20b—C23b—C24b      | 120.8 (11)   |
| H12—C20—C19    | 109.37 (8)   | C22b—C23b—C24b      | 118.5 (16)   |
| H12—C20—H11    | 108.0        | C22b—C23b—H20b      | 120.8 (11)   |
| C10—C11—C12    | 118.68 (14)  | C23b—C22b—C21b      | 119.2 (15)   |
| H15—C11—C12    | 120.66 (9)   | H19b—C22b—C21b      | 120.4 (10)   |
| H15—C11—C10    | 120.66 (9)   | H19b—C22b—C23b      | 120.4 (11)   |
| H7—C15—N6      | 118.62 (9)   |                     |              |
| Fe1—N4—C8—C7   | −7.60 (9)    | C9—N5—C13—C14       | 177.69 (13)  |
| Fe1—N4—C14—C13 | −43.18 (8)   | C8—C7—N3—C3         | 177.81 (14)  |
| Fe1—N4—C20—C19 | 42.60 (8)    | C8—C7—C6—C5         | −177.63 (17) |
| Fe1—N5—C13—C12 | 179.46 (10)  | C7—N3—C3—C4         | −0.58 (18)   |
| Fe1—N5—C13—C14 | −2.91 (10)   | C7—C8—N4—C14        | −125.23 (13) |
| Fe1—N5—C9—C10  | 179.95 (12)  | C7—C8—N4—C20        | 110.58 (13)  |
| Fe1—N6—C19—C18 | 177.92 (10)  | C7—C6—C5—C4         | 0.3 (2)      |
| Fe1—N6—C19—C20 | 1.07 (10)    | C14—C13—C12—C11     | −177.06 (15) |
| Fe1—N6—C15—C16 | −176.95 (12) | C20—C19—N6—C15      | −178.01 (13) |
| Fe1—N3—C7—C8   | −6.72 (11)   | C20—C19—C18—C17     | 175.85 (15)  |
| Fe1—N3—C7—C6   | 175.96 (11)  | C3—N3—C7—C6         | 0.49 (18)    |
| Fe1—N3—C3—C4   | −175.61 (16) | C3—C4—C5—C6         | −0.3 (2)     |
| N4—C8—C7—N3    | 9.80 (15)    | C24a—C23a—C22a—C21a | −0.0 (4)     |
| N4—C8—C7—C6    | −172.81 (13) | C24a—C25a—C26a—C21a | 0.9 (4)      |
| N4—C14—C13—N5  | 32.43 (13)   | C26—C21—C22—C23     | 0.4 (4)      |
| N4—C14—C13—C12 | −149.92 (12) | C26—C25—C24—C23     | 1.3 (6)      |
| N4—C20—C19—N6  | −30.76 (14)  | C26a—C21a—C22a—C23a | −0.8 (5)     |
| N4—C20—C19—C18 | 152.37 (12)  | C26a—C25a—C24a—C23a | −1.8 (4)     |
| N5—C13—C12—C11 | 0.44 (16)    | C23a—C22a—C21a—F1a  | −175.0 (7)   |
| N5—C9—C10—C11  | 0.87 (17)    | F1—C21—C26—C25      | 179.6 (4)    |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N6—C19—C18—C17  | −0.83 (16)   | F1—C21—C22—C23      | −178.4 (3)  |
| N6—C15—C16—C17  | −0.85 (19)   | C22a—C23a—C24a—C25a | 1.3 (6)     |
| N3—C7—C6—C5     | −0.34 (19)   | C22a—C21a—C26a—C25a | 0.4 (4)     |
| N3—C3—C4—C5     | 0.5 (2)      | C21—C26—C25—C24     | −1.8 (4)    |
| C19—N6—C15—C16  | 2.01 (15)    | C21—C22—C23—C24     | −0.8 (5)    |
| C19—C18—C17—C16 | 1.97 (17)    | C25a—C26a—C21a—F1a  | 176.0 (6)   |
| C19—C20—N4—C8   | −78.67 (13)  | C25—C26—C21—C22     | 0.9 (4)     |
| C19—C20—N4—C14  | 156.72 (12)  | C25—C24—C23—C22     | −0.0 (4)    |
| C18—C19—N6—C15  | −1.15 (17)   | F1b—C21b—C26b—C25b  | −175.5 (13) |
| C18—C17—C16—C15 | −1.18 (19)   | F1b—C21b—C22b—C23b  | 176.6 (18)  |
| C13—N5—C9—C10   | −0.72 (15)   | C21b—C26b—C25b—C24b | 1 (3)       |
| C13—C12—C11—C10 | −0.28 (17)   | C21b—C22b—C23b—C24b | −4 (3)      |
| C13—C14—N4—C8   | 77.63 (13)   | C26b—C21b—C22b—C23b | 3 (2)       |
| C13—C14—N4—C20  | −157.33 (12) | C26b—C25b—C24b—C23b | −2 (2)      |
| C12—C13—N5—C9   | 0.05 (17)    | C25b—C26b—C21b—C22b | −1 (3)      |
| C12—C11—C10—C9  | −0.34 (19)   | C25b—C24b—C23b—C22b | 3 (3)       |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_190K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.2839$  (3) Å

$b = 12.0065$  (2) Å

$c = 15.4519$  (3) Å

$\beta = 110.423$  (2)°

$V = 2657.28$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.396$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 56804 reflections

$\theta = 2.1$ – $32.5^\circ$

$\mu = 0.76$  mm<sup>−1</sup>

$T = 190$  K

Block, yellow

$0.31 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.660$ ,  $T_{\max} = 1.000$

97972 measured reflections

6584 independent reflections

6118 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.090$

$S = 1.05$

6584 reflections

408 parameters

149 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.9148P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.80$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.58$  e Å<sup>−3</sup>

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | x             | y             | z             | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Fe1 | 0.655768 (13) | 0.329796 (15) | 0.802726 (12) | 0.02758 (7)                      |           |
| S2  | 0.47149 (3)   | 0.00253 (4)   | 0.67425 (3)   | 0.05010 (11)                     |           |
| S1  | 0.76476 (10)  | 0.34377 (11)  | 0.54270 (8)   | 0.07183 (19)                     |           |
| N4  | 0.74100 (7)   | 0.47161 (9)   | 0.88380 (7)   | 0.0278 (2)                       |           |
| N5  | 0.78651 (8)   | 0.25084 (9)   | 0.89113 (7)   | 0.0290 (2)                       |           |
| N6  | 0.56579 (8)   | 0.47652 (9)   | 0.75502 (7)   | 0.0298 (2)                       |           |
| N3  | 0.60873 (8)   | 0.33847 (10)  | 0.92164 (8)   | 0.0339 (2)                       |           |
| N1  | 0.70874 (10)  | 0.35372 (11)  | 0.69489 (9)   | 0.0405 (3)                       |           |
| N2  | 0.57674 (10)  | 0.19209 (12)  | 0.74633 (10)  | 0.0455 (3)                       |           |
| C19 | 0.60986 (9)   | 0.57476 (11)  | 0.77403 (8)   | 0.0293 (2)                       |           |
| C18 | 0.56318 (11)  | 0.67549 (11)  | 0.74603 (10)  | 0.0369 (3)                       |           |
| H10 | 0.60106 (11)  | 0.75527 (11)  | 0.76346 (10)  | 0.0443 (4)*                      |           |
| C13 | 0.85766 (9)   | 0.32156 (10)  | 0.92827 (9)   | 0.0294 (3)                       |           |
| C12 | 0.94504 (9)   | 0.28562 (12)  | 0.98643 (10)  | 0.0360 (3)                       |           |
| H16 | 1.00210 (9)   | 0.34589 (12)  | 1.01634 (10)  | 0.0432 (3)*                      |           |
| C9  | 0.80048 (10)  | 0.14216 (11)  | 0.91106 (9)   | 0.0340 (3)                       |           |
| H13 | 0.74175 (10)  | 0.08403 (11)  | 0.88124 (9)   | 0.0408 (3)*                      |           |
| C8  | 0.72429 (10)  | 0.48792 (12)  | 0.97158 (9)   | 0.0372 (3)                       |           |
| H5  | 0.78972 (10)  | 0.46804 (12)  | 1.02955 (9)   | 0.0446 (4)*                      |           |
| H6  | 0.70857 (10)  | 0.57733 (12)  | 0.97758 (9)   | 0.0446 (4)*                      |           |
| C2  | 0.53291 (10)  | 0.11262 (12)  | 0.71665 (10)  | 0.0356 (3)                       |           |
| C7  | 0.64631 (9)   | 0.41931 (12)  | 0.98262 (9)   | 0.0336 (3)                       |           |
| C14 | 0.83903 (9)   | 0.44198 (11)  | 0.90064 (10)  | 0.0340 (3)                       |           |
| H18 | 0.85532 (9)   | 0.45736 (11)  | 0.83699 (10)  | 0.0407 (3)*                      |           |
| H17 | 0.88545 (9)   | 0.49576 (11)  | 0.95631 (10)  | 0.0407 (3)*                      |           |
| C10 | 0.88560 (11)  | 0.09975 (12)  | 0.96754 (10)  | 0.0399 (3)                       |           |
| H14 | 0.89457 (11)  | 0.00958 (12)  | 0.98149 (10)  | 0.0478 (4)*                      |           |
| C20 | 0.71373 (10)  | 0.57014 (11)  | 0.82316 (10)  | 0.0345 (3)                       |           |
| H11 | 0.73703 (10)  | 0.64682 (11)  | 0.86517 (10)  | 0.0415 (3)*                      |           |
| H12 | 0.74911 (10)  | 0.56728 (11)  | 0.77143 (10)  | 0.0415 (3)*                      |           |
| C11 | 0.95933 (11)  | 0.17275 (12)  | 1.00623 (11)  | 0.0404 (3)                       |           |
| H15 | 1.02790 (11)  | 0.14182 (12)  | 1.05177 (11)  | 0.0485 (4)*                      |           |
| C15 | 0.47359 (10)  | 0.47553 (13)  | 0.70886 (10)  | 0.0378 (3)                       |           |
| H7  | 0.43698 (10)  | 0.39473 (13)  | 0.69526 (10)  | 0.0454 (4)*                      |           |
| C17 | 0.46828 (11)  | 0.67349 (13)  | 0.69590 (11)  | 0.0429 (3)                       |           |
| H9  | 0.42980 (11)  | 0.75164 (13)  | 0.67095 (11)  | 0.0515 (4)*                      |           |
| C1  | 0.73227 (11)  | 0.35096 (12)  | 0.63134 (10)  | 0.0386 (3)                       |           |
| C16 | 0.42229 (11)  | 0.57167 (14)  | 0.67750 (10)  | 0.0434 (3)                       |           |
| H8  | 0.34658 (11)  | 0.56751 (14)  | 0.63876 (10)  | 0.0520 (4)*                      |           |
| C3  | 0.54151 (11)  | 0.27479 (16)  | 0.93319 (11)  | 0.0489 (4)                       |           |
| H1  | 0.51008 (11)  | 0.20860 (16)  | 0.88232 (11)  | 0.0587 (5)*                      |           |
| C6  | 0.61816 (12)  | 0.43854 (18)  | 1.05775 (10)  | 0.0510 (4)                       |           |
| H4  | 0.65063 (12)  | 0.50559 (18)  | 1.10736 (10)  | 0.0612 (5)*                      |           |
| C25 | 0.7796 (4)    | 0.2202 (5)    | 1.1323 (3)    | 0.0651 (13)                      | 0.696 (5) |
| H22 | 0.8020 (4)    | 0.2455 (5)    | 1.0746 (3)    | 0.0781 (16)*                     | 0.696 (5) |

|      |              |             |              |              |           |
|------|--------------|-------------|--------------|--------------|-----------|
| C26  | 0.8086 (2)   | 0.2806 (3)  | 1.2122 (3)   | 0.0642 (8)   | 0.696 (5) |
| H23  | 0.8554 (2)   | 0.3526 (3)  | 1.2196 (3)   | 0.0770 (10)* | 0.696 (5) |
| C4   | 0.50996 (13) | 0.2887 (2)  | 1.00631 (13) | 0.0645 (6)   |           |
| H2   | 0.45503 (13) | 0.2342 (2)  | 1.01406 (13) | 0.0773 (7)*  |           |
| C5   | 0.54904 (13) | 0.3726 (2)  | 1.06951 (13) | 0.0662 (6)   |           |
| H3   | 0.52532 (13) | 0.3867 (2)  | 1.12835 (13) | 0.0794 (7)*  |           |
| C21  | 0.7794 (3)   | 0.2514 (4)  | 1.2822 (2)   | 0.0853 (11)  | 0.696 (5) |
| C24  | 0.7235 (3)   | 0.1290 (4)  | 1.1223 (4)   | 0.0985 (16)  | 0.696 (5) |
| H21  | 0.7017 (3)   | 0.0795 (4)  | 1.0582 (4)   | 0.1182 (19)* | 0.696 (5) |
| C22  | 0.7223 (4)   | 0.1618 (5)  | 1.2754 (4)   | 0.116 (2)    | 0.696 (5) |
| H19  | 0.6990 (4)   | 0.1392 (5)  | 1.3331 (4)   | 0.139 (3)*   | 0.696 (5) |
| C23  | 0.6946 (3)   | 0.1005 (4)  | 1.1955 (6)   | 0.131 (2)    | 0.696 (5) |
| H20  | 0.6488 (3)   | 0.0276 (4)  | 1.1891 (6)   | 0.158 (3)*   | 0.696 (5) |
| F1   | 0.8038 (3)   | 0.3017 (4)  | 1.3575 (2)   | 0.191 (3)    | 0.696 (5) |
| C21a | 0.7223 (4)   | 0.1618 (5)  | 1.2754 (4)   | 0.116 (2)    | 0.184 (5) |
| C22a | 0.6946 (3)   | 0.1005 (4)  | 1.1955 (6)   | 0.131 (2)    | 0.184 (5) |
| H19a | 0.6488 (3)   | 0.0276 (4)  | 1.1891 (6)   | 0.158 (3)*   | 0.184 (5) |
| C23a | 0.7235 (3)   | 0.1290 (4)  | 1.1223 (4)   | 0.0985 (16)  | 0.184 (5) |
| H20a | 0.7017 (3)   | 0.0795 (4)  | 1.0582 (4)   | 0.1182 (19)* | 0.184 (5) |
| C24a | 0.7796 (4)   | 0.2202 (5)  | 1.1323 (3)   | 0.0651 (13)  | 0.184 (5) |
| H21a | 0.8020 (4)   | 0.2455 (5)  | 1.0746 (3)   | 0.0781 (16)* | 0.184 (5) |
| C25a | 0.8086 (2)   | 0.2806 (3)  | 1.2122 (3)   | 0.0642 (8)   | 0.184 (5) |
| H22a | 0.8554 (2)   | 0.3526 (3)  | 1.2196 (3)   | 0.0770 (10)* | 0.184 (5) |
| C26a | 0.7794 (3)   | 0.2514 (4)  | 1.2822 (2)   | 0.0853 (11)  | 0.184 (5) |
| H23a | 0.8022 (3)   | 0.3011 (4)  | 1.3462 (2)   | 0.1023 (13)* | 0.184 (5) |
| F1a  | 0.7111 (8)   | 0.1505 (9)  | 1.3573 (6)   | 0.108 (4)    | 0.184 (5) |
| F2   | 0.6250 (8)   | 0.0059 (9)  | 1.1195 (8)   | 0.077 (3)    | 0.12      |
| C21b | 0.6890 (14)  | 0.0861 (16) | 1.1523 (10)  | 0.062 (6)    | 0.12      |
| C26b | 0.7011 (15)  | 0.1129 (16) | 1.2418 (11)  | 0.069 (7)    | 0.12      |
| H23b | 0.6650 (15)  | 0.0660 (16) | 1.2808 (11)  | 0.082 (8)*   | 0.12      |
| C25b | 0.7594 (14)  | 0.1997 (14) | 1.2815 (12)  | 0.045 (4)    | 0.12      |
| H22b | 0.7694 (14)  | 0.2247 (14) | 1.3530 (12)  | 0.054 (5)*   | 0.12      |
| C24b | 0.8053 (16)  | 0.2555 (17) | 1.2330 (13)  | 0.047 (5)    | 0.12      |
| H21b | 0.8538 (16)  | 0.3233 (17) | 1.2667 (13)  | 0.056 (6)*   | 0.12      |
| C23b | 0.7918 (18)  | 0.228 (2)   | 1.1421 (16)  | 0.036 (5)    | 0.12      |
| H20b | 0.8292 (18)  | 0.273 (2)   | 1.1033 (16)  | 0.044 (6)*   | 0.12      |
| C22b | 0.7304 (16)  | 0.1430 (19) | 1.1013 (13)  | 0.042 (4)    | 0.12      |
| H19b | 0.7152 (16)  | 0.1217 (19) | 1.0280 (13)  | 0.050 (5)*   | 0.12      |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.03134 (11) | 0.02513 (10) | 0.02506 (10) | −0.00224 (6)  | 0.00830 (7)  | 0.00058 (6)   |
| S2  | 0.0436 (2)   | 0.0399 (2)   | 0.0700 (3)   | −0.01407 (16) | 0.02387 (19) | −0.01268 (18) |
| S1  | 0.1092 (5)   | 0.0749 (3)   | 0.0480 (3)   | 0.0507 (3)    | 0.0484 (3)   | 0.0278 (2)    |
| N4  | 0.0291 (5)   | 0.0234 (5)   | 0.0293 (5)   | −0.0037 (4)   | 0.0082 (4)   | 0.0004 (4)    |
| N5  | 0.0333 (5)   | 0.0247 (5)   | 0.0269 (5)   | −0.0022 (4)   | 0.0078 (4)   | 0.0008 (4)    |
| N6  | 0.0323 (5)   | 0.0268 (5)   | 0.0274 (5)   | −0.0005 (4)   | 0.0070 (4)   | −0.0008 (4)   |

|      |             |             |             |              |             |             |
|------|-------------|-------------|-------------|--------------|-------------|-------------|
| N3   | 0.0332 (5)  | 0.0419 (6)  | 0.0276 (5)  | −0.0049 (5)  | 0.0117 (4)  | 0.0022 (4)  |
| N1   | 0.0526 (7)  | 0.0376 (6)  | 0.0358 (6)  | 0.0067 (5)   | 0.0211 (6)  | 0.0043 (5)  |
| N2   | 0.0479 (7)  | 0.0388 (7)  | 0.0445 (7)  | −0.0114 (6)  | 0.0095 (6)  | −0.0085 (6) |
| C19  | 0.0347 (6)  | 0.0246 (6)  | 0.0277 (6)  | 0.0003 (5)   | 0.0096 (5)  | 0.0016 (4)  |
| C18  | 0.0409 (7)  | 0.0292 (7)  | 0.0384 (7)  | 0.0043 (5)   | 0.0109 (6)  | 0.0043 (5)  |
| C13  | 0.0293 (6)  | 0.0262 (6)  | 0.0312 (6)  | −0.0007 (5)  | 0.0088 (5)  | 0.0006 (5)  |
| C12  | 0.0304 (6)  | 0.0335 (7)  | 0.0399 (7)  | 0.0020 (5)   | 0.0070 (5)  | −0.0010 (6) |
| C9   | 0.0427 (7)  | 0.0249 (6)  | 0.0310 (6)  | −0.0012 (5)  | 0.0086 (5)  | −0.0009 (5) |
| C8   | 0.0420 (7)  | 0.0378 (7)  | 0.0299 (6)  | −0.0052 (6)  | 0.0102 (5)  | −0.0074 (5) |
| C2   | 0.0348 (6)  | 0.0331 (7)  | 0.0383 (7)  | −0.0067 (5)  | 0.0121 (5)  | −0.0062 (5) |
| C7   | 0.0322 (6)  | 0.0441 (7)  | 0.0240 (6)  | 0.0035 (5)   | 0.0090 (5)  | 0.0012 (5)  |
| C14  | 0.0292 (6)  | 0.0271 (6)  | 0.0433 (7)  | −0.0038 (5)  | 0.0098 (5)  | 0.0030 (5)  |
| C10  | 0.0496 (8)  | 0.0282 (6)  | 0.0374 (7)  | 0.0067 (6)   | 0.0097 (6)  | 0.0009 (5)  |
| C20  | 0.0346 (6)  | 0.0245 (6)  | 0.0412 (7)  | −0.0036 (5)  | 0.0090 (5)  | 0.0056 (5)  |
| C11  | 0.0383 (7)  | 0.0366 (8)  | 0.0408 (7)  | 0.0096 (6)   | 0.0069 (6)  | 0.0023 (6)  |
| C15  | 0.0345 (7)  | 0.0372 (7)  | 0.0356 (7)  | −0.0027 (6)  | 0.0044 (5)  | −0.0032 (6) |
| C17  | 0.0423 (8)  | 0.0393 (8)  | 0.0433 (8)  | 0.0115 (6)   | 0.0101 (6)  | 0.0075 (6)  |
| C1   | 0.0510 (8)  | 0.0369 (7)  | 0.0304 (7)  | 0.0149 (6)   | 0.0176 (6)  | 0.0079 (5)  |
| C16  | 0.0350 (7)  | 0.0465 (8)  | 0.0407 (7)  | 0.0058 (6)   | 0.0032 (6)  | 0.0015 (6)  |
| C3   | 0.0413 (8)  | 0.0660 (11) | 0.0411 (8)  | −0.0161 (7)  | 0.0164 (6)  | 0.0059 (7)  |
| C6   | 0.0469 (8)  | 0.0784 (12) | 0.0305 (7)  | 0.0048 (8)   | 0.0170 (6)  | −0.0064 (7) |
| C25  | 0.066 (3)   | 0.078 (3)   | 0.0597 (18) | 0.0236 (19)  | 0.0330 (18) | 0.0238 (17) |
| C26  | 0.0456 (12) | 0.071 (2)   | 0.081 (2)   | 0.0107 (12)  | 0.0283 (13) | 0.0252 (17) |
| C4   | 0.0466 (9)  | 0.1043 (17) | 0.0495 (10) | −0.0137 (10) | 0.0255 (8)  | 0.0146 (11) |
| C5   | 0.0512 (10) | 0.1161 (18) | 0.0408 (9)  | 0.0003 (11)  | 0.0280 (8)  | 0.0037 (10) |
| C21  | 0.077 (2)   | 0.127 (3)   | 0.0585 (17) | 0.051 (2)    | 0.0317 (15) | 0.033 (2)   |
| C24  | 0.072 (2)   | 0.070 (2)   | 0.144 (5)   | 0.0188 (18)  | 0.027 (3)   | 0.020 (3)   |
| C22  | 0.105 (3)   | 0.124 (4)   | 0.161 (5)   | 0.058 (3)    | 0.100 (4)   | 0.099 (4)   |
| C23  | 0.081 (3)   | 0.064 (2)   | 0.268 (8)   | 0.0158 (19)  | 0.086 (4)   | 0.060 (4)   |
| F1   | 0.178 (4)   | 0.315 (6)   | 0.0708 (18) | 0.133 (4)    | 0.0305 (19) | −0.013 (2)  |
| C21a | 0.105 (3)   | 0.124 (4)   | 0.161 (5)   | 0.058 (3)    | 0.100 (4)   | 0.099 (4)   |
| C22a | 0.081 (3)   | 0.064 (2)   | 0.268 (8)   | 0.0158 (19)  | 0.086 (4)   | 0.060 (4)   |
| C23a | 0.072 (2)   | 0.070 (2)   | 0.144 (5)   | 0.0188 (18)  | 0.027 (3)   | 0.020 (3)   |
| C24a | 0.066 (3)   | 0.078 (3)   | 0.0597 (18) | 0.0236 (19)  | 0.0330 (18) | 0.0238 (17) |
| C25a | 0.0456 (12) | 0.071 (2)   | 0.081 (2)   | 0.0107 (12)  | 0.0283 (13) | 0.0252 (17) |
| C26a | 0.077 (2)   | 0.127 (3)   | 0.0585 (17) | 0.051 (2)    | 0.0317 (15) | 0.033 (2)   |
| F1a  | 0.126 (8)   | 0.125 (8)   | 0.092 (7)   | 0.018 (6)    | 0.062 (6)   | 0.043 (6)   |
| F2   | 0.087 (6)   | 0.069 (6)   | 0.082 (6)   | −0.025 (4)   | 0.040 (4)   | −0.007 (4)  |
| C21b | 0.080 (10)  | 0.066 (9)   | 0.051 (6)   | −0.021 (5)   | 0.037 (4)   | −0.004 (4)  |
| C26b | 0.102 (13)  | 0.056 (10)  | 0.061 (6)   | −0.035 (6)   | 0.046 (5)   | −0.006 (4)  |
| C25b | 0.065 (9)   | 0.032 (7)   | 0.046 (6)   | −0.005 (5)   | 0.031 (4)   | 0.003 (4)   |
| C24b | 0.053 (8)   | 0.046 (8)   | 0.047 (7)   | 0.003 (5)    | 0.024 (4)   | 0.015 (4)   |
| C23b | 0.029 (7)   | 0.040 (8)   | 0.044 (8)   | 0.013 (4)    | 0.018 (4)   | 0.019 (4)   |
| C22b | 0.040 (7)   | 0.047 (8)   | 0.041 (5)   | 0.005 (4)    | 0.019 (4)   | 0.014 (4)   |

*Geometric parameters (Å, °)*

|           |             |            |             |
|-----------|-------------|------------|-------------|
| Fe1—N4    | 2.2420 (10) | C16—H8     | 1.1030      |
| Fe1—N5    | 2.2028 (11) | C3—H1      | 1.1030      |
| Fe1—N6    | 2.1978 (11) | C3—C4      | 1.385 (2)   |
| Fe1—N3    | 2.1968 (12) | C6—H4      | 1.1030      |
| Fe1—N1    | 2.1109 (13) | C6—C5      | 1.382 (3)   |
| Fe1—N2    | 2.0536 (13) | C25—H22    | 1.1030      |
| S2—C2     | 1.6213 (14) | C25—C26    | 1.366 (5)   |
| S1—C1     | 1.6138 (18) | C25—C24    | 1.365 (6)   |
| N4—C8     | 1.4782 (17) | C26—H23    | 1.1030      |
| N4—C14    | 1.4722 (16) | C26—C21    | 1.352 (4)   |
| N4—C20    | 1.4760 (16) | C4—H2      | 1.1030      |
| N5—C13    | 1.3407 (16) | C4—C5      | 1.385 (3)   |
| N5—C9     | 1.3406 (17) | C5—H3      | 1.1030      |
| N6—C19    | 1.3390 (16) | C21—C22    | 1.366 (7)   |
| N6—C15    | 1.3391 (17) | C21—F1     | 1.247 (5)   |
| N3—C7     | 1.3349 (18) | C24—H21    | 1.1030      |
| N3—C3     | 1.3416 (18) | C24—C23    | 1.393 (8)   |
| N1—C1     | 1.1581 (19) | C22—H19    | 1.1030      |
| N2—C2     | 1.1640 (19) | C22—C23    | 1.371 (8)   |
| C19—C18   | 1.3941 (18) | C23—H20    | 1.1030      |
| C19—C20   | 1.5028 (19) | C21a—C22a  | 1.371 (8)   |
| C18—H10   | 1.1030      | C21a—C26a  | 1.366 (7)   |
| C18—C17   | 1.386 (2)   | C21a—F1a   | 1.341 (9)   |
| C13—C12   | 1.3929 (18) | C22a—H19a  | 1.1030      |
| C13—C14   | 1.5063 (18) | C22a—C23a  | 1.393 (8)   |
| C12—H16   | 1.1030      | C23a—H20a  | 1.1030      |
| C12—C11   | 1.390 (2)   | C23a—C24a  | 1.365 (6)   |
| C9—H13    | 1.1030      | C24a—H21a  | 1.1030      |
| C9—C10    | 1.386 (2)   | C24a—C25a  | 1.366 (5)   |
| C8—H5     | 1.1110      | C25a—H22a  | 1.1030      |
| C8—H6     | 1.1110      | C25a—C26a  | 1.352 (4)   |
| C8—C7     | 1.507 (2)   | C26a—H23a  | 1.1030      |
| C7—C6     | 1.3910 (19) | F2—C21b    | 1.340 (14)  |
| C14—H18   | 1.1110      | C21b—C26b  | 1.368 (13)  |
| C14—H17   | 1.1110      | C21b—C22b  | 1.355 (13)  |
| C10—H14   | 1.1030      | C26b—H23b  | 1.1030      |
| C10—C11   | 1.388 (2)   | C26b—C25b  | 1.369 (13)  |
| C20—H11   | 1.1110      | C25b—H22b  | 1.1030      |
| C20—H12   | 1.1110      | C25b—C24b  | 1.367 (12)  |
| C11—H15   | 1.1030      | C24b—H21b  | 1.1030      |
| C15—H7    | 1.1030      | C24b—C23b  | 1.385 (14)  |
| C15—C16   | 1.384 (2)   | C23b—H20b  | 1.1030      |
| C17—H9    | 1.1030      | C23b—C22b  | 1.383 (14)  |
| C17—C16   | 1.389 (2)   | C22b—H19b  | 1.1030      |
| N5—Fe1—N4 | 75.87 (4)   | C16—C15—N6 | 122.77 (14) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N6—Fe1—N4   | 75.65 (4)   | C16—C15—H7     | 118.62 (9)  |
| N6—Fe1—N5   | 151.52 (4)  | H9—C17—C18     | 120.47 (9)  |
| N3—Fe1—N4   | 78.03 (4)   | C16—C17—C18    | 119.06 (13) |
| N3—Fe1—N5   | 89.57 (4)   | C16—C17—H9     | 120.47 (9)  |
| N3—Fe1—N6   | 83.88 (4)   | N1—C1—S1       | 178.57 (15) |
| N1—Fe1—N4   | 91.69 (5)   | C17—C16—C15    | 118.54 (13) |
| N1—Fe1—N5   | 91.91 (5)   | H8—C16—C15     | 120.73 (9)  |
| N1—Fe1—N6   | 89.63 (4)   | H8—C16—C17     | 120.73 (9)  |
| N1—Fe1—N3   | 168.93 (5)  | H1—C3—N3       | 118.63 (9)  |
| N2—Fe1—N4   | 171.72 (5)  | C4—C3—N3       | 122.73 (18) |
| N2—Fe1—N5   | 100.87 (5)  | C4—C3—H1       | 118.63 (12) |
| N2—Fe1—N6   | 107.25 (5)  | H4—C6—C7       | 120.39 (10) |
| N2—Fe1—N3   | 94.44 (5)   | C5—C6—C7       | 119.22 (17) |
| N2—Fe1—N1   | 96.05 (6)   | C5—C6—H4       | 120.39 (11) |
| C8—N4—Fe1   | 111.14 (8)  | C26—C25—H22    | 119.4 (3)   |
| C14—N4—Fe1  | 105.80 (8)  | C24—C25—H22    | 119.4 (3)   |
| C14—N4—C8   | 111.15 (10) | C24—C25—C26    | 121.3 (4)   |
| C20—N4—Fe1  | 105.89 (8)  | H23—C26—C25    | 120.2 (3)   |
| C20—N4—C8   | 112.05 (11) | C21—C26—C25    | 119.5 (4)   |
| C20—N4—C14  | 110.52 (10) | C21—C26—H23    | 120.2 (3)   |
| C13—N5—Fe1  | 114.68 (8)  | H2—C4—C3       | 120.80 (12) |
| C9—N5—Fe1   | 126.77 (9)  | C5—C4—C3       | 118.40 (17) |
| C9—N5—C13   | 118.54 (12) | C5—C4—H2       | 120.80 (10) |
| C19—N6—Fe1  | 115.12 (8)  | C4—C5—C6       | 119.04 (16) |
| C15—N6—Fe1  | 126.19 (9)  | H3—C5—C6       | 120.48 (11) |
| C15—N6—C19  | 118.68 (12) | H3—C5—C4       | 120.48 (10) |
| C7—N3—Fe1   | 116.04 (9)  | C22—C21—C26    | 121.5 (4)   |
| C3—N3—Fe1   | 125.00 (11) | F1—C21—C26     | 123.1 (6)   |
| C3—N3—C7    | 118.78 (13) | F1—C21—C22     | 115.4 (5)   |
| C1—N1—Fe1   | 169.59 (13) | H21—C24—C25    | 121.0 (3)   |
| C2—N2—Fe1   | 178.16 (13) | C23—C24—C25    | 118.1 (5)   |
| C18—C19—N6  | 122.16 (12) | C23—C24—H21    | 121.0 (3)   |
| C20—C19—N6  | 116.11 (11) | H19—C22—C21    | 120.6 (3)   |
| C20—C19—C18 | 121.65 (12) | C23—C22—C21    | 118.7 (4)   |
| H10—C18—C19 | 120.63 (8)  | C23—C22—H19    | 120.6 (3)   |
| C17—C18—C19 | 118.75 (13) | C22—C23—C24    | 120.8 (4)   |
| C17—C18—H10 | 120.63 (9)  | H20—C23—C24    | 119.6 (3)   |
| C12—C13—N5  | 122.13 (12) | H20—C23—C22    | 119.6 (3)   |
| C14—C13—N5  | 116.22 (11) | C26a—C21a—C22a | 118.7 (4)   |
| C14—C13—C12 | 121.61 (12) | F1a—C21a—C22a  | 133.9 (8)   |
| H16—C12—C13 | 120.47 (8)  | F1a—C21a—C26a  | 107.2 (8)   |
| C11—C12—C13 | 119.07 (13) | H19a—C22a—C21a | 119.6 (3)   |
| C11—C12—H16 | 120.47 (9)  | C23a—C22a—C21a | 120.8 (4)   |
| H13—C9—N5   | 118.61 (8)  | C23a—C22a—H19a | 119.6 (3)   |
| C10—C9—N5   | 122.79 (13) | H20a—C23a—C22a | 121.0 (3)   |
| C10—C9—H13  | 118.61 (8)  | C24a—C23a—C22a | 118.1 (5)   |
| H5—C8—N4    | 108.44 (7)  | C24a—C23a—H20a | 121.0 (3)   |
| H6—C8—N4    | 108.44 (7)  | H21a—C24a—C23a | 119.4 (3)   |



|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| H6—C8—H5       | 107.5        | C25a—C24a—C23a  | 121.3 (4)    |
| C7—C8—N4       | 115.36 (11)  | C25a—C24a—H21a  | 119.4 (3)    |
| C7—C8—H5       | 108.44 (7)   | H22a—C25a—C24a  | 120.2 (3)    |
| C7—C8—H6       | 108.44 (8)   | C26a—C25a—C24a  | 119.5 (4)    |
| N2—C2—S2       | 179.40 (14)  | C26a—C25a—H22a  | 120.2 (3)    |
| C8—C7—N3       | 118.70 (12)  | C25a—C26a—C21a  | 121.5 (4)    |
| C6—C7—N3       | 121.83 (14)  | H23a—C26a—C21a  | 119.3 (3)    |
| C6—C7—C8       | 119.41 (14)  | H23a—C26a—C25a  | 119.3 (3)    |
| C13—C14—N4     | 111.23 (10)  | C26b—C21b—F2    | 111.9 (14)   |
| H18—C14—N4     | 109.39 (7)   | C22b—C21b—F2    | 124.8 (14)   |
| H18—C14—C13    | 109.39 (7)   | C22b—C21b—C26b  | 122.9 (15)   |
| H17—C14—N4     | 109.39 (7)   | H23b—C26b—C21b  | 121.0 (10)   |
| H17—C14—C13    | 109.39 (7)   | C25b—C26b—C21b  | 118.0 (15)   |
| H17—C14—H18    | 108.0        | C25b—C26b—H23b  | 121.0 (10)   |
| H14—C10—C9     | 120.59 (8)   | H22b—C25b—C26b  | 119.8 (10)   |
| C11—C10—C9     | 118.81 (13)  | C24b—C25b—C26b  | 120.3 (14)   |
| C11—C10—H14    | 120.59 (8)   | C24b—C25b—H22b  | 119.8 (10)   |
| C19—C20—N4     | 111.39 (10)  | H21b—C24b—C25b  | 119.4 (10)   |
| H11—C20—N4     | 109.35 (7)   | C23b—C24b—C25b  | 121.1 (15)   |
| H11—C20—C19    | 109.35 (7)   | C23b—C24b—H21b  | 119.4 (10)   |
| H12—C20—N4     | 109.35 (7)   | H20b—C23b—C24b  | 120.9 (10)   |
| H12—C20—C19    | 109.35 (7)   | C22b—C23b—C24b  | 118.3 (16)   |
| H12—C20—H11    | 108.0        | C22b—C23b—H20b  | 120.9 (10)   |
| C10—C11—C12    | 118.65 (13)  | C23b—C22b—C21b  | 119.2 (15)   |
| H15—C11—C12    | 120.67 (9)   | H19b—C22b—C21b  | 120.4 (10)   |
| H15—C11—C10    | 120.67 (8)   | H19b—C22b—C23b  | 120.4 (10)   |
| H7—C15—N6      | 118.62 (8)   |                 |              |
| Fe1—N4—C8—C7   | −7.98 (9)    | C9—N5—C13—C14   | 177.77 (12)  |
| Fe1—N4—C14—C13 | −43.21 (8)   | C8—C7—N3—C3     | 177.72 (14)  |
| Fe1—N4—C20—C19 | 42.58 (8)    | C8—C7—C6—C5     | −177.59 (17) |
| Fe1—N5—C13—C12 | 179.51 (9)   | C7—N3—C3—C4     | −0.60 (17)   |
| Fe1—N5—C13—C14 | −2.82 (10)   | C7—C8—N4—C14    | −125.53 (12) |
| Fe1—N5—C9—C10  | 179.91 (11)  | C7—C8—N4—C20    | 110.26 (12)  |
| Fe1—N6—C19—C18 | 178.06 (9)   | C7—C6—C5—C4     | 0.4 (2)      |
| Fe1—N6—C19—C20 | 1.12 (10)    | C14—C13—C12—C11 | −177.11 (14) |
| Fe1—N6—C15—C16 | −177.05 (12) | C20—C19—N6—C15  | −178.03 (12) |
| Fe1—N3—C7—C8   | −6.98 (11)   | C20—C19—C18—C17 | 175.95 (14)  |
| Fe1—N3—C7—C6   | 175.76 (11)  | C3—N3—C7—C6     | 0.46 (17)    |
| Fe1—N3—C3—C4   | −175.45 (15) | C3—C4—C5—C6     | −0.5 (2)     |
| N4—C8—C7—N3    | 10.25 (15)   | C25—C26—C21—C22 | 0.8 (4)      |
| N4—C8—C7—C6    | −172.42 (13) | C25—C26—C21—F1  | 179.0 (4)    |
| N4—C14—C13—N5  | 32.41 (13)   | C25—C24—C23—C22 | −0.5 (4)     |
| N4—C14—C13—C12 | −149.91 (11) | C26—C25—C24—C23 | 1.4 (6)      |
| N4—C20—C19—N6  | −30.79 (13)  | C26—C21—C22—C23 | 0.1 (3)      |
| N4—C20—C19—C18 | 152.26 (11)  | C21—C26—C25—C24 | −1.6 (4)     |
| N5—C13—C12—C11 | 0.43 (16)    | C21—C22—C23—C24 | −0.3 (4)     |
| N5—C9—C10—C11  | 0.87 (17)    | C23—C22—C21—F1  | −178.2 (4)   |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N6—C19—C18—C17  | −0.82 (15)   | C21a—C22a—C23a—C24a | −0.5 (5)    |
| N6—C15—C16—C17  | −0.95 (18)   | C21a—C26a—C25a—C24a | 0.8 (4)     |
| N3—C7—C6—C5     | −0.34 (18)   | C22a—C21a—C26a—C25a | 0.1 (4)     |
| N3—C3—C4—C5     | 0.6 (2)      | C22a—C23a—C24a—C25a | 1.4 (5)     |
| C19—N6—C15—C16  | 2.00 (14)    | C23a—C22a—C21a—C26a | −0.3 (5)    |
| C19—C18—C17—C16 | 1.85 (17)    | C23a—C22a—C21a—F1a  | −174.8 (7)  |
| C19—C20—N4—C8   | −78.74 (12)  | C23a—C24a—C25a—C26a | −1.6 (5)    |
| C19—C20—N4—C14  | 156.71 (11)  | C25a—C26a—C21a—F1a  | 176.0 (5)   |
| C18—C19—N6—C15  | −1.09 (16)   | F2—C21b—C26b—C25b   | −175.1 (12) |
| C18—C17—C16—C15 | −1.02 (19)   | F2—C21b—C22b—C23b   | 176.5 (17)  |
| C13—N5—C9—C10   | −0.76 (14)   | C21b—C26b—C25b—C24b | −1 (2)      |
| C13—C12—C11—C10 | −0.30 (16)   | C21b—C22b—C23b—C24b | −3 (3)      |
| C13—C14—N4—C8   | 77.54 (12)   | C26b—C21b—C22b—C23b | 4 (2)       |
| C13—C14—N4—C20  | −157.39 (11) | C26b—C25b—C24b—C23b | 2 (2)       |
| C12—C13—N5—C9   | 0.10 (16)    | C25b—C26b—C21b—C22b | −1 (3)      |
| C12—C11—C10—C9  | −0.31 (18)   | C25b—C24b—C23b—C22b | 0 (3)       |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_230K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.3904$  (3) Å

$b = 12.0283$  (2) Å

$c = 15.4700$  (3) Å

$\beta = 110.394$  (2)°

$V = 2684.30$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.382$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 52407 reflections

$\theta = 2.1$ – $32.5^\circ$

$\mu = 0.75$  mm<sup>−1</sup>

$T = 230$  K

Block, yellow

$0.31 \times 0.2 \times 0.12$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.650$ ,  $T_{\max} = 1.000$

77392 measured reflections

6649 independent reflections

6045 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -16 \rightarrow 16$

$l = -17 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.099$

$S = 1.05$

6649 reflections

408 parameters

149 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.8065P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.90$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.70$  e Å<sup>−3</sup>

*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  | 0.656677 (14) | 0.328726 (16) | 0.302373 (13) | 0.03307 (7)                      |           |
| S2   | 0.47213 (3)   | 0.00363 (4)   | 0.17412 (4)   | 0.06003 (13)                     |           |
| S1   | 0.76275 (12)  | 0.34326 (12)  | 0.04157 (9)   | 0.0880 (2)                       |           |
| N4   | 0.74134 (8)   | 0.47053 (9)   | 0.38366 (8)   | 0.0340 (2)                       |           |
| N5   | 0.78720 (8)   | 0.25047 (9)   | 0.39081 (8)   | 0.0349 (2)                       |           |
| N6   | 0.56704 (8)   | 0.47568 (10)  | 0.25510 (8)   | 0.0364 (2)                       |           |
| N3   | 0.61008 (9)   | 0.33734 (11)  | 0.42160 (9)   | 0.0413 (3)                       |           |
| N1   | 0.70903 (11)  | 0.35263 (12)  | 0.19415 (10)  | 0.0482 (3)                       |           |
| N2   | 0.57778 (11)  | 0.19161 (13)  | 0.24624 (11)  | 0.0543 (4)                       |           |
| C19  | 0.61064 (10)  | 0.57377 (11)  | 0.27489 (9)   | 0.0357 (3)                       |           |
| C18  | 0.56407 (12)  | 0.67415 (13)  | 0.24785 (11)  | 0.0451 (3)                       |           |
| C13  | 0.85778 (10)  | 0.32132 (11)  | 0.42744 (10)  | 0.0360 (3)                       |           |
| C12  | 0.94459 (10)  | 0.28597 (14)  | 0.48507 (11)  | 0.0442 (3)                       |           |
| C7   | 0.64717 (10)  | 0.41821 (14)  | 0.48225 (9)   | 0.0409 (3)                       |           |
| C9   | 0.80142 (11)  | 0.14229 (12)  | 0.41068 (10)  | 0.0411 (3)                       |           |
| C8   | 0.72517 (11)  | 0.48571 (14)  | 0.47149 (10)  | 0.0454 (3)                       |           |
| C2   | 0.53379 (11)  | 0.11268 (13)  | 0.21663 (11)  | 0.0427 (3)                       |           |
| C11  | 0.95915 (12)  | 0.17353 (14)  | 0.50492 (12)  | 0.0493 (4)                       |           |
| C14  | 0.83867 (10)  | 0.44157 (12)  | 0.39989 (11)  | 0.0413 (3)                       |           |
| C10  | 0.88629 (12)  | 0.10021 (14)  | 0.46672 (11)  | 0.0489 (4)                       |           |
| C20  | 0.71367 (11)  | 0.56916 (12)  | 0.32363 (11)  | 0.0426 (3)                       |           |
| C15  | 0.47557 (11)  | 0.47471 (14)  | 0.20909 (11)  | 0.0460 (3)                       |           |
| C1   | 0.73144 (12)  | 0.35002 (14)  | 0.13030 (11)  | 0.0459 (4)                       |           |
| C17  | 0.46995 (13)  | 0.67229 (15)  | 0.19786 (13)  | 0.0525 (4)                       |           |
| C16  | 0.42450 (12)  | 0.57097 (16)  | 0.17859 (12)  | 0.0534 (4)                       |           |
| C6   | 0.61871 (14)  | 0.4380 (2)    | 0.55676 (12)  | 0.0629 (5)                       |           |
| C3   | 0.54300 (13)  | 0.27434 (19)  | 0.43311 (13)  | 0.0595 (5)                       |           |
| C24a | 0.7806 (4)    | 0.2194 (5)    | 0.6340 (3)    | 0.0849 (17)                      | 0.195 (6) |
| C4   | 0.51121 (15)  | 0.2892 (3)    | 0.50561 (16)  | 0.0795 (7)                       |           |
| C25a | 0.8094 (2)    | 0.2794 (4)    | 0.7137 (3)    | 0.0851 (11)                      | 0.195 (6) |
| C5   | 0.55004 (16)  | 0.3729 (3)    | 0.56859 (15)  | 0.0815 (7)                       |           |
| C21a | 0.7212 (5)    | 0.1615 (7)    | 0.7750 (6)    | 0.139 (3)                        | 0.195 (6) |
| C23a | 0.7246 (4)    | 0.1292 (5)    | 0.6231 (5)    | 0.130 (2)                        | 0.195 (6) |
| C26a | 0.7792 (3)    | 0.2504 (5)    | 0.7827 (3)    | 0.1067 (15)                      | 0.195 (6) |
| C22a | 0.6950 (4)    | 0.1011 (5)    | 0.6944 (8)    | 0.162 (3)                        | 0.195 (6) |
| F1   | 0.8020 (5)    | 0.2978 (7)    | 0.8556 (3)    | 0.278 (5)                        | 0.685 (6) |
| H13  | 0.74372 (11)  | 0.08458 (12)  | 0.38154 (10)  | 0.0494 (4)*                      |           |
| H14  | 0.89554 (12)  | 0.01104 (14)  | 0.48048 (11)  | 0.0586 (4)*                      |           |
| H15  | 1.02681 (12)  | 0.14332 (14)  | 0.54984 (12)  | 0.0591 (5)*                      |           |
| H16  | 1.00059 (10)  | 0.34585 (14)  | 0.51435 (11)  | 0.0530 (4)*                      |           |
| H7   | 0.43962 (11)  | 0.39475 (14)  | 0.19496 (11)  | 0.0552 (4)*                      |           |
| H8   | 0.35002 (12)  | 0.56699 (16)  | 0.14020 (12)  | 0.0640 (5)*                      |           |
| H9   | 0.43203 (13)  | 0.74971 (15)  | 0.17391 (13)  | 0.0630 (5)*                      |           |
| H10  | 0.60119 (12)  | 0.75310 (13)  | 0.26580 (11)  | 0.0541 (4)*                      |           |
| H1   | 0.51220 (13)  | 0.20851 (19)  | 0.38310 (13)  | 0.0713 (6)*                      |           |

|      |              |              |              |              |           |
|------|--------------|--------------|--------------|--------------|-----------|
| H2   | 0.45663 (15) | 0.2361 (3)   | 0.51300 (16) | 0.0954 (8)*  |           |
| H3   | 0.52661 (16) | 0.3872 (3)   | 0.62674 (15) | 0.0978 (9)*  |           |
| H4   | 0.65054 (14) | 0.5045 (2)   | 0.60561 (12) | 0.0755 (6)*  |           |
| H11  | 0.73654 (11) | 0.64475 (12) | 0.36556 (11) | 0.0511 (4)*  |           |
| H12  | 0.74829 (11) | 0.56710 (12) | 0.27223 (11) | 0.0511 (4)*  |           |
| H5   | 0.78944 (11) | 0.46454 (14) | 0.52844 (10) | 0.0545 (4)*  |           |
| H6   | 0.71080 (11) | 0.57427 (14) | 0.47856 (10) | 0.0545 (4)*  |           |
| H18  | 0.85423 (10) | 0.45705 (12) | 0.33666 (11) | 0.0495 (4)*  |           |
| H17  | 0.88435 (10) | 0.49494 (12) | 0.45483 (11) | 0.0495 (4)*  |           |
| F1a  | 0.7106 (8)   | 0.1506 (10)  | 0.8564 (8)   | 0.131 (6)    | 0.195 (6) |
| C22  | 0.7212 (5)   | 0.1615 (7)   | 0.7750 (6)   | 0.139 (3)    | 0.685 (6) |
| C23  | 0.6950 (4)   | 0.1011 (5)   | 0.6944 (8)   | 0.162 (3)    | 0.685 (6) |
| C24  | 0.7246 (4)   | 0.1292 (5)   | 0.6231 (5)   | 0.130 (2)    | 0.685 (6) |
| C25  | 0.7806 (4)   | 0.2194 (5)   | 0.6340 (3)   | 0.0849 (17)  | 0.685 (6) |
| C26  | 0.8094 (2)   | 0.2794 (4)   | 0.7137 (3)   | 0.0851 (11)  | 0.685 (6) |
| C21  | 0.7792 (3)   | 0.2504 (5)   | 0.7827 (3)   | 0.1067 (15)  | 0.685 (6) |
| F1b  | 0.6256 (9)   | 0.0089 (10)  | 0.6164 (9)   | 0.088 (3)    | 0.12      |
| C21b | 0.6903 (16)  | 0.0866 (18)  | 0.6501 (12)  | 0.083 (7)    | 0.12      |
| C26b | 0.6998 (16)  | 0.1110 (16)  | 0.7387 (12)  | 0.072 (6)    | 0.12      |
| C25b | 0.7569 (17)  | 0.1957 (15)  | 0.7830 (14)  | 0.059 (5)    | 0.12      |
| C24b | 0.8046 (17)  | 0.2546 (18)  | 0.7377 (15)  | 0.061 (6)    | 0.12      |
| C23b | 0.7953 (19)  | 0.229 (2)    | 0.6479 (17)  | 0.048 (5)    | 0.12      |
| C22b | 0.7347 (17)  | 0.1462 (19)  | 0.6036 (14)  | 0.055 (5)    | 0.12      |
| H21a | 0.8032 (4)   | 0.2444 (5)   | 0.5773 (3)   | 0.102 (2)*   | 0.195 (6) |
| H20a | 0.7037 (4)   | 0.0805 (5)   | 0.5594 (5)   | 0.156 (3)*   | 0.195 (6) |
| H19a | 0.6497 (4)   | 0.0291 (5)   | 0.6869 (8)   | 0.194 (4)*   | 0.195 (6) |
| H23a | 0.8017 (3)   | 0.2991 (5)   | 0.8464 (3)   | 0.1280 (18)* | 0.195 (6) |
| H22a | 0.8562 (2)   | 0.3501 (4)   | 0.7216 (3)   | 0.1021 (14)* | 0.195 (6) |
| H22  | 0.8032 (4)   | 0.2444 (5)   | 0.5773 (3)   | 0.102 (2)*   | 0.685 (6) |
| H23  | 0.8562 (2)   | 0.3501 (4)   | 0.7216 (3)   | 0.1021 (14)* | 0.685 (6) |
| H19  | 0.6966 (5)   | 0.1394 (7)   | 0.8310 (6)   | 0.167 (4)*   | 0.685 (6) |
| H20  | 0.6497 (4)   | 0.0291 (5)   | 0.6869 (8)   | 0.194 (4)*   | 0.685 (6) |
| H21  | 0.7037 (4)   | 0.0805 (5)   | 0.5594 (5)   | 0.156 (3)*   | 0.685 (6) |
| H20b | 0.8354 (19)  | 0.274 (2)    | 0.6128 (17)  | 0.057 (6)*   | 0.12      |
| H19b | 0.7223 (17)  | 0.1283 (19)  | 0.5310 (14)  | 0.066 (5)*   | 0.12      |
| H23b | 0.6618 (16)  | 0.0632 (16)  | 0.7742 (12)  | 0.086 (8)*   | 0.12      |
| H22b | 0.7648 (17)  | 0.2166 (15)  | 0.8541 (14)  | 0.071 (6)*   | 0.12      |
| H21b | 0.8503 (17)  | 0.3225 (18)  | 0.7732 (15)  | 0.073 (7)*   | 0.12      |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|---------------|-------------|--------------|
| Fe1 | 0.03809 (12) | 0.02910 (11) | 0.03000 (11) | −0.00355 (7)  | 0.00930 (8) | −0.00083 (7) |
| S2  | 0.0529 (2)   | 0.0464 (2)   | 0.0845 (3)   | −0.01824 (19) | 0.0285 (2)  | −0.0171 (2)  |
| S1  | 0.1355 (6)   | 0.0914 (4)   | 0.0576 (3)   | 0.0615 (4)    | 0.0595 (4)  | 0.0330 (3)   |
| N4  | 0.0360 (6)   | 0.0280 (5)   | 0.0358 (6)   | −0.0045 (4)   | 0.0098 (4)  | 0.0003 (4)   |
| N5  | 0.0396 (6)   | 0.0292 (5)   | 0.0330 (5)   | −0.0018 (5)   | 0.0092 (5)  | 0.0009 (4)   |
| N6  | 0.0392 (6)   | 0.0323 (6)   | 0.0336 (5)   | −0.0007 (5)   | 0.0076 (5)  | −0.0009 (4)  |

|      |             |             |             |              |             |             |
|------|-------------|-------------|-------------|--------------|-------------|-------------|
| N3   | 0.0411 (6)  | 0.0492 (7)  | 0.0346 (6)  | −0.0060 (5)  | 0.0145 (5)  | 0.0023 (5)  |
| N1   | 0.0640 (9)  | 0.0453 (7)  | 0.0412 (7)  | 0.0075 (6)   | 0.0257 (6)  | 0.0049 (6)  |
| N2   | 0.0580 (9)  | 0.0433 (7)  | 0.0546 (8)  | −0.0162 (6)  | 0.0106 (7)  | −0.0123 (6) |
| C19  | 0.0419 (7)  | 0.0293 (6)  | 0.0341 (6)  | 0.0001 (5)   | 0.0109 (5)  | 0.0013 (5)  |
| C18  | 0.0503 (9)  | 0.0357 (8)  | 0.0462 (8)  | 0.0057 (6)   | 0.0129 (7)  | 0.0048 (6)  |
| C13  | 0.0358 (7)  | 0.0321 (7)  | 0.0380 (7)  | −0.0005 (5)  | 0.0100 (5)  | 0.0009 (5)  |
| C12  | 0.0369 (7)  | 0.0408 (8)  | 0.0493 (8)  | 0.0021 (6)   | 0.0081 (6)  | −0.0017 (6) |
| C7   | 0.0403 (7)  | 0.0512 (8)  | 0.0301 (6)  | 0.0049 (6)   | 0.0110 (5)  | 0.0004 (6)  |
| C9   | 0.0519 (8)  | 0.0297 (6)  | 0.0377 (7)  | −0.0007 (6)  | 0.0105 (6)  | −0.0005 (5) |
| C8   | 0.0509 (9)  | 0.0451 (8)  | 0.0375 (7)  | −0.0060 (7)  | 0.0119 (6)  | −0.0084 (6) |
| C2   | 0.0427 (7)  | 0.0375 (8)  | 0.0474 (8)  | −0.0080 (6)  | 0.0152 (6)  | −0.0080 (6) |
| C11  | 0.0466 (9)  | 0.0439 (9)  | 0.0501 (9)  | 0.0114 (7)   | 0.0077 (7)  | 0.0027 (7)  |
| C14  | 0.0350 (7)  | 0.0330 (7)  | 0.0528 (8)  | −0.0047 (5)  | 0.0114 (6)  | 0.0037 (6)  |
| C10  | 0.0596 (10) | 0.0353 (8)  | 0.0458 (8)  | 0.0088 (7)   | 0.0110 (7)  | 0.0019 (6)  |
| C20  | 0.0422 (7)  | 0.0297 (7)  | 0.0513 (8)  | −0.0043 (6)  | 0.0107 (6)  | 0.0062 (6)  |
| C15  | 0.0413 (8)  | 0.0450 (8)  | 0.0436 (8)  | −0.0023 (6)  | 0.0046 (6)  | −0.0032 (6) |
| C1   | 0.0613 (9)  | 0.0430 (8)  | 0.0362 (7)  | 0.0177 (7)   | 0.0205 (7)  | 0.0089 (6)  |
| C17  | 0.0507 (9)  | 0.0480 (9)  | 0.0539 (9)  | 0.0132 (7)   | 0.0121 (8)  | 0.0085 (7)  |
| C16  | 0.0422 (8)  | 0.0575 (10) | 0.0504 (9)  | 0.0074 (7)   | 0.0035 (7)  | 0.0019 (8)  |
| C6   | 0.0596 (10) | 0.0947 (15) | 0.0385 (8)  | 0.0057 (10)  | 0.0224 (8)  | −0.0073 (9) |
| C3   | 0.0512 (9)  | 0.0783 (13) | 0.0516 (9)  | −0.0182 (9)  | 0.0214 (8)  | 0.0075 (9)  |
| C24a | 0.084 (3)   | 0.102 (4)   | 0.080 (2)   | 0.036 (2)    | 0.042 (2)   | 0.031 (2)   |
| C4   | 0.0579 (11) | 0.126 (2)   | 0.0644 (12) | −0.0155 (13) | 0.0339 (10) | 0.0179 (14) |
| C25a | 0.0557 (15) | 0.094 (3)   | 0.113 (3)   | 0.0154 (15)  | 0.0382 (18) | 0.037 (3)   |
| C5   | 0.0646 (12) | 0.141 (2)   | 0.0513 (11) | −0.0008 (14) | 0.0355 (10) | 0.0035 (13) |
| C21a | 0.120 (4)   | 0.148 (6)   | 0.198 (7)   | 0.062 (4)    | 0.116 (5)   | 0.117 (5)   |
| C23a | 0.088 (3)   | 0.095 (3)   | 0.201 (6)   | 0.026 (2)    | 0.044 (3)   | 0.045 (4)   |
| C26a | 0.099 (3)   | 0.154 (5)   | 0.072 (2)   | 0.061 (3)    | 0.036 (2)   | 0.034 (3)   |
| C22a | 0.098 (4)   | 0.084 (4)   | 0.315 (11)  | 0.017 (3)    | 0.088 (6)   | 0.067 (5)   |
| F1   | 0.261 (7)   | 0.449 (12)  | 0.117 (3)   | 0.181 (8)    | 0.057 (4)   | −0.013 (5)  |
| F1a  | 0.137 (9)   | 0.156 (11)  | 0.123 (9)   | 0.024 (7)    | 0.075 (7)   | 0.067 (7)   |
| C22  | 0.120 (4)   | 0.148 (6)   | 0.198 (7)   | 0.062 (4)    | 0.116 (5)   | 0.117 (5)   |
| C23  | 0.098 (4)   | 0.084 (4)   | 0.315 (11)  | 0.017 (3)    | 0.088 (6)   | 0.067 (5)   |
| C24  | 0.088 (3)   | 0.095 (3)   | 0.201 (6)   | 0.026 (2)    | 0.044 (3)   | 0.045 (4)   |
| C25  | 0.084 (3)   | 0.102 (4)   | 0.080 (2)   | 0.036 (2)    | 0.042 (2)   | 0.031 (2)   |
| C26  | 0.0557 (15) | 0.094 (3)   | 0.113 (3)   | 0.0154 (15)  | 0.0382 (18) | 0.037 (3)   |
| C21  | 0.099 (3)   | 0.154 (5)   | 0.072 (2)   | 0.061 (3)    | 0.036 (2)   | 0.034 (3)   |
| F1b  | 0.104 (7)   | 0.068 (6)   | 0.103 (7)   | −0.021 (5)   | 0.050 (5)   | −0.009 (5)  |
| C21b | 0.113 (13)  | 0.086 (11)  | 0.070 (7)   | −0.034 (6)   | 0.056 (5)   | −0.009 (5)  |
| C26b | 0.106 (13)  | 0.053 (10)  | 0.076 (7)   | −0.035 (7)   | 0.055 (5)   | −0.006 (4)  |
| C25b | 0.092 (11)  | 0.037 (7)   | 0.064 (7)   | −0.019 (6)   | 0.046 (5)   | 0.002 (4)   |
| C24b | 0.074 (10)  | 0.060 (9)   | 0.058 (8)   | −0.009 (5)   | 0.034 (5)   | 0.018 (5)   |
| C23b | 0.047 (8)   | 0.048 (9)   | 0.053 (8)   | 0.011 (5)    | 0.024 (5)   | 0.024 (4)   |
| C22b | 0.059 (8)   | 0.052 (8)   | 0.061 (7)   | 0.003 (5)    | 0.030 (4)   | 0.013 (4)   |

*Geometric parameters (Å, °)*

|           |             |           |            |
|-----------|-------------|-----------|------------|
| Fe1—N4    | 2.2464 (11) | C16—H8    | 1.0930     |
| Fe1—N5    | 2.2085 (12) | C6—C5     | 1.377 (3)  |
| Fe1—N6    | 2.2044 (12) | C6—H4     | 1.0930     |
| Fe1—N3    | 2.2020 (13) | C3—C4     | 1.383 (3)  |
| Fe1—N1    | 2.1154 (14) | C3—H1     | 1.0930     |
| Fe1—N2    | 2.0527 (14) | C24a—C25a | 1.363 (6)  |
| S2—C2     | 1.6181 (16) | C24a—C23a | 1.359 (7)  |
| S1—C1     | 1.607 (2)   | C24a—H21a | 1.0930     |
| N4—C8     | 1.4756 (19) | C4—C5     | 1.384 (4)  |
| N4—C14    | 1.4716 (18) | C4—H2     | 1.0930     |
| N4—C20    | 1.4750 (18) | C25a—C26a | 1.350 (5)  |
| N5—C13    | 1.3409 (18) | C25a—H22a | 1.0930     |
| N5—C9     | 1.3374 (18) | C5—H3     | 1.0930     |
| N6—C19    | 1.3389 (18) | C21a—C26a | 1.371 (8)  |
| N6—C15    | 1.3380 (19) | C21a—C22a | 1.377 (9)  |
| N3—C7     | 1.334 (2)   | C21a—F1a  | 1.332 (11) |
| N3—C3     | 1.342 (2)   | C23a—C22a | 1.374 (9)  |
| N1—C1     | 1.155 (2)   | C23a—H20a | 1.0930     |
| N2—C2     | 1.163 (2)   | C26a—H23a | 1.0930     |
| C19—C18   | 1.392 (2)   | C22a—H19a | 1.0930     |
| C19—C20   | 1.501 (2)   | F1—C21    | 1.201 (7)  |
| C18—C17   | 1.384 (2)   | C22—C23   | 1.377 (9)  |
| C18—H10   | 1.0930      | C22—C21   | 1.371 (8)  |
| C13—C12   | 1.389 (2)   | C22—H19   | 1.0930     |
| C13—C14   | 1.5076 (19) | C23—C24   | 1.374 (9)  |
| C12—C11   | 1.388 (2)   | C23—H20   | 1.0930     |
| C12—H16   | 1.0930      | C24—C25   | 1.359 (7)  |
| C7—C8     | 1.506 (2)   | C24—H21   | 1.0930     |
| C7—C6     | 1.389 (2)   | C25—C26   | 1.363 (6)  |
| C9—C10    | 1.387 (2)   | C25—H22   | 1.0930     |
| C9—H13    | 1.0930      | C26—C21   | 1.350 (5)  |
| C8—H5     | 1.1010      | C26—H23   | 1.0930     |
| C8—H6     | 1.1010      | F1b—C21b  | 1.332 (15) |
| C11—C10   | 1.387 (2)   | C21b—C26b | 1.360 (13) |
| C11—H15   | 1.0930      | C21b—C22b | 1.356 (13) |
| C14—H18   | 1.1010      | C26b—C25b | 1.364 (13) |
| C14—H17   | 1.1010      | C26b—H23b | 1.0930     |
| C10—H14   | 1.0930      | C25b—C24b | 1.374 (13) |
| C20—H11   | 1.1010      | C25b—H22b | 1.0930     |
| C20—H12   | 1.1010      | C24b—C23b | 1.382 (14) |
| C15—C16   | 1.387 (2)   | C24b—H21b | 1.0930     |
| C15—H7    | 1.0930      | C23b—C22b | 1.373 (13) |
| C17—C16   | 1.385 (3)   | C23b—H20b | 1.0930     |
| C17—H9    | 1.0930      | C22b—H19b | 1.0930     |
| N5—Fe1—N4 | 75.63 (4)   | H7—C15—N6 | 118.65 (9) |

|             |             |                |             |
|-------------|-------------|----------------|-------------|
| N6—Fe1—N4   | 75.57 (4)   | H7—C15—C16     | 118.65 (10) |
| N6—Fe1—N5   | 151.19 (4)  | N1—C1—S1       | 178.66 (16) |
| N3—Fe1—N4   | 77.88 (5)   | C16—C17—C18    | 119.10 (15) |
| N3—Fe1—N5   | 89.58 (5)   | H9—C17—C18     | 120.45 (10) |
| N3—Fe1—N6   | 83.76 (5)   | H9—C17—C16     | 120.45 (10) |
| N1—Fe1—N4   | 91.90 (5)   | C17—C16—C15    | 118.53 (15) |
| N1—Fe1—N5   | 91.90 (5)   | H8—C16—C15     | 120.73 (10) |
| N1—Fe1—N6   | 89.73 (5)   | H8—C16—C17     | 120.73 (10) |
| N1—Fe1—N3   | 168.98 (5)  | C5—C6—C7       | 119.5 (2)   |
| N2—Fe1—N4   | 171.52 (6)  | H4—C6—C7       | 120.27 (12) |
| N2—Fe1—N5   | 101.30 (5)  | H4—C6—C5       | 120.27 (13) |
| N2—Fe1—N6   | 107.12 (6)  | C4—C3—N3       | 122.7 (2)   |
| N2—Fe1—N3   | 94.29 (6)   | H1—C3—N3       | 118.64 (11) |
| N2—Fe1—N1   | 96.12 (6)   | H1—C3—C4       | 118.64 (14) |
| C8—N4—Fe1   | 111.00 (9)  | C23a—C24a—C25a | 121.9 (5)   |
| C14—N4—Fe1  | 105.86 (8)  | H21a—C24a—C25a | 119.1 (3)   |
| C14—N4—C8   | 111.13 (11) | H21a—C24a—C23a | 119.1 (4)   |
| C20—N4—Fe1  | 105.92 (8)  | C5—C4—C3       | 118.5 (2)   |
| C20—N4—C8   | 112.07 (12) | H2—C4—C3       | 120.74 (14) |
| C20—N4—C14  | 110.57 (11) | H2—C4—C5       | 120.74 (12) |
| C13—N5—Fe1  | 114.76 (9)  | C26a—C25a—C24a | 119.0 (5)   |
| C9—N5—Fe1   | 126.71 (10) | H22a—C25a—C24a | 120.5 (3)   |
| C9—N5—C13   | 118.53 (13) | H22a—C25a—C26a | 120.5 (4)   |
| C19—N6—Fe1  | 115.14 (9)  | C4—C5—C6       | 118.86 (19) |
| C15—N6—Fe1  | 126.18 (10) | H3—C5—C6       | 120.57 (13) |
| C15—N6—C19  | 118.68 (13) | H3—C5—C4       | 120.57 (12) |
| C7—N3—Fe1   | 116.05 (10) | C22a—C21a—C26a | 117.8 (5)   |
| C3—N3—Fe1   | 125.09 (12) | F1a—C21a—C26a  | 106.6 (10)  |
| C3—N3—C7    | 118.62 (15) | F1a—C21a—C22a  | 135.3 (10)  |
| C1—N1—Fe1   | 169.45 (15) | C22a—C23a—C24a | 118.0 (6)   |
| C2—N2—Fe1   | 178.27 (15) | H20a—C23a—C24a | 121.0 (4)   |
| C18—C19—N6  | 122.08 (13) | H20a—C23a—C22a | 121.0 (4)   |
| C20—C19—N6  | 116.10 (12) | C21a—C26a—C25a | 121.7 (5)   |
| C20—C19—C18 | 121.75 (13) | H23a—C26a—C25a | 119.1 (4)   |
| C17—C18—C19 | 118.87 (15) | H23a—C26a—C21a | 119.1 (4)   |
| H10—C18—C19 | 120.57 (9)  | C23a—C22a—C21a | 121.5 (6)   |
| H10—C18—C17 | 120.57 (10) | H19a—C22a—C21a | 119.2 (4)   |
| C12—C13—N5  | 122.20 (13) | H19a—C22a—C23a | 119.2 (4)   |
| C14—C13—N5  | 116.15 (12) | C21—C22—C23    | 117.8 (5)   |
| C14—C13—C12 | 121.62 (13) | H19—C22—C23    | 121.1 (4)   |
| C11—C12—C13 | 119.03 (15) | H19—C22—C21    | 121.1 (4)   |
| H16—C12—C13 | 120.48 (9)  | C24—C23—C22    | 121.5 (6)   |
| H16—C12—C11 | 120.48 (10) | H20—C23—C22    | 119.2 (4)   |
| C8—C7—N3    | 118.54 (13) | H20—C23—C24    | 119.2 (4)   |
| C6—C7—N3    | 121.81 (16) | C25—C24—C23    | 118.0 (6)   |
| C6—C7—C8    | 119.59 (16) | H21—C24—C23    | 121.0 (4)   |
| C10—C9—N5   | 122.80 (15) | H21—C24—C25    | 121.0 (4)   |
| H13—C9—N5   | 118.60 (9)  | C26—C25—C24    | 121.9 (5)   |



|                |              |                     |              |
|----------------|--------------|---------------------|--------------|
| H13—C9—C10     | 118.60 (9)   | H22—C25—C24         | 119.1 (4)    |
| C7—C8—N4       | 115.59 (12)  | H22—C25—C26         | 119.1 (3)    |
| H5—C8—N4       | 108.38 (8)   | C21—C26—C25         | 119.0 (5)    |
| H5—C8—C7       | 108.38 (8)   | H23—C26—C25         | 120.5 (3)    |
| H6—C8—N4       | 108.38 (8)   | H23—C26—C21         | 120.5 (4)    |
| H6—C8—C7       | 108.38 (9)   | C22—C21—F1          | 114.8 (7)    |
| H6—C8—H5       | 107.4        | C26—C21—F1          | 123.5 (8)    |
| N2—C2—S2       | 179.25 (16)  | C26—C21—C22         | 121.7 (5)    |
| C10—C11—C12    | 118.77 (15)  | C26b—C21b—F1b       | 110.4 (14)   |
| H15—C11—C12    | 120.62 (10)  | C22b—C21b—F1b       | 127.6 (15)   |
| H15—C11—C10    | 120.62 (9)   | C22b—C21b—C26b      | 121.5 (15)   |
| C13—C14—N4     | 111.22 (11)  | C25b—C26b—C21b      | 119.5 (16)   |
| H18—C14—N4     | 109.39 (8)   | H23b—C26b—C21b      | 120.3 (10)   |
| H18—C14—C13    | 109.39 (8)   | H23b—C26b—C25b      | 120.3 (10)   |
| H17—C14—N4     | 109.39 (8)   | C24b—C25b—C26b      | 119.6 (15)   |
| H17—C14—C13    | 109.39 (8)   | H22b—C25b—C26b      | 120.2 (10)   |
| H17—C14—H18    | 108.0        | H22b—C25b—C24b      | 120.2 (10)   |
| C11—C10—C9     | 118.67 (15)  | C23b—C24b—C25b      | 120.8 (15)   |
| H14—C10—C9     | 120.67 (9)   | H21b—C24b—C25b      | 119.6 (10)   |
| H14—C10—C11    | 120.67 (9)   | H21b—C24b—C23b      | 119.6 (10)   |
| C19—C20—N4     | 111.64 (11)  | C22b—C23b—C24b      | 118.5 (16)   |
| H11—C20—N4     | 109.29 (8)   | H20b—C23b—C24b      | 120.8 (10)   |
| H11—C20—C19    | 109.29 (8)   | H20b—C23b—C22b      | 120.8 (11)   |
| H12—C20—N4     | 109.29 (8)   | C23b—C22b—C21b      | 120.0 (16)   |
| H12—C20—C19    | 109.29 (9)   | H19b—C22b—C21b      | 120.0 (10)   |
| H12—C20—H11    | 108.0        | H19b—C22b—C23b      | 120.0 (11)   |
| C16—C15—N6     | 122.70 (15)  |                     |              |
| Fe1—N4—C8—C7   | −9.03 (10)   | C7—N3—C3—C4         | −0.5 (2)     |
| Fe1—N4—C14—C13 | −43.43 (9)   | C7—C8—N4—C14        | −126.55 (13) |
| Fe1—N4—C20—C19 | 42.45 (9)    | C7—C8—N4—C20        | 109.18 (14)  |
| Fe1—N5—C13—C12 | 179.67 (10)  | C7—C6—C5—C4         | 0.1 (2)      |
| Fe1—N5—C13—C14 | −2.40 (11)   | C9—N5—C13—C14       | 177.88 (14)  |
| Fe1—N5—C9—C10  | 179.59 (13)  | C8—C7—N3—C3         | 177.49 (16)  |
| Fe1—N6—C19—C18 | 178.50 (10)  | C8—C7—C6—C5         | −177.3 (2)   |
| Fe1—N6—C19—C20 | 1.62 (11)    | C11—C12—C13—C14     | −177.30 (15) |
| Fe1—N6—C15—C16 | −177.47 (13) | C20—C19—N6—C15      | −177.92 (13) |
| Fe1—N3—C7—C8   | −7.87 (12)   | C20—C19—C18—C17     | 175.82 (16)  |
| Fe1—N3—C7—C6   | 175.15 (12)  | C6—C7—N3—C3         | 0.5 (2)      |
| Fe1—N3—C3—C4   | −174.61 (18) | C6—C5—C4—C3         | −0.1 (3)     |
| N4—C8—C7—N3    | 11.60 (16)   | C24a—C25a—C26a—C21a | 0.6 (5)      |
| N4—C8—C7—C6    | −171.35 (15) | C24a—C23a—C22a—C21a | −0.3 (5)     |
| N4—C14—C13—N5  | 32.25 (14)   | C25a—C24a—C23a—C22a | 1.7 (6)      |
| N4—C14—C13—C12 | −149.80 (12) | C25a—C26a—C21a—C22a | 0.7 (4)      |
| N4—C20—C19—N6  | −31.04 (15)  | C25a—C26a—C21a—F1a  | 175.4 (6)    |
| N4—C20—C19—C18 | 152.07 (13)  | C23a—C24a—C25a—C26a | −1.9 (5)     |
| N5—C13—C12—C11 | 0.52 (18)    | C23a—C22a—C21a—C26a | −0.9 (6)     |
| N5—C9—C10—C11  | 1.01 (19)    | C23a—C22a—C21a—F1a  | −173.6 (8)   |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N6—C19—C18—C17  | −0.89 (17)   | F1—C21—C22—C23      | −177.8 (5)  |
| N6—C15—C16—C17  | −1.0 (2)     | F1—C21—C26—C25      | 179.0 (5)   |
| N3—C7—C6—C5     | −0.3 (2)     | C22—C23—C24—C25     | −0.3 (6)    |
| N3—C3—C4—C5     | 0.3 (3)      | C22—C21—C26—C25     | 0.6 (5)     |
| C19—N6—C15—C16  | 2.02 (16)    | C23—C22—C21—C26     | 0.7 (5)     |
| C19—C18—C17—C16 | 1.87 (19)    | C23—C24—C25—C26     | 1.7 (6)     |
| C19—C20—N4—C8   | −78.73 (14)  | C24—C23—C22—C21     | −0.9 (6)    |
| C19—C20—N4—C14  | 156.69 (13)  | C24—C25—C26—C21     | −1.9 (5)    |
| C18—C19—N6—C15  | −1.04 (18)   | F1b—C21b—C26b—C25b  | −174.3 (13) |
| C18—C17—C16—C15 | −1.0 (2)     | F1b—C21b—C22b—C23b  | 175 (2)     |
| C13—N5—C9—C10   | −0.72 (16)   | C21b—C26b—C25b—C24b | −0 (2)      |
| C13—C12—C11—C10 | −0.22 (18)   | C21b—C22b—C23b—C24b | −4 (3)      |
| C13—C14—N4—C8   | 77.17 (14)   | C26b—C21b—C22b—C23b | 4 (2)       |
| C13—C14—N4—C20  | −157.71 (12) | C26b—C25b—C24b—C23b | −0 (3)      |
| C12—C13—N5—C9   | −0.05 (18)   | C25b—C26b—C21b—C22b | −2 (3)      |
| C12—C11—C10—C9  | −0.5 (2)     | C25b—C24b—C23b—C22b | 2 (3)       |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) fluorobenzene monosolvate (f\_270K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>F

$M_r = 558.49$

Monoclinic,  $P2_1/n$

$a = 15.4857$  (3) Å

$b = 12.0619$  (2) Å

$c = 15.4996$  (4) Å

$\beta = 110.400$  (2)°

$V = 2713.55$  (10) Å<sup>3</sup>

$Z = 4$

$F(000) = 1155.072$

$D_x = 1.367$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 45168 reflections

$\theta = 2.1$ – $32.3^\circ$

$\mu = 0.74$  mm<sup>−1</sup>

$T = 270$  K

Block, yellow

$0.33 \times 0.22 \times 0.16$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.518$ ,  $T_{\max} = 1.000$

78108 measured reflections

6740 independent reflections

5967 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 17$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

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

$wR(F^2) = 0.113$

$S = 1.04$

6740 reflections

408 parameters

149 restraints

76 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.655P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.91$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.72$  e Å<sup>−3</sup>

*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  | 0.697609 (14) | 0.672269 (17) | 0.342341 (15) | 0.04006 (8)                      |           |
| S2   | 0.82588 (5)   | 0.99494 (5)   | 0.52708 (4)   | 0.07203 (16)                     |           |
| S1   | 0.95960 (10)  | 0.65816 (14)  | 0.24039 (14)  | 0.1064 (3)                       |           |
| N4   | 0.61633 (8)   | 0.53065 (10)  | 0.25798 (9)   | 0.0411 (3)                       |           |
| N5   | 0.60943 (8)   | 0.74996 (10)  | 0.21213 (9)   | 0.0423 (3)                       |           |
| N6   | 0.74474 (9)   | 0.52540 (11)  | 0.43166 (9)   | 0.0443 (3)                       |           |
| N3   | 0.57822 (10)  | 0.66349 (12)  | 0.38842 (10)  | 0.0497 (3)                       |           |
| N1   | 0.80624 (11)  | 0.64887 (13)  | 0.29091 (12)  | 0.0580 (4)                       |           |
| N2   | 0.75352 (12)  | 0.80884 (14)  | 0.42114 (12)  | 0.0647 (4)                       |           |
| C19  | 0.72423 (10)  | 0.42756 (13)  | 0.38836 (11)  | 0.0435 (3)                       |           |
| C18  | 0.75038 (13)  | 0.32751 (14)  | 0.43456 (14)  | 0.0548 (4)                       |           |
| H00B | 0.73211 (13)  | 0.24951 (14)  | 0.39811 (14)  | 0.0658 (5)*                      |           |
| C13  | 0.57330 (11)  | 0.67925 (12)  | 0.14219 (11)  | 0.0438 (3)                       |           |
| C7   | 0.51797 (10)  | 0.58257 (15)  | 0.35154 (11)  | 0.0498 (4)                       |           |
| C12  | 0.51624 (12)  | 0.71455 (16)  | 0.05578 (12)  | 0.0535 (4)                       |           |
| H00E | 0.48773 (12)  | 0.65539 (16)  | 0.00057 (12)  | 0.0643 (5)*                      |           |
| C2   | 0.78308 (12)  | 0.88673 (14)  | 0.46535 (12)  | 0.0511 (4)                       |           |
| C9   | 0.58960 (11)  | 0.85780 (14)  | 0.19797 (13)  | 0.0494 (4)                       |           |
| H00G | 0.61808 (11)  | 0.91491 (14)  | 0.25475 (13)  | 0.0593 (4)*                      |           |
| C11  | 0.49631 (14)  | 0.82635 (16)  | 0.04121 (14)  | 0.0604 (5)                       |           |
| H00H | 0.45204 (14)  | 0.85603 (16)  | −0.02550 (14) | 0.0724 (6)*                      |           |
| C20  | 0.67589 (13)  | 0.43211 (13)  | 0.28589 (12)  | 0.0517 (4)                       |           |
| H00a | 0.63415 (13)  | 0.35759 (13)  | 0.26331 (12)  | 0.0620 (5)*                      |           |
| H00c | 0.72703 (13)  | 0.43364 (13)  | 0.25213 (12)  | 0.0620 (5)*                      |           |
| C8   | 0.52847 (12)  | 0.51661 (16)  | 0.27339 (13)  | 0.0549 (4)                       |           |
| H00d | 0.47262 (12)  | 0.53937 (16)  | 0.21028 (13)  | 0.0659 (5)*                      |           |
| H00f | 0.52019 (12)  | 0.42897 (16)  | 0.28610 (13)  | 0.0659 (5)*                      |           |
| C1   | 0.87046 (12)  | 0.65155 (15)  | 0.26986 (14)  | 0.0552 (4)                       |           |
| C14  | 0.60086 (13)  | 0.55945 (13)  | 0.16155 (11)  | 0.0501 (4)                       |           |
| H00i | 0.66378 (13)  | 0.54420 (13)  | 0.14684 (11)  | 0.0601 (4)*                      |           |
| H00j | 0.54676 (13)  | 0.50660 (13)  | 0.11632 (11)  | 0.0601 (4)*                      |           |
| C10  | 0.53392 (13)  | 0.89949 (15)  | 0.11330 (14)  | 0.0588 (4)                       |           |
| H00M | 0.52023 (13)  | 0.98755 (15)  | 0.10393 (14)  | 0.0706 (5)*                      |           |
| C15  | 0.79053 (12)  | 0.52595 (16)  | 0.52232 (12)  | 0.0554 (4)                       |           |
| H00N | 0.80513 (12)  | 0.60492 (16)  | 0.55770 (12)  | 0.0665 (5)*                      |           |
| C17  | 0.80032 (15)  | 0.32941 (17)  | 0.52824 (15)  | 0.0646 (5)                       |           |
| H00O | 0.82343 (15)  | 0.25286 (17)  | 0.56570 (15)  | 0.0775 (6)*                      |           |
| C16  | 0.82018 (13)  | 0.43009 (19)  | 0.57308 (13)  | 0.0652 (5)                       |           |
| H00P | 0.85813 (13)  | 0.43401 (19)  | 0.64638 (13)  | 0.0782 (6)*                      |           |
| C6   | 0.44402 (14)  | 0.5622 (2)    | 0.38018 (16)  | 0.0760 (6)                       |           |
| H00Q | 0.39578 (14)  | 0.4963 (2)    | 0.34871 (16)  | 0.0912 (8)*                      |           |
| C3   | 0.56676 (15)  | 0.7255 (2)    | 0.45527 (14)  | 0.0713 (6)                       |           |
| H00R | 0.61591 (15)  | 0.7909 (2)    | 0.48556 (14)  | 0.0855 (7)*                      |           |
| C4   | 0.49483 (19)  | 0.7094 (3)    | 0.48716 (18)  | 0.0945 (9)                       |           |
| H00T | 0.48764 (19)  | 0.7612 (3)    | 0.54131 (18)  | 0.1134 (10)*                     |           |

|      |              |             |              |              |           |
|------|--------------|-------------|--------------|--------------|-----------|
| C5   | 0.43231 (18) | 0.6260 (3)  | 0.44866 (19) | 0.0977 (9)   |           |
| H00V | 0.37507 (18) | 0.6110 (3)  | 0.47202 (19) | 0.1172 (11)* |           |
| C25  | 0.3641 (4)   | 0.7822 (6)  | 0.2186 (5)   | 0.118 (2)    | 0.703 (8) |
| C26  | 0.2857 (5)   | 0.7213 (4)  | 0.1902 (3)   | 0.1085 (17)  | 0.703 (8) |
| C21a | 0.2251 (7)   | 0.8381 (9)  | 0.2795 (6)   | 0.165 (4)    | 0.177 (8) |
| C21  | 0.2172 (4)   | 0.7494 (6)  | 0.2207 (4)   | 0.1287 (19)  | 0.703 (8) |
| C24  | 0.3752 (6)   | 0.8714 (6)  | 0.2745 (5)   | 0.163 (3)    | 0.703 (8) |
| F1   | 0.1464 (5)   | 0.7059 (12) | 0.2000 (7)   | 0.379 (9)    | 0.703 (8) |
| C23  | 0.3050 (9)   | 0.8983 (6)  | 0.3046 (5)   | 0.198 (5)    | 0.703 (8) |
| C26a | 0.2172 (4)   | 0.7494 (6)  | 0.2207 (4)   | 0.1287 (19)  | 0.177 (8) |
| C22  | 0.2251 (7)   | 0.8381 (9)  | 0.2795 (6)   | 0.165 (4)    | 0.703 (8) |
| C22a | 0.3050 (9)   | 0.8983 (6)  | 0.3046 (5)   | 0.198 (5)    | 0.177 (8) |
| C23a | 0.3752 (6)   | 0.8714 (6)  | 0.2745 (5)   | 0.163 (3)    | 0.177 (8) |
| C24a | 0.3641 (4)   | 0.7822 (6)  | 0.2186 (5)   | 0.118 (2)    | 0.177 (8) |
| C25a | 0.2857 (5)   | 0.7213 (4)  | 0.1902 (3)   | 0.1085 (17)  | 0.177 (8) |
| F1a  | 0.1450 (8)   | 0.8486 (12) | 0.2911 (9)   | 0.136 (7)    | 0.177 (8) |
| F1b  | 0.3876 (11)  | 0.9896 (11) | 0.3750 (11)  | 0.114 (4)    | 0.12      |
| C21b | 0.3511 (14)  | 0.912 (2)   | 0.3116 (18)  | 0.124 (9)    | 0.12      |
| C26b | 0.2621 (14)  | 0.8880 (19) | 0.3024 (19)  | 0.110 (9)    | 0.12      |
| C25b | 0.2162 (14)  | 0.8041 (17) | 0.2448 (18)  | 0.074 (5)    | 0.12      |
| C24b | 0.2617 (13)  | 0.7457 (17) | 0.1966 (16)  | 0.064 (5)    | 0.12      |
| C23b | 0.3519 (13)  | 0.7719 (16) | 0.2069 (13)  | 0.044 (4)    | 0.12      |
| C22b | 0.3974 (14)  | 0.8533 (17) | 0.2666 (15)  | 0.073 (6)    | 0.12      |
| H12  | 0.4691 (14)  | 0.8706 (17) | 0.2781 (15)  | 0.088 (7)*   | 0.12      |
| H11  | 0.3864 (13)  | 0.7276 (16) | 0.1673 (13)  | 0.052 (4)*   | 0.12      |
| H10  | 0.2266 (13)  | 0.6792 (17) | 0.1508 (16)  | 0.076 (6)*   | 0.12      |
| H9   | 0.1458 (14)  | 0.7840 (17) | 0.2372 (18)  | 0.089 (6)*   | 0.12      |
| H8   | 0.2277 (14)  | 0.9349 (19) | 0.3405 (19)  | 0.132 (10)*  | 0.12      |
| H6   | 0.3126 (9)   | 0.9692 (6)  | 0.3495 (5)   | 0.238 (6)*   | 0.703 (8) |
| H4a  | 0.1702 (7)   | 0.8590 (9)  | 0.3048 (6)   | 0.198 (5)*   | 0.703 (8) |
| H2   | 0.2783 (5)   | 0.6517 (4)  | 0.1440 (3)   | 0.130 (2)*   | 0.703 (8) |
| H1   | 0.4201 (4)   | 0.7585 (6)  | 0.1958 (5)   | 0.141 (3)*   | 0.703 (8) |
| H5   | 0.4379 (6)   | 0.9199 (6)  | 0.2946 (5)   | 0.196 (4)*   | 0.703 (8) |
| H3a  | 0.3126 (9)   | 0.9692 (6)  | 0.3495 (5)   | 0.238 (6)*   | 0.177 (8) |
| H6a  | 0.4379 (6)   | 0.9199 (6)  | 0.2946 (5)   | 0.196 (4)*   | 0.177 (8) |
| H5a  | 0.4201 (4)   | 0.7585 (6)  | 0.1958 (5)   | 0.141 (3)*   | 0.177 (8) |
| H1a  | 0.2783 (5)   | 0.6517 (4)  | 0.1440 (3)   | 0.130 (2)*   | 0.177 (8) |
| H2a  | 0.1542 (4)   | 0.7012 (6)  | 0.1986 (4)   | 0.154 (2)*   | 0.177 (8) |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1 | 0.03617 (13) | 0.03473 (13) | 0.04657 (14) | −0.00162 (7) | 0.01099 (9) | −0.00459 (8) |
| S2  | 0.1007 (4)   | 0.0553 (3)   | 0.0644 (3)   | −0.0213 (3)  | 0.0343 (3)  | −0.0224 (2)  |
| S1  | 0.0682 (4)   | 0.1090 (5)   | 0.1661 (8)   | 0.0379 (4)   | 0.0711 (5)  | 0.0727 (5)   |
| N4  | 0.0428 (6)   | 0.0330 (6)   | 0.0438 (6)   | 0.0006 (5)   | 0.0107 (5)  | −0.0049 (5)  |
| N5  | 0.0399 (6)   | 0.0355 (6)   | 0.0482 (7)   | 0.0009 (5)   | 0.0110 (5)  | −0.0020 (5)  |
| N6  | 0.0404 (6)   | 0.0391 (6)   | 0.0483 (7)   | −0.0016 (5)  | 0.0091 (5)  | −0.0003 (5)  |

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|      |             |             |             |              |             |              |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| N3   | 0.0419 (7)  | 0.0589 (9)  | 0.0499 (7)  | 0.0030 (6)   | 0.0179 (6)  | −0.0066 (6)  |
| N1   | 0.0485 (8)  | 0.0541 (8)  | 0.0783 (10) | 0.0049 (6)   | 0.0307 (7)  | 0.0095 (7)   |
| N2   | 0.0654 (10) | 0.0509 (9)  | 0.0691 (10) | −0.0156 (7)  | 0.0126 (8)  | −0.0201 (7)  |
| C19  | 0.0413 (7)  | 0.0361 (7)  | 0.0505 (8)  | 0.0016 (6)   | 0.0128 (6)  | 0.0007 (6)   |
| C18  | 0.0565 (10) | 0.0431 (9)  | 0.0610 (10) | 0.0057 (7)   | 0.0156 (8)  | 0.0082 (7)   |
| C13  | 0.0464 (8)  | 0.0386 (7)  | 0.0440 (8)  | 0.0008 (6)   | 0.0126 (6)  | −0.0004 (6)  |
| C7   | 0.0372 (7)  | 0.0612 (10) | 0.0494 (8)  | 0.0009 (7)   | 0.0132 (6)  | 0.0057 (7)   |
| C12  | 0.0591 (9)  | 0.0500 (9)  | 0.0454 (8)  | −0.0014 (8)  | 0.0104 (7)  | 0.0029 (7)   |
| C2   | 0.0569 (9)  | 0.0451 (9)  | 0.0506 (8)  | −0.0096 (7)  | 0.0179 (7)  | −0.0095 (7)  |
| C9   | 0.0450 (8)  | 0.0360 (7)  | 0.0628 (10) | 0.0000 (6)   | 0.0131 (7)  | 0.0001 (7)   |
| C11  | 0.0614 (11) | 0.0541 (11) | 0.0565 (10) | 0.0035 (8)   | 0.0091 (8)  | 0.0145 (8)   |
| C20  | 0.0621 (10) | 0.0349 (7)  | 0.0517 (9)  | 0.0085 (7)   | 0.0119 (7)  | −0.0043 (6)  |
| C8   | 0.0459 (8)  | 0.0530 (10) | 0.0622 (10) | −0.0106 (7)  | 0.0142 (7)  | −0.0069 (8)  |
| C1   | 0.0426 (8)  | 0.0508 (9)  | 0.0747 (11) | 0.0100 (7)   | 0.0238 (8)  | 0.0204 (8)   |
| C14  | 0.0634 (10) | 0.0390 (8)  | 0.0431 (8)  | 0.0042 (7)   | 0.0125 (7)  | −0.0060 (6)  |
| C10  | 0.0569 (9)  | 0.0416 (9)  | 0.0710 (11) | 0.0021 (7)   | 0.0135 (8)  | 0.0103 (8)   |
| C15  | 0.0514 (9)  | 0.0554 (10) | 0.0493 (9)  | −0.0040 (7)  | 0.0046 (7)  | −0.0022 (7)  |
| C17  | 0.0657 (11) | 0.0590 (12) | 0.0638 (11) | 0.0101 (9)   | 0.0160 (9)  | 0.0176 (9)   |
| C16  | 0.0603 (10) | 0.0709 (13) | 0.0520 (10) | 0.0027 (9)   | 0.0040 (8)  | 0.0106 (9)   |
| C6   | 0.0489 (10) | 0.1107 (19) | 0.0733 (13) | −0.0097 (11) | 0.0274 (9)  | 0.0069 (12)  |
| C3   | 0.0622 (11) | 0.0921 (16) | 0.0637 (11) | 0.0080 (11)  | 0.0271 (9)  | −0.0218 (11) |
| C4   | 0.0793 (15) | 0.145 (3)   | 0.0714 (14) | 0.0205 (17)  | 0.0422 (13) | −0.0165 (16) |
| C5   | 0.0631 (13) | 0.165 (3)   | 0.0800 (15) | 0.0008 (17)  | 0.0436 (12) | −0.0024 (18) |
| C25  | 0.111 (4)   | 0.133 (5)   | 0.116 (4)   | 0.035 (3)    | 0.049 (3)   | 0.051 (4)    |
| C26  | 0.146 (5)   | 0.120 (3)   | 0.0705 (19) | 0.059 (4)    | 0.052 (3)   | 0.020 (2)    |
| C21a | 0.240 (9)   | 0.177 (7)   | 0.131 (5)   | 0.141 (7)    | 0.131 (6)   | 0.070 (5)    |
| C21  | 0.095 (3)   | 0.174 (6)   | 0.118 (4)   | 0.039 (3)    | 0.038 (3)   | 0.065 (4)    |
| C24  | 0.252 (9)   | 0.120 (5)   | 0.114 (4)   | 0.066 (5)    | 0.058 (5)   | 0.033 (3)    |
| F1   | 0.175 (6)   | 0.59 (2)    | 0.366 (12)  | −0.004 (8)   | 0.093 (7)   | 0.239 (14)   |
| C23  | 0.378 (15)  | 0.099 (4)   | 0.121 (5)   | 0.084 (7)    | 0.092 (8)   | 0.023 (3)    |
| C26a | 0.095 (3)   | 0.174 (6)   | 0.118 (4)   | 0.039 (3)    | 0.038 (3)   | 0.065 (4)    |
| C22  | 0.240 (9)   | 0.177 (7)   | 0.131 (5)   | 0.141 (7)    | 0.131 (6)   | 0.070 (5)    |
| C22a | 0.378 (15)  | 0.099 (4)   | 0.121 (5)   | 0.084 (7)    | 0.092 (8)   | 0.023 (3)    |
| C23a | 0.252 (9)   | 0.120 (5)   | 0.114 (4)   | 0.066 (5)    | 0.058 (5)   | 0.033 (3)    |
| C24a | 0.111 (4)   | 0.133 (5)   | 0.116 (4)   | 0.035 (3)    | 0.049 (3)   | 0.051 (4)    |
| C25a | 0.146 (5)   | 0.120 (3)   | 0.0705 (19) | 0.059 (4)    | 0.052 (3)   | 0.020 (2)    |
| F1a  | 0.120 (10)  | 0.178 (13)  | 0.128 (10)  | 0.067 (8)    | 0.064 (8)   | 0.011 (8)    |
| F1b  | 0.138 (10)  | 0.081 (7)   | 0.146 (10)  | −0.005 (6)   | 0.078 (7)   | −0.025 (6)   |
| C21b | 0.112 (9)   | 0.123 (13)  | 0.176 (15)  | −0.016 (6)   | 0.100 (6)   | −0.062 (8)   |
| C26b | 0.114 (9)   | 0.085 (13)  | 0.169 (16)  | −0.011 (6)   | 0.097 (6)   | −0.058 (8)   |
| C25b | 0.085 (7)   | 0.049 (8)   | 0.126 (13)  | 0.009 (5)    | 0.084 (6)   | −0.021 (6)   |
| C24b | 0.060 (8)   | 0.072 (9)   | 0.082 (10)  | 0.030 (5)    | 0.052 (5)   | −0.004 (6)   |
| C23b | 0.049 (7)   | 0.055 (8)   | 0.046 (7)   | 0.038 (5)    | 0.040 (5)   | 0.020 (5)    |
| C22b | 0.082 (8)   | 0.071 (10)  | 0.080 (10)  | 0.017 (5)    | 0.047 (5)   | 0.000 (6)    |

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*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N4    | 2.2504 (12) | C16—H00P    | 1.0830      |
| Fe1—N5    | 2.2123 (13) | C6—H00Q     | 1.0830      |
| Fe1—N6    | 2.2098 (13) | C6—C5       | 1.373 (4)   |
| Fe1—N3    | 2.2046 (14) | C3—H00R     | 1.0830      |
| Fe1—N1    | 2.1165 (16) | C3—C4       | 1.381 (3)   |
| Fe1—N2    | 2.0537 (15) | C4—H00T     | 1.0830      |
| S2—C2     | 1.6175 (17) | C4—C5       | 1.380 (5)   |
| S1—C1     | 1.600 (2)   | C5—H00V     | 1.0830      |
| N4—C20    | 1.4739 (19) | C25—C26     | 1.354 (7)   |
| N4—C8     | 1.471 (2)   | C25—C24     | 1.354 (8)   |
| N4—C14    | 1.471 (2)   | C25—H1      | 1.0830      |
| N5—C13    | 1.339 (2)   | C26—C21     | 1.347 (6)   |
| N5—C9     | 1.337 (2)   | C26—H2      | 1.0830      |
| N6—C19    | 1.340 (2)   | C21a—C26a   | 1.383 (10)  |
| N6—C15    | 1.335 (2)   | C21a—C22a   | 1.370 (11)  |
| N3—C7     | 1.333 (2)   | C21a—F1a    | 1.320 (14)  |
| N3—C3     | 1.339 (2)   | C21—F1      | 1.155 (9)   |
| N1—C1     | 1.150 (2)   | C21—C22     | 1.383 (10)  |
| N2—C2     | 1.159 (2)   | C24—C23     | 1.363 (11)  |
| C19—C18   | 1.390 (2)   | C24—H5      | 1.0830      |
| C19—C20   | 1.502 (2)   | C23—C22     | 1.370 (11)  |
| C18—H00B  | 1.0830      | C23—H6      | 1.0830      |
| C18—C17   | 1.387 (3)   | C26a—C25a   | 1.347 (6)   |
| C13—C12   | 1.390 (2)   | C26a—H2a    | 1.0830      |
| C13—C14   | 1.507 (2)   | C22—H4a     | 1.0830      |
| C7—C8     | 1.505 (3)   | C22a—C23a   | 1.363 (11)  |
| C7—C6     | 1.387 (2)   | C22a—H3a    | 1.0830      |
| C12—H00E  | 1.0830      | C23a—C24a   | 1.354 (8)   |
| C12—C11   | 1.384 (3)   | C23a—H6a    | 1.0830      |
| C9—H00G   | 1.0830      | C24a—C25a   | 1.354 (7)   |
| C9—C10    | 1.389 (2)   | C24a—H5a    | 1.0830      |
| C11—H00H  | 1.0830      | C25a—H1a    | 1.0830      |
| C11—C10   | 1.383 (3)   | F1b—C21b    | 1.328 (15)  |
| C20—H00a  | 1.0910      | C21b—C26b   | 1.367 (14)  |
| C20—H00c  | 1.0910      | C21b—C22b   | 1.363 (13)  |
| C8—H00d   | 1.0910      | C26b—C25b   | 1.372 (13)  |
| C8—H00f   | 1.0910      | C26b—H8     | 1.0830      |
| C14—H00i  | 1.0910      | C25b—C24b   | 1.384 (12)  |
| C14—H00j  | 1.0910      | C25b—H9     | 1.0830      |
| C10—H00M  | 1.0830      | C24b—C23b   | 1.387 (13)  |
| C15—H00N  | 1.0830      | C24b—H10    | 1.0830      |
| C15—C16   | 1.384 (3)   | C23b—C22b   | 1.365 (13)  |
| C17—H00O  | 1.0830      | C23b—H11    | 1.0830      |
| C17—C16   | 1.380 (3)   | C22b—H12    | 1.0830      |
| N5—Fe1—N4 | 75.47 (4)   | H00N—C15—N6 | 118.54 (10) |

|              |             |                |             |
|--------------|-------------|----------------|-------------|
| N6—Fe1—N4    | 75.59 (5)   | C16—C15—N6     | 122.92 (17) |
| N6—Fe1—N5    | 151.06 (5)  | C16—C15—H00N   | 118.54 (11) |
| N3—Fe1—N4    | 77.84 (5)   | H00O—C17—C18   | 120.43 (11) |
| N3—Fe1—N5    | 89.73 (5)   | C16—C17—C18    | 119.14 (17) |
| N3—Fe1—N6    | 83.66 (5)   | C16—C17—H00O   | 120.43 (11) |
| N1—Fe1—N4    | 92.12 (6)   | C17—C16—C15    | 118.52 (17) |
| N1—Fe1—N5    | 91.82 (6)   | H00P—C16—C15   | 120.74 (11) |
| N1—Fe1—N6    | 89.82 (6)   | H00P—C16—C17   | 120.74 (11) |
| N1—Fe1—N3    | 169.12 (6)  | H00Q—C6—C7     | 120.19 (14) |
| N2—Fe1—N4    | 171.43 (6)  | C5—C6—C7       | 119.6 (2)   |
| N2—Fe1—N5    | 101.60 (6)  | C5—C6—H00Q     | 120.19 (16) |
| N2—Fe1—N6    | 106.96 (6)  | H00R—C3—N3     | 118.67 (12) |
| N2—Fe1—N3    | 94.19 (7)   | C4—C3—N3       | 122.7 (2)   |
| N2—Fe1—N1    | 96.04 (7)   | C4—C3—H00R     | 118.67 (17) |
| C20—N4—Fe1   | 106.00 (9)  | H00T—C4—C3     | 120.67 (17) |
| C8—N4—Fe1    | 110.78 (10) | C5—C4—C3       | 118.7 (2)   |
| C8—N4—C20    | 112.28 (14) | C5—C4—H00T     | 120.67 (14) |
| C14—N4—Fe1   | 105.74 (9)  | C4—C5—C6       | 118.8 (2)   |
| C14—N4—C20   | 110.64 (13) | H00V—C5—C6     | 120.62 (16) |
| C14—N4—C8    | 111.10 (13) | H00V—C5—C4     | 120.62 (14) |
| C13—N5—Fe1   | 114.81 (10) | C24—C25—C26    | 122.6 (7)   |
| C9—N5—Fe1    | 126.53 (11) | H1—C25—C26     | 118.7 (4)   |
| C9—N5—C13    | 118.65 (14) | H1—C25—C24     | 118.7 (5)   |
| C19—N6—Fe1   | 115.05 (10) | C21—C26—C25    | 118.4 (6)   |
| C15—N6—Fe1   | 126.42 (12) | H2—C26—C25     | 120.8 (4)   |
| C15—N6—C19   | 118.53 (14) | H2—C26—C21     | 120.8 (5)   |
| C7—N3—Fe1    | 115.89 (11) | C22a—C21a—C26a | 117.4 (6)   |
| C3—N3—Fe1    | 125.18 (14) | F1a—C21a—C26a  | 107.0 (12)  |
| C3—N3—C7     | 118.62 (17) | F1a—C21a—C22a  | 135.2 (13)  |
| C1—N1—Fe1    | 169.26 (17) | F1—C21—C26     | 125.1 (12)  |
| C2—N2—Fe1    | 178.46 (17) | C22—C21—C26    | 121.8 (6)   |
| C18—C19—N6   | 122.09 (15) | C22—C21—F1     | 113.1 (10)  |
| C20—C19—N6   | 116.17 (13) | C23—C24—C25    | 118.0 (8)   |
| C20—C19—C18  | 121.65 (14) | H5—C24—C25     | 121.0 (5)   |
| H00B—C18—C19 | 120.63 (10) | H5—C24—C23     | 121.0 (6)   |
| C17—C18—C19  | 118.75 (17) | C22—C23—C24    | 121.7 (7)   |
| C17—C18—H00B | 120.63 (11) | H6—C23—C24     | 119.1 (6)   |
| C12—C13—N5   | 122.08 (15) | H6—C23—C22     | 119.1 (6)   |
| C14—C13—N5   | 116.11 (14) | C25a—C26a—C21a | 121.8 (6)   |
| C14—C13—C12  | 121.77 (15) | H2a—C26a—C21a  | 119.1 (5)   |
| C8—C7—N3     | 118.46 (14) | H2a—C26a—C25a  | 119.1 (5)   |
| C6—C7—N3     | 121.66 (19) | C23—C22—C21    | 117.4 (6)   |
| C6—C7—C8     | 119.79 (18) | H4a—C22—C21    | 121.3 (5)   |
| H00E—C12—C13 | 120.45 (10) | H4a—C22—C23    | 121.3 (6)   |
| C11—C12—C13  | 119.11 (17) | C23a—C22a—C21a | 121.7 (7)   |
| C11—C12—H00E | 120.45 (11) | H3a—C22a—C21a  | 119.1 (6)   |
| N2—C2—S2     | 179.13 (18) | H3a—C22a—C23a  | 119.1 (6)   |
| H00G—C9—N5   | 118.70 (10) | C24a—C23a—C22a | 118.0 (8)   |



|                |              |                     |              |
|----------------|--------------|---------------------|--------------|
| C10—C9—N5      | 122.60 (17)  | H6a—C23a—C22a       | 121.0 (6)    |
| C10—C9—H00G    | 118.70 (11)  | H6a—C23a—C24a       | 121.0 (5)    |
| H00H—C11—C12   | 120.58 (11)  | C25a—C24a—C23a      | 122.6 (7)    |
| C10—C11—C12    | 118.85 (17)  | H5a—C24a—C23a       | 118.7 (5)    |
| C10—C11—H00H   | 120.58 (10)  | H5a—C24a—C25a       | 118.7 (4)    |
| C19—C20—N4     | 111.71 (13)  | C24a—C25a—C26a      | 118.4 (6)    |
| H00a—C20—N4    | 109.28 (9)   | H1a—C25a—C26a       | 120.8 (5)    |
| H00a—C20—C19   | 109.28 (9)   | H1a—C25a—C24a       | 120.8 (4)    |
| H00c—C20—N4    | 109.28 (9)   | C26b—C21b—F1b       | 112.5 (15)   |
| H00c—C20—C19   | 109.28 (10)  | C22b—C21b—F1b       | 125.5 (17)   |
| H00c—C20—H00a  | 107.9        | C22b—C21b—C26b      | 121.6 (16)   |
| C7—C8—N4       | 115.81 (13)  | C25b—C26b—C21b      | 120.1 (16)   |
| H00d—C8—N4     | 108.33 (9)   | H8—C26b—C21b        | 119.9 (10)   |
| H00d—C8—C7     | 108.33 (9)   | H8—C26b—C25b        | 119.9 (10)   |
| H00f—C8—N4     | 108.33 (9)   | C24b—C25b—C26b      | 118.8 (15)   |
| H00f—C8—C7     | 108.33 (10)  | H9—C25b—C26b        | 120.6 (10)   |
| H00f—C8—H00d   | 107.4        | H9—C25b—C24b        | 120.6 (10)   |
| N1—C1—S1       | 178.75 (18)  | C23b—C24b—C25b      | 120.3 (14)   |
| C13—C14—N4     | 111.31 (13)  | H10—C24b—C25b       | 119.9 (10)   |
| H00i—C14—N4    | 109.37 (9)   | H10—C24b—C23b       | 119.9 (9)    |
| H00i—C14—C13   | 109.37 (10)  | C22b—C23b—C24b      | 120.1 (13)   |
| H00j—C14—N4    | 109.37 (8)   | H11—C23b—C24b       | 119.9 (9)    |
| H00j—C14—C13   | 109.37 (9)   | H11—C23b—C22b       | 119.9 (9)    |
| H00j—C14—H00i  | 108.0        | C23b—C22b—C21b      | 119.0 (15)   |
| C11—C10—C9     | 118.71 (16)  | H12—C22b—C21b       | 120.5 (11)   |
| H00M—C10—C9    | 120.65 (11)  | H12—C22b—C23b       | 120.5 (9)    |
| H00M—C10—C11   | 120.65 (10)  |                     |              |
| Fe1—N4—C20—C19 | 42.34 (10)   | C7—C8—N4—C14        | −127.52 (15) |
| Fe1—N4—C8—C7   | −10.31 (11)  | C7—C6—C5—C4         | 0.2 (3)      |
| Fe1—N4—C14—C13 | −43.68 (10)  | C12—C13—N5—C9       | 0.1 (2)      |
| Fe1—N5—C13—C12 | 179.88 (12)  | C12—C11—C10—C9      | −0.6 (2)     |
| Fe1—N5—C13—C14 | −2.27 (12)   | C9—N5—C13—C14       | 177.95 (15)  |
| Fe1—N5—C9—C10  | 179.46 (14)  | C11—C12—C13—C14     | −177.43 (18) |
| Fe1—N6—C19—C18 | 178.93 (12)  | C20—C19—N6—C15      | −177.73 (15) |
| Fe1—N6—C19—C20 | 2.25 (12)    | C20—C19—C18—C17     | 175.65 (19)  |
| Fe1—N6—C15—C16 | −177.97 (15) | C8—C7—N3—C3         | 177.15 (18)  |
| Fe1—N3—C7—C8   | −8.81 (13)   | C8—C7—C6—C5         | −177.0 (2)   |
| Fe1—N3—C7—C6   | 174.60 (14)  | C6—C7—N3—C3         | 0.6 (2)      |
| Fe1—N3—C3—C4   | −173.8 (2)   | C6—C5—C4—C3         | −0.0 (3)     |
| N4—C20—C19—N6  | −31.40 (17)  | C25—C26—C21—F1      | 178.1 (6)    |
| N4—C20—C19—C18 | 151.90 (14)  | C25—C26—C21—C22     | 0.1 (5)      |
| N4—C8—C7—N3    | 13.16 (18)   | C25—C24—C23—C22     | −0.2 (6)     |
| N4—C8—C7—C6    | −170.19 (17) | C26—C25—C24—C23     | 1.7 (7)      |
| N4—C14—C13—N5  | 32.38 (16)   | C26—C21—C22—C23     | 1.3 (5)      |
| N4—C14—C13—C12 | −149.76 (14) | C21a—C26a—C25a—C24a | 0.1 (6)      |
| N5—C13—C12—C11 | 0.3 (2)      | C21a—C22a—C23a—C24a | −0.2 (8)     |
| N5—C9—C10—C11  | 1.1 (2)      | C21—C26—C25—C24     | −1.7 (5)     |

|                 |              |                     |             |
|-----------------|--------------|---------------------|-------------|
| N6—C19—C18—C17  | −0.85 (19)   | C21—C22—C23—C24     | −1.2 (6)    |
| N6—C15—C16—C17  | −1.0 (2)     | F1—C21—C22—C23      | −177.0 (7)  |
| N3—C7—C6—C5     | −0.5 (2)     | C26a—C21a—C22a—C23a | −1.2 (6)    |
| N3—C3—C4—C5     | 0.1 (3)      | C26a—C25a—C24a—C23a | −1.7 (5)    |
| C19—N6—C15—C16  | 2.00 (18)    | C22a—C21a—C26a—C25a | 1.3 (7)     |
| C19—C18—C17—C16 | 1.8 (2)      | C22a—C23a—C24a—C25a | 1.7 (6)     |
| C19—C20—N4—C8   | −78.74 (16)  | C23a—C22a—C21a—F1a  | −173.6 (9)  |
| C19—C20—N4—C14  | 156.51 (14)  | C25a—C26a—C21a—F1a  | 175.6 (7)   |
| C18—C19—N6—C15  | −1.0 (2)     | F1b—C21b—C26b—C25b  | −175.1 (14) |
| C18—C17—C16—C15 | −1.0 (2)     | F1b—C21b—C22b—C23b  | 176 (2)     |
| C13—N5—C9—C10   | −0.79 (18)   | C21b—C26b—C25b—C24b | −0 (2)      |
| C13—C12—C11—C10 | −0.0 (2)     | C21b—C22b—C23b—C24b | −4 (3)      |
| C13—C14—N4—C20  | −158.01 (14) | C26b—C21b—C22b—C23b | 4 (2)       |
| C13—C14—N4—C8   | 76.57 (16)   | C26b—C25b—C24b—C23b | 0 (3)       |
| C7—N3—C3—C4     | −0.4 (2)     | C25b—C26b—C21b—C22b | −2 (3)      |
| C7—C8—N4—C20    | 107.98 (16)  | C25b—C24b—C23b—C22b | 2 (3)       |

### Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) iodobenzene monosolvate (I\_115K)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 14.1401$  (13) Å

$b = 12.8265$  (9) Å

$c = 15.2870$  (13) Å

$\beta = 109.139$  (10)°

$V = 2619.3$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.690$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 6382 reflections

$\theta = 2.1$ – $24.2^\circ$

$\mu = 1.94$  mm<sup>−1</sup>

$T = 115$  K

Plate, red

$0.17 \times 0.1 \times 0.03$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.594$ ,  $T_{\max} = 1.000$

40403 measured reflections

4628 independent reflections

3199 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.142$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 1.7^\circ$

$h = -16 \rightarrow 16$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.108$

$wR(F^2) = 0.298$

$S = 1.85$

4628 reflections

313 parameters

0 restraints

44 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 2.52$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -2.42$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| Il   | 0.36822 (10) | 0.03590 (9)  | 0.43663 (8)  | 0.0665 (5)                       |
| Fe1  | 0.33700 (11) | 0.34064 (12) | 0.67850 (10) | 0.0182 (4)                       |
| S1   | 0.2556 (3)   | 0.3036 (3)   | 0.9519 (2)   | 0.0338 (8)                       |
| S2   | 0.5235 (2)   | 0.0540 (2)   | 0.8317 (2)   | 0.0341 (8)                       |
| N5   | 0.2277 (7)   | 0.2475 (7)   | 0.6068 (6)   | 0.021 (2)                        |
| N3   | 0.3898 (7)   | 0.3504 (7)   | 0.5765 (6)   | 0.024 (2)                        |
| N4   | 0.2432 (6)   | 0.4529 (7)   | 0.6140 (6)   | 0.020 (2)                        |
| N1   | 0.2846 (7)   | 0.3375 (7)   | 0.7822 (6)   | 0.026 (2)                        |
| N2   | 0.4272 (7)   | 0.2307 (8)   | 0.7399 (6)   | 0.025 (2)                        |
| N6   | 0.4212 (7)   | 0.4582 (7)   | 0.7366 (6)   | 0.024 (2)                        |
| C8   | 0.2644 (8)   | 0.4839 (8)   | 0.5299 (7)   | 0.022 (2)                        |
| C9   | 0.2333 (9)   | 0.1428 (9)   | 0.5938 (7)   | 0.025 (3)                        |
| C7   | 0.3458 (8)   | 0.4222 (8)   | 0.5127 (7)   | 0.021 (2)                        |
| C3   | 0.4625 (9)   | 0.2925 (9)   | 0.5639 (8)   | 0.026 (3)                        |
| C19  | 0.3704 (8)   | 0.5484 (9)   | 0.7268 (7)   | 0.023 (2)                        |
| C13  | 0.1414 (8)   | 0.2966 (9)   | 0.5675 (7)   | 0.020 (2)                        |
| C14  | 0.1409 (8)   | 0.4085 (8)   | 0.5925 (8)   | 0.024 (2)                        |
| C18  | 0.4184 (9)   | 0.6418 (9)   | 0.7619 (8)   | 0.027 (3)                        |
| C2   | 0.4678 (8)   | 0.1584 (8)   | 0.7787 (7)   | 0.021 (2)                        |
| C16  | 0.5731 (8)   | 0.5477 (9)   | 0.8182 (7)   | 0.024 (3)                        |
| C6   | 0.3777 (8)   | 0.4413 (10)  | 0.4365 (8)   | 0.028 (3)                        |
| C4   | 0.5001 (9)   | 0.3054 (9)   | 0.4915 (8)   | 0.031 (3)                        |
| C17  | 0.5214 (9)   | 0.6418 (10)  | 0.8086 (8)   | 0.032 (3)                        |
| C15  | 0.5213 (8)   | 0.4589 (10)  | 0.7809 (7)   | 0.026 (3)                        |
| C1   | 0.2728 (9)   | 0.3242 (8)   | 0.8531 (8)   | 0.024 (2)                        |
| C20  | 0.2607 (7)   | 0.5403 (8)   | 0.6798 (7)   | 0.020 (2)                        |
| C11  | 0.0620 (9)   | 0.1408 (9)   | 0.4975 (8)   | 0.029 (3)                        |
| C10  | 0.1511 (9)   | 0.0880 (9)   | 0.5391 (8)   | 0.030 (3)                        |
| C12  | 0.0576 (9)   | 0.2460 (9)   | 0.5110 (7)   | 0.026 (3)                        |
| C5   | 0.4524 (9)   | 0.3834 (10)  | 0.4237 (8)   | 0.031 (3)                        |
| C26  | 0.3015 (7)   | 0.1326 (7)   | 0.3291 (6)   | 0.048 (4)                        |
| C21  | 0.3184 (7)   | 0.1170 (7)   | 0.2454 (8)   | 0.050 (4)                        |
| C25  | 0.2438 (7)   | 0.2164 (8)   | 0.3394 (6)   | 0.051 (4)                        |
| C24  | 0.2028 (7)   | 0.2846 (7)   | 0.2660 (8)   | 0.058 (4)                        |
| C23  | 0.2197 (8)   | 0.2690 (8)   | 0.1824 (7)   | 0.075 (6)                        |
| C22  | 0.2775 (8)   | 0.1852 (9)   | 0.1721 (6)   | 0.065 (5)                        |
| H00X | 0.2304 (7)   | 0.2288 (8)   | 0.4058 (6)   | 0.062 (5)*                       |
| H00Y | 0.1570 (7)   | 0.3511 (7)   | 0.2742 (8)   | 0.070 (5)*                       |
| H00Z | 0.1872 (8)   | 0.3231 (8)   | 0.1242 (7)   | 0.090 (7)*                       |
| H010 | 0.2908 (8)   | 0.1728 (9)   | 0.1057 (6)   | 0.078 (6)*                       |
| H00W | 0.3642 (7)   | 0.0505 (7)   | 0.2373 (8)   | 0.060 (5)*                       |
| H00E | 0.4950 (9)   | 0.2301 (9)   | 0.6145 (8)   | 0.031 (3)*                       |
| H00L | 0.3420 (8)   | 0.5035 (10)  | 0.3868 (8)   | 0.034 (3)*                       |
| H00U | 0.4761 (9)   | 0.3959 (10)  | 0.3626 (8)   | 0.037 (3)*                       |
| H00M | 0.5632 (9)   | 0.2583 (9)   | 0.4860 (8)   | 0.037 (3)*                       |

|      |             |             |            |            |
|------|-------------|-------------|------------|------------|
| H00C | 0.3043 (9)  | 0.1014 (9)  | 0.6273 (7) | 0.030 (3)* |
| H00T | −0.0120 (9) | 0.2897 (9)  | 0.4771 (7) | 0.031 (3)* |
| H00R | −0.0047 (9) | 0.0984 (9)  | 0.4540 (8) | 0.035 (3)* |
| H00S | 0.1562 (9)  | 0.0034 (9)  | 0.5286 (8) | 0.036 (3)* |
| H00O | 0.5630 (8)  | 0.3850 (10) | 0.7872 (7) | 0.032 (3)* |
| H00K | 0.6544 (8)  | 0.5446 (9)  | 0.8552 (7) | 0.029 (3)* |
| H00N | 0.5610 (9)  | 0.7146 (10) | 0.8374 (8) | 0.039 (3)* |
| H00I | 0.3754 (9)  | 0.7151 (9)  | 0.7528 (8) | 0.032 (3)* |
| H00a | 0.1950 (8)  | 0.4750 (8)  | 0.4695 (7) | 0.027 (3)* |
| H00b | 0.2855 (8)  | 0.5676 (8)  | 0.5354 (7) | 0.027 (3)* |
| H00d | 0.1155 (8)  | 0.4166 (8)  | 0.6537 (8) | 0.028 (3)* |
| H00f | 0.0878 (8)  | 0.4520 (8)  | 0.5339 (8) | 0.028 (3)* |
| H00g | 0.2312 (7)  | 0.6139 (8)  | 0.6426 (7) | 0.024 (3)* |
| H00h | 0.2223 (7)  | 0.5257 (8)  | 0.7314 (7) | 0.024 (3)* |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| Il  | 0.0747 (9)  | 0.0564 (8)  | 0.0561 (8)  | −0.0002 (6)  | 0.0045 (6)  | −0.0017 (5)  |
| Fe1 | 0.0186 (8)  | 0.0199 (8)  | 0.0119 (7)  | 0.0022 (6)   | −0.0008 (6) | 0.0010 (6)   |
| S1  | 0.049 (2)   | 0.0323 (17) | 0.0217 (15) | −0.0086 (15) | 0.0141 (14) | −0.0036 (13) |
| S2  | 0.0308 (17) | 0.0295 (17) | 0.0376 (18) | 0.0072 (13)  | 0.0054 (14) | 0.0149 (14)  |
| N5  | 0.020 (5)   | 0.023 (5)   | 0.015 (4)   | 0.001 (4)    | 0.001 (4)   | −0.007 (4)   |
| N3  | 0.030 (5)   | 0.020 (5)   | 0.024 (5)   | −0.001 (4)   | 0.013 (4)   | −0.002 (4)   |
| N4  | 0.014 (4)   | 0.020 (5)   | 0.018 (5)   | −0.000 (4)   | −0.004 (4)  | 0.002 (4)    |
| N1  | 0.037 (6)   | 0.026 (5)   | 0.020 (5)   | −0.002 (4)   | 0.017 (4)   | 0.001 (4)    |
| N2  | 0.019 (5)   | 0.029 (6)   | 0.022 (5)   | 0.005 (4)    | 0.002 (4)   | 0.002 (4)    |
| N6  | 0.019 (5)   | 0.037 (6)   | 0.010 (4)   | −0.003 (4)   | −0.003 (4)  | −0.001 (4)   |
| C8  | 0.025 (6)   | 0.020 (6)   | 0.018 (5)   | 0.000 (5)    | 0.001 (5)   | −0.001 (4)   |
| C9  | 0.031 (6)   | 0.023 (6)   | 0.018 (5)   | 0.006 (5)    | 0.005 (5)   | 0.005 (4)    |
| C7  | 0.029 (6)   | 0.019 (6)   | 0.012 (5)   | −0.002 (4)   | 0.003 (5)   | −0.001 (4)   |
| C3  | 0.026 (6)   | 0.027 (6)   | 0.022 (6)   | −0.002 (5)   | 0.005 (5)   | −0.004 (5)   |
| C19 | 0.017 (6)   | 0.027 (6)   | 0.017 (5)   | 0.001 (5)    | −0.006 (4)  | −0.005 (4)   |
| C13 | 0.016 (6)   | 0.025 (6)   | 0.014 (5)   | −0.002 (5)   | −0.005 (4)  | 0.001 (4)    |
| C14 | 0.016 (5)   | 0.021 (6)   | 0.026 (6)   | −0.001 (4)   | −0.003 (5)  | −0.006 (5)   |
| C18 | 0.023 (6)   | 0.033 (7)   | 0.022 (6)   | 0.003 (5)    | 0.002 (5)   | 0.000 (5)    |
| C2  | 0.019 (5)   | 0.019 (6)   | 0.019 (5)   | 0.002 (5)    | −0.002 (4)  | 0.003 (5)    |
| C16 | 0.015 (6)   | 0.036 (7)   | 0.016 (5)   | −0.002 (5)   | −0.004 (4)  | −0.001 (5)   |
| C6  | 0.027 (6)   | 0.037 (7)   | 0.019 (6)   | −0.001 (5)   | 0.004 (5)   | 0.010 (5)    |
| C4  | 0.027 (6)   | 0.033 (7)   | 0.030 (7)   | 0.001 (5)    | 0.007 (5)   | −0.005 (5)   |
| C17 | 0.030 (7)   | 0.042 (8)   | 0.020 (6)   | −0.011 (6)   | 0.001 (5)   | 0.000 (5)    |
| C15 | 0.015 (6)   | 0.042 (7)   | 0.016 (5)   | 0.007 (5)    | −0.004 (5)  | 0.009 (5)    |
| C1  | 0.030 (6)   | 0.024 (6)   | 0.022 (6)   | −0.003 (5)   | 0.014 (5)   | −0.006 (5)   |
| C20 | 0.015 (5)   | 0.018 (5)   | 0.024 (6)   | −0.002 (4)   | 0.002 (5)   | −0.004 (4)   |
| C11 | 0.033 (7)   | 0.027 (7)   | 0.020 (6)   | −0.002 (5)   | −0.000 (5)  | 0.001 (5)    |
| C10 | 0.041 (7)   | 0.024 (6)   | 0.020 (6)   | −0.010 (5)   | 0.004 (5)   | −0.009 (5)   |
| C12 | 0.023 (6)   | 0.025 (6)   | 0.022 (6)   | −0.000 (5)   | −0.002 (5)  | −0.003 (5)   |
| C5  | 0.034 (7)   | 0.042 (7)   | 0.019 (6)   | −0.011 (6)   | 0.012 (5)   | −0.000 (5)   |

|     |            |            |            |            |            |             |
|-----|------------|------------|------------|------------|------------|-------------|
| C26 | 0.033 (7)  | 0.031 (8)  | 0.072 (11) | −0.011 (6) | 0.007 (7)  | −0.007 (7)  |
| C21 | 0.034 (8)  | 0.032 (8)  | 0.075 (11) | −0.014 (6) | 0.005 (8)  | 0.000 (7)   |
| C25 | 0.034 (8)  | 0.060 (10) | 0.060 (10) | −0.018 (7) | 0.016 (7)  | −0.039 (8)  |
| C24 | 0.041 (9)  | 0.059 (11) | 0.062 (11) | −0.005 (7) | −0.001 (8) | −0.009 (9)  |
| C23 | 0.050 (11) | 0.078 (14) | 0.093 (15) | −0.031 (9) | 0.017 (10) | −0.033 (11) |
| C22 | 0.075 (12) | 0.056 (11) | 0.063 (11) | −0.023 (9) | 0.020 (10) | −0.024 (9)  |

*Geometric parameters (Å, °)*

|           |            |               |            |
|-----------|------------|---------------|------------|
| Il—C26    | 2.028 (8)  | C14—H00d      | 1.1110     |
| Fe1—N5    | 1.977 (9)  | C14—H00f      | 1.1110     |
| Fe1—N3    | 1.940 (9)  | C18—C17       | 1.396 (16) |
| Fe1—N4    | 1.988 (8)  | C18—H00I      | 1.1030     |
| Fe1—N1    | 1.958 (9)  | C16—C17       | 1.394 (17) |
| Fe1—N2    | 1.926 (10) | C16—C15       | 1.373 (16) |
| Fe1—N6    | 1.946 (9)  | C16—H00K      | 1.1030     |
| S1—C1     | 1.627 (12) | C6—C5         | 1.356 (17) |
| S2—C2     | 1.628 (11) | C6—H00L       | 1.1030     |
| N5—C9     | 1.363 (14) | C4—C5         | 1.439 (17) |
| N5—C13    | 1.329 (14) | C4—H00M       | 1.1030     |
| N3—C7     | 1.338 (14) | C17—H00N      | 1.1030     |
| N3—C3     | 1.332 (15) | C15—H00O      | 1.1030     |
| N4—C8     | 1.467 (14) | C20—H00g      | 1.1110     |
| N4—C14    | 1.488 (13) | C20—H00h      | 1.1110     |
| N4—C20    | 1.472 (13) | C11—C10       | 1.387 (17) |
| N1—C1     | 1.162 (14) | C11—C12       | 1.370 (16) |
| N2—C2     | 1.148 (14) | C11—H00R      | 1.1030     |
| N6—C19    | 1.345 (14) | C10—H00S      | 1.1030     |
| N6—C15    | 1.354 (14) | C12—H00T      | 1.1030     |
| C8—C7     | 1.490 (16) | C5—H00U       | 1.1030     |
| C8—H00a   | 1.1110     | C26—C21       | 1.3900     |
| C8—H00b   | 1.1110     | C26—C25       | 1.3900     |
| C9—C10    | 1.381 (16) | C21—C22       | 1.3900     |
| C9—H00C   | 1.1030     | C21—H00W      | 1.1030     |
| C7—C6     | 1.402 (15) | C25—C24       | 1.3900     |
| C3—C4     | 1.386 (16) | C25—H00X      | 1.1030     |
| C3—H00E   | 1.1030     | C24—C23       | 1.3900     |
| C19—C18   | 1.395 (16) | C24—H00Y      | 1.1030     |
| C19—C20   | 1.483 (14) | C23—C22       | 1.3900     |
| C13—C14   | 1.487 (15) | C23—H00Z      | 1.1030     |
| C13—C12   | 1.378 (15) | C22—H010      | 1.1030     |
| N3—Fe1—N5 | 91.7 (4)   | H00f—C14—H00d | 108.2      |
| N4—Fe1—N5 | 83.7 (4)   | C17—C18—C19   | 119.4 (11) |
| N4—Fe1—N3 | 85.7 (4)   | H00I—C18—C19  | 120.3 (6)  |
| N1—Fe1—N5 | 90.1 (4)   | H00I—C18—C17  | 120.3 (7)  |
| N1—Fe1—N3 | 177.4 (4)  | N2—C2—S2      | 178.5 (10) |
| N1—Fe1—N4 | 92.8 (4)   | C15—C16—C17   | 119.0 (10) |

|              |            |               |              |
|--------------|------------|---------------|--------------|
| N2—Fe1—N5    | 95.7 (4)   | H00K—C16—C17  | 120.5 (7)    |
| N2—Fe1—N3    | 94.0 (4)   | H00K—C16—C15  | 120.5 (7)    |
| N2—Fe1—N4    | 179.3 (4)  | C5—C6—C7      | 120.1 (11)   |
| N2—Fe1—N1    | 87.5 (4)   | H00L—C6—C7    | 120.0 (7)    |
| N6—Fe1—N5    | 166.4 (4)  | H00L—C6—C5    | 120.0 (7)    |
| N6—Fe1—N3    | 88.7 (4)   | C5—C4—C3      | 116.4 (11)   |
| N6—Fe1—N4    | 82.8 (4)   | H00M—C4—C3    | 121.8 (7)    |
| N6—Fe1—N1    | 89.0 (4)   | H00M—C4—C5    | 121.8 (7)    |
| N6—Fe1—N2    | 97.8 (4)   | C16—C17—C18   | 118.5 (11)   |
| C9—N5—Fe1    | 127.2 (7)  | H00N—C17—C18  | 120.8 (7)    |
| C13—N5—Fe1   | 113.5 (7)  | H00N—C17—C16  | 120.8 (7)    |
| C13—N5—C9    | 119.3 (9)  | C16—C15—N6    | 122.7 (11)   |
| C7—N3—Fe1    | 114.9 (7)  | H00O—C15—N6   | 118.6 (7)    |
| C3—N3—Fe1    | 126.8 (8)  | H00O—C15—C16  | 118.6 (7)    |
| C3—N3—C7     | 118.3 (10) | N1—C1—S1      | 179.1 (10)   |
| C8—N4—Fe1    | 109.6 (7)  | C19—C20—N4    | 107.5 (8)    |
| C14—N4—Fe1   | 105.9 (6)  | H00g—C20—N4   | 110.2 (5)    |
| C14—N4—C8    | 111.9 (8)  | H00g—C20—C19  | 110.2 (6)    |
| C20—N4—Fe1   | 106.1 (6)  | H00h—C20—N4   | 110.2 (5)    |
| C20—N4—C8    | 110.9 (8)  | H00h—C20—C19  | 110.2 (6)    |
| C20—N4—C14   | 112.1 (8)  | H00h—C20—H00g | 108.5024 (1) |
| C1—N1—Fe1    | 165.1 (10) | C12—C11—C10   | 119.4 (11)   |
| C2—N2—Fe1    | 169.1 (9)  | H00R—C11—C10  | 120.3 (7)    |
| C19—N6—Fe1   | 113.0 (7)  | H00R—C11—C12  | 120.3 (7)    |
| C15—N6—Fe1   | 128.0 (8)  | C11—C10—C9    | 119.1 (11)   |
| C15—N6—C19   | 118.9 (10) | H00S—C10—C9   | 120.5 (7)    |
| C7—C8—N4     | 113.8 (9)  | H00S—C10—C11  | 120.5 (7)    |
| H00a—C8—N4   | 108.8 (5)  | C11—C12—C13   | 119.1 (11)   |
| H00a—C8—C7   | 108.8 (6)  | H00T—C12—C13  | 120.5 (7)    |
| H00b—C8—N4   | 108.8 (5)  | H00T—C12—C11  | 120.5 (7)    |
| H00b—C8—C7   | 108.8 (6)  | C4—C5—C6      | 118.8 (11)   |
| H00b—C8—H00a | 107.7      | H00U—C5—C6    | 120.6 (7)    |
| C10—C9—N5    | 120.8 (10) | H00U—C5—C4    | 120.6 (7)    |
| H00C—C9—N5   | 119.6 (6)  | C21—C26—I1    | 118.9        |
| H00C—C9—C10  | 119.6 (7)  | C25—C26—I1    | 121.0        |
| C8—C7—N3     | 116.0 (9)  | C25—C26—C21   | 120.0        |
| C6—C7—N3     | 121.9 (11) | C22—C21—C26   | 120.0        |
| C6—C7—C8     | 122.1 (10) | H00W—C21—C26  | 120.0 (4)    |
| C4—C3—N3     | 124.5 (11) | H00W—C21—C22  | 120.0 (4)    |
| H00E—C3—N3   | 117.8 (7)  | C24—C25—C26   | 120.0        |
| H00E—C3—C4   | 117.8 (7)  | H00X—C25—C26  | 120.0 (4)    |
| C18—C19—N6   | 121.4 (10) | H00X—C25—C24  | 120.0 (4)    |
| C20—C19—N6   | 115.4 (9)  | C23—C24—C25   | 120.0        |
| C20—C19—C18  | 123.1 (10) | H00Y—C24—C25  | 120.0 (4)    |
| C14—C13—N5   | 115.0 (9)  | H00Y—C24—C23  | 120.0 (4)    |
| C12—C13—N5   | 122.3 (11) | C22—C23—C24   | 120.0        |
| C12—C13—C14  | 122.7 (10) | H00Z—C23—C24  | 120.0 (4)    |
| C13—C14—N4   | 109.9 (9)  | H00Z—C23—C22  | 120.0 (4)    |

|                |            |                 |             |
|----------------|------------|-----------------|-------------|
| H00d—C14—N4    | 109.7 (6)  | C23—C22—C21     | 120.0       |
| H00d—C14—C13   | 109.7 (6)  | H010—C22—C21    | 120.0 (4)   |
| H00f—C14—N4    | 109.7 (5)  | H010—C22—C23    | 120.0 (4)   |
| H00f—C14—C13   | 109.7 (6)  |                 |             |
| I1—C26—C21—C22 | −176.9 (2) | C8—C7—N3—C3     | 179.4 (10)  |
| I1—C26—C25—C24 | 176.8 (2)  | C8—C7—C6—C5     | 179.8 (11)  |
| Fe1—N5—C9—C10  | −177.8 (9) | C9—N5—C13—C14   | 175.0 (10)  |
| Fe1—N5—C13—C14 | −5.9 (8)   | C9—N5—C13—C12   | −2.3 (12)   |
| Fe1—N5—C13—C12 | 176.8 (7)  | C9—C10—C11—C12  | 0.5 (13)    |
| Fe1—N3—C7—C8   | 0.5 (8)    | C7—N3—C3—C4     | 3.9 (12)    |
| Fe1—N3—C7—C6   | 177.7 (7)  | C7—C8—N4—C14    | −115.0 (9)  |
| Fe1—N3—C3—C4   | −177.4 (9) | C7—C8—N4—C20    | 119.0 (9)   |
| Fe1—N4—C8—C7   | 2.2 (7)    | C7—C6—C5—C4     | −2.3 (13)   |
| Fe1—N4—C14—C13 | −36.7 (7)  | C3—N3—C7—C6     | −3.4 (12)   |
| Fe1—N4—C20—C19 | 40.8 (7)   | C3—C4—C5—C6     | 2.5 (12)    |
| Fe1—N6—C19—C18 | −178.4 (7) | C19—N6—C15—C16  | 1.5 (12)    |
| Fe1—N6—C19—C20 | 3.8 (8)    | C19—C18—C17—C16 | 0.4 (13)    |
| Fe1—N6—C15—C16 | 179.0 (9)  | C19—C20—N4—C14  | 156.0 (9)   |
| N5—C9—C10—C11  | −0.2 (13)  | C13—N5—C9—C10   | 1.2 (12)    |
| N5—C13—C14—N4  | 29.1 (10)  | C13—C14—N4—C20  | −151.9 (9)  |
| N5—C13—C12—C11 | 2.5 (13)   | C13—C12—C11—C10 | −1.6 (13)   |
| N3—C7—C8—N4    | −1.9 (10)  | C14—C13—C12—C11 | −174.6 (11) |
| N3—C7—C6—C5    | 2.7 (12)   | C18—C19—N6—C15  | −0.6 (13)   |
| N3—C3—C4—C5    | −3.4 (13)  | C18—C17—C16—C15 | 0.5 (13)    |
| N4—C8—C7—C6    | −179.1 (9) | C17—C18—C19—C20 | 177.3 (10)  |
| N4—C14—C13—C12 | −153.6 (9) | C15—N6—C19—C20  | −178.4 (10) |
| N4—C20—C19—N6  | −30.4 (10) | C26—C21—C22—C23 | −0.0 (7)    |
| N4—C20—C19—C18 | 151.9 (8)  | C26—C25—C24—C23 | −0.0 (7)    |
| N6—C19—C18—C17 | −0.3 (13)  | C21—C26—C25—C24 | 0.0 (7)     |
| N6—C15—C16—C17 | −1.5 (13)  | C21—C22—C23—C24 | 0.0 (7)     |
| C8—N4—C14—C13  | 82.7 (9)   | C25—C26—C21—C22 | −0.0 (7)    |
| C8—N4—C20—C19  | −78.1 (9)  | C25—C24—C23—C22 | −0.0 (7)    |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) iodobenzene monosolvate (I\_135K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 14.1597$  (14) Å

$b = 12.8265$  (9) Å

$c = 15.2977$  (13) Å

$\beta = 109.102$  (10)°

$V = 2625.4$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.686$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4965 reflections

$\theta = 2.1$ – $24.2^\circ$

$\mu = 1.94$  mm<sup>−1</sup>

$T = 135$  K

Plate, red

$0.17 \times 0.1 \times 0.03$  mm



*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector  
Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source  
Mirror monochromator  
Detector resolution: 10.0000 pixels mm<sup>-1</sup>  
 $\omega$  scans  
Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.680$ ,  $T_{\max} = 0.989$   
33749 measured reflections  
4638 independent reflections  
2955 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.160$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.1^\circ$   
 $h = -16 \rightarrow 16$   
 $k = -15 \rightarrow 15$   
 $l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.119$   
 $wR(F^2) = 0.318$   
 $S = 1.80$   
4638 reflections  
313 parameters  
0 restraints

44 constraints  
Primary atom site location: dual  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = -0.0004$   
 $\Delta\rho_{\max} = 3.20 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -2.58 \text{ e } \text{\AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Il  | 0.63211 (11) | 0.03540 (11) | 0.56351 (10) | 0.0755 (6)                       |
| Fe1 | 0.66316 (13) | 0.34042 (13) | 0.32132 (11) | 0.0203 (5)                       |
| S2  | 0.4767 (3)   | 0.0539 (3)   | 0.1685 (3)   | 0.0378 (9)                       |
| S1  | 0.7444 (3)   | 0.3035 (3)   | 0.0481 (2)   | 0.0387 (9)                       |
| N5  | 0.7728 (7)   | 0.2462 (8)   | 0.3934 (6)   | 0.022 (2)                        |
| N3  | 0.6105 (8)   | 0.3497 (7)   | 0.4229 (7)   | 0.027 (2)                        |
| N4  | 0.7560 (7)   | 0.4538 (8)   | 0.3862 (6)   | 0.022 (2)                        |
| N1  | 0.7161 (8)   | 0.3376 (8)   | 0.2186 (7)   | 0.029 (2)                        |
| N6  | 0.5790 (7)   | 0.4573 (8)   | 0.2634 (6)   | 0.028 (3)                        |
| N2  | 0.5734 (8)   | 0.2307 (8)   | 0.2608 (7)   | 0.028 (3)                        |
| C8  | 0.7349 (9)   | 0.4835 (9)   | 0.4704 (8)   | 0.025 (3)                        |
| C19 | 0.6291 (8)   | 0.5479 (9)   | 0.2730 (8)   | 0.022 (3)                        |
| C7  | 0.6544 (9)   | 0.4220 (9)   | 0.4881 (8)   | 0.024 (3)                        |
| C9  | 0.7667 (10)  | 0.1421 (10)  | 0.4060 (8)   | 0.028 (3)                        |
| C13 | 0.8579 (9)   | 0.2969 (9)   | 0.4318 (7)   | 0.022 (3)                        |
| C3  | 0.5381 (10)  | 0.2926 (11)  | 0.4358 (9)   | 0.032 (3)                        |
| C18 | 0.5819 (9)   | 0.6416 (10)  | 0.2385 (8)   | 0.029 (3)                        |
| C16 | 0.4271 (9)   | 0.5480 (11)  | 0.1826 (8)   | 0.030 (3)                        |
| C2  | 0.5328 (9)   | 0.1577 (9)   | 0.2216 (8)   | 0.023 (3)                        |
| C14 | 0.8593 (8)   | 0.4079 (10)  | 0.4072 (9)   | 0.027 (3)                        |
| C15 | 0.4788 (9)   | 0.4577 (11)  | 0.2196 (8)   | 0.029 (3)                        |
| C1  | 0.7271 (10)  | 0.3252 (9)   | 0.1468 (9)   | 0.027 (3)                        |
| C6  | 0.6224 (10)  | 0.4414 (11)  | 0.5629 (8)   | 0.032 (3)                        |
| C20 | 0.7377 (8)   | 0.5400 (9)   | 0.3202 (8)   | 0.022 (3)                        |
| C4  | 0.5019 (11)  | 0.3054 (10)  | 0.5090 (9)   | 0.036 (3)                        |
| C11 | 0.9371 (10)  | 0.1412 (10)  | 0.5021 (8)   | 0.031 (3)                        |
| C12 | 0.9424 (9)   | 0.2454 (10)  | 0.4891 (8)   | 0.025 (3)                        |

|      |             |             |             |            |
|------|-------------|-------------|-------------|------------|
| C17  | 0.4782 (11) | 0.6410 (12) | 0.1928 (9)  | 0.039 (4)  |
| C21  | 0.6818 (7)  | 0.1170 (8)  | 0.7544 (9)  | 0.053 (5)  |
| C5   | 0.5477 (11) | 0.3842 (12) | 0.5760 (9)  | 0.037 (3)  |
| C26  | 0.6989 (8)  | 0.1321 (8)  | 0.6709 (7)  | 0.056 (5)  |
| C10  | 0.8490 (11) | 0.0881 (11) | 0.4611 (9)  | 0.038 (4)  |
| C25  | 0.7568 (8)  | 0.2158 (9)  | 0.6604 (7)  | 0.056 (5)  |
| C24  | 0.7975 (7)  | 0.2842 (8)  | 0.7335 (10) | 0.065 (5)  |
| C23  | 0.7804 (9)  | 0.2690 (9)  | 0.8171 (8)  | 0.072 (6)  |
| C22  | 0.7226 (9)  | 0.1854 (10) | 0.8275 (7)  | 0.077 (6)  |
| H00X | 0.7703 (8)  | 0.2278 (9)  | 0.5941 (7)  | 0.067 (6)* |
| H00Y | 0.8434 (7)  | 0.3506 (8)  | 0.7253 (10) | 0.078 (6)* |
| H00Z | 0.8127 (9)  | 0.3234 (9)  | 0.8751 (8)  | 0.086 (7)* |
| H010 | 0.7090 (9)  | 0.1734 (10) | 0.8938 (7)  | 0.092 (7)* |
| H00T | 0.6359 (7)  | 0.0506 (8)  | 0.7627 (9)  | 0.063 (5)* |
| H00E | 0.6960 (10) | 0.1005 (10) | 0.3721 (8)  | 0.034 (4)* |
| H00R | 1.0121 (9)  | 0.2885 (10) | 0.5232 (8)  | 0.030 (3)* |
| H00Q | 1.0036 (10) | 0.0988 (10) | 0.5456 (8)  | 0.037 (4)* |
| H00W | 0.8443 (11) | 0.0036 (11) | 0.4722 (9)  | 0.045 (4)* |
| H00G | 0.5045 (10) | 0.2309 (11) | 0.3849 (9)  | 0.039 (4)* |
| H00N | 0.6580 (10) | 0.5039 (11) | 0.6123 (8)  | 0.038 (4)* |
| H00U | 0.5228 (11) | 0.3978 (12) | 0.6363 (9)  | 0.045 (4)* |
| H00P | 0.4402 (11) | 0.2568 (10) | 0.5155 (9)  | 0.043 (4)* |
| H00L | 0.4371 (9)  | 0.3839 (11) | 0.2132 (8)  | 0.035 (4)* |
| H00I | 0.3460 (9)  | 0.5453 (11) | 0.1453 (8)  | 0.036 (4)* |
| H00S | 0.4382 (11) | 0.7140 (12) | 0.1655 (9)  | 0.047 (4)* |
| H00H | 0.6250 (9)  | 0.7147 (10) | 0.2468 (8)  | 0.035 (4)* |
| H00a | 0.8044 (9)  | 0.4746 (9)  | 0.5305 (8)  | 0.030 (3)* |
| H00b | 0.7137 (9)  | 0.5672 (9)  | 0.4654 (8)  | 0.030 (3)* |
| H00c | 0.8841 (8)  | 0.4157 (10) | 0.3457 (9)  | 0.032 (4)* |
| H00d | 0.9129 (8)  | 0.4509 (10) | 0.4655 (9)  | 0.032 (4)* |
| H00f | 0.7667 (8)  | 0.6140 (9)  | 0.3570 (8)  | 0.027 (3)* |
| H00j | 0.7764 (8)  | 0.5254 (9)  | 0.2689 (8)  | 0.027 (3)* |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| I1  | 0.0840 (11) | 0.0606 (9)  | 0.0649 (9)  | −0.0004 (7)  | 0.0012 (7)  | 0.0016 (6)   |
| Fe1 | 0.0203 (9)  | 0.0211 (9)  | 0.0142 (8)  | −0.0018 (7)  | −0.0017 (7) | −0.0003 (7)  |
| S2  | 0.0335 (19) | 0.0329 (19) | 0.041 (2)   | −0.0083 (15) | 0.0042 (16) | −0.0156 (16) |
| S1  | 0.058 (2)   | 0.036 (2)   | 0.0250 (17) | 0.0105 (17)  | 0.0172 (17) | 0.0061 (15)  |
| N5  | 0.022 (6)   | 0.023 (6)   | 0.018 (5)   | −0.001 (4)   | 0.001 (4)   | 0.008 (4)    |
| N3  | 0.032 (6)   | 0.015 (5)   | 0.034 (6)   | −0.000 (4)   | 0.012 (5)   | −0.001 (4)   |
| N4  | 0.015 (5)   | 0.024 (6)   | 0.021 (5)   | −0.003 (4)   | −0.003 (4)  | −0.001 (4)   |
| N1  | 0.043 (7)   | 0.019 (5)   | 0.027 (6)   | 0.002 (5)    | 0.017 (5)   | 0.005 (5)    |
| N6  | 0.025 (6)   | 0.040 (7)   | 0.010 (5)   | 0.005 (5)    | −0.005 (4)  | −0.001 (4)   |
| N2  | 0.021 (6)   | 0.030 (6)   | 0.025 (5)   | −0.006 (5)   | −0.001 (5)  | 0.002 (5)    |
| C8  | 0.030 (7)   | 0.020 (6)   | 0.022 (6)   | −0.002 (5)   | 0.002 (5)   | 0.005 (5)    |
| C19 | 0.017 (6)   | 0.021 (6)   | 0.020 (6)   | 0.001 (5)    | −0.005 (5)  | 0.003 (5)    |

|     |            |            |            |            |            |            |
|-----|------------|------------|------------|------------|------------|------------|
| C7  | 0.030 (7)  | 0.017 (6)  | 0.017 (6)  | −0.001 (5) | −0.001 (5) | 0.002 (5)  |
| C9  | 0.032 (7)  | 0.029 (7)  | 0.023 (6)  | −0.012 (6) | 0.007 (6)  | −0.008 (5) |
| C13 | 0.020 (6)  | 0.022 (6)  | 0.014 (5)  | 0.002 (5)  | −0.006 (5) | −0.004 (5) |
| C3  | 0.030 (7)  | 0.039 (8)  | 0.020 (6)  | 0.002 (6)  | −0.002 (6) | 0.006 (6)  |
| C18 | 0.024 (7)  | 0.030 (7)  | 0.027 (6)  | −0.002 (5) | 0.001 (5)  | 0.004 (5)  |
| C16 | 0.020 (7)  | 0.044 (8)  | 0.021 (6)  | 0.007 (6)  | −0.000 (5) | 0.003 (6)  |
| C2  | 0.021 (6)  | 0.020 (7)  | 0.019 (6)  | −0.000 (5) | −0.004 (5) | −0.002 (5) |
| C14 | 0.015 (6)  | 0.031 (7)  | 0.024 (6)  | 0.001 (5)  | −0.007 (5) | 0.006 (5)  |
| C15 | 0.019 (6)  | 0.043 (8)  | 0.020 (6)  | −0.003 (6) | −0.002 (5) | −0.011 (6) |
| C1  | 0.034 (7)  | 0.027 (7)  | 0.028 (7)  | 0.011 (5)  | 0.019 (6)  | 0.011 (6)  |
| C6  | 0.028 (7)  | 0.044 (8)  | 0.021 (6)  | 0.003 (6)  | 0.004 (6)  | −0.006 (6) |
| C20 | 0.019 (6)  | 0.018 (6)  | 0.027 (6)  | 0.002 (5)  | 0.004 (5)  | 0.002 (5)  |
| C4  | 0.038 (8)  | 0.031 (7)  | 0.034 (8)  | −0.002 (6) | 0.005 (6)  | 0.000 (6)  |
| C11 | 0.032 (7)  | 0.030 (8)  | 0.023 (6)  | 0.001 (6)  | 0.000 (6)  | −0.000 (5) |
| C12 | 0.018 (6)  | 0.028 (7)  | 0.020 (6)  | 0.005 (5)  | −0.005 (5) | 0.004 (5)  |
| C17 | 0.039 (8)  | 0.043 (9)  | 0.028 (7)  | 0.007 (7)  | 0.003 (6)  | −0.003 (6) |
| C21 | 0.035 (8)  | 0.039 (9)  | 0.076 (12) | 0.022 (7)  | 0.006 (9)  | 0.009 (9)  |
| C5  | 0.040 (8)  | 0.051 (9)  | 0.019 (7)  | 0.009 (7)  | 0.006 (6)  | 0.007 (6)  |
| C26 | 0.038 (9)  | 0.040 (9)  | 0.085 (14) | 0.008 (7)  | 0.012 (9)  | 0.007 (9)  |
| C10 | 0.049 (9)  | 0.027 (8)  | 0.029 (7)  | 0.002 (6)  | 0.001 (7)  | 0.006 (6)  |
| C25 | 0.049 (10) | 0.053 (11) | 0.064 (11) | 0.017 (8)  | 0.017 (9)  | 0.038 (9)  |
| C24 | 0.037 (10) | 0.076 (14) | 0.072 (13) | 0.008 (9)  | 0.004 (10) | 0.013 (11) |
| C23 | 0.057 (12) | 0.054 (12) | 0.097 (16) | 0.026 (9)  | 0.017 (11) | 0.019 (11) |
| C22 | 0.084 (15) | 0.075 (15) | 0.064 (13) | 0.027 (12) | 0.015 (11) | 0.018 (11) |

*Geometric parameters (Å, °)*

|        |            |          |            |
|--------|------------|----------|------------|
| I1—C26 | 2.029 (9)  | C18—C17  | 1.404 (18) |
| Fe1—N5 | 1.991 (9)  | C18—H00H | 1.1030     |
| Fe1—N3 | 1.934 (11) | C16—C15  | 1.388 (18) |
| Fe1—N4 | 1.993 (9)  | C16—C17  | 1.377 (19) |
| Fe1—N1 | 1.950 (11) | C16—H00I | 1.1030     |
| Fe1—N6 | 1.940 (10) | C14—H00c | 1.1110     |
| Fe1—N2 | 1.919 (10) | C14—H00d | 1.1110     |
| S2—C2  | 1.625 (12) | C15—H00L | 1.1030     |
| S1—C1  | 1.632 (14) | C6—C5    | 1.36 (2)   |
| N5—C9  | 1.356 (15) | C6—H00N  | 1.1030     |
| N5—C13 | 1.325 (15) | C20—H00f | 1.1110     |
| N3—C7  | 1.355 (15) | C20—H00j | 1.1110     |
| N3—C3  | 1.326 (17) | C4—C5    | 1.435 (19) |
| N4—C8  | 1.464 (16) | C4—H00P  | 1.1030     |
| N4—C14 | 1.511 (15) | C11—C12  | 1.356 (17) |
| N4—C20 | 1.463 (14) | C11—C10  | 1.380 (18) |
| N1—C1  | 1.170 (16) | C11—H00Q | 1.1030     |
| N6—C19 | 1.345 (15) | C12—H00R | 1.1030     |
| N6—C15 | 1.357 (15) | C17—H00S | 1.1030     |
| N2—C2  | 1.158 (15) | C21—C26  | 1.3900     |
| C8—C7  | 1.482 (18) | C21—C22  | 1.3900     |

|            |            |               |            |
|------------|------------|---------------|------------|
| C8—H00a    | 1.1110     | C21—H00T      | 1.1030     |
| C8—H00b    | 1.1110     | C5—H00U       | 1.1030     |
| C19—C18    | 1.392 (16) | C26—C25       | 1.3900     |
| C19—C20    | 1.472 (15) | C10—H00W      | 1.1030     |
| C7—C6      | 1.385 (18) | C25—C24       | 1.3900     |
| C9—C10     | 1.380 (18) | C25—H00X      | 1.1030     |
| C9—H00E    | 1.1030     | C24—C23       | 1.3900     |
| C13—C14    | 1.474 (17) | C24—H00Y      | 1.1030     |
| C13—C12    | 1.397 (15) | C23—C22       | 1.3900     |
| C3—C4      | 1.386 (19) | C23—H00Z      | 1.1030     |
| C3—H00G    | 1.1030     | C22—H010      | 1.1030     |
| N3—Fe1—N5  | 91.6 (4)   | H00I—C16—C17  | 120.4 (8)  |
| N4—Fe1—N5  | 84.2 (4)   | N2—C2—S2      | 178.9 (11) |
| N4—Fe1—N3  | 85.4 (4)   | C13—C14—N4    | 109.7 (10) |
| N1—Fe1—N5  | 90.0 (4)   | H00c—C14—N4   | 109.7 (6)  |
| N1—Fe1—N3  | 177.5 (4)  | H00c—C14—C13  | 109.7 (7)  |
| N1—Fe1—N4  | 92.9 (4)   | H00d—C14—N4   | 109.7 (6)  |
| N6—Fe1—N5  | 166.7 (4)  | H00d—C14—C13  | 109.7 (6)  |
| N6—Fe1—N3  | 88.7 (4)   | H00d—C14—H00c | 108.2      |
| N6—Fe1—N4  | 82.5 (4)   | C16—C15—N6    | 122.1 (12) |
| N6—Fe1—N1  | 89.3 (4)   | H00L—C15—N6   | 118.9 (8)  |
| N2—Fe1—N5  | 95.5 (4)   | H00L—C15—C16  | 118.9 (8)  |
| N2—Fe1—N3  | 93.7 (5)   | N1—C1—S1      | 177.7 (11) |
| N2—Fe1—N4  | 179.0 (5)  | C5—C6—C7      | 120.4 (13) |
| N2—Fe1—N1  | 88.1 (5)   | H00N—C6—C7    | 119.8 (8)  |
| N2—Fe1—N6  | 97.8 (4)   | H00N—C6—C5    | 119.8 (9)  |
| C9—N5—Fe1  | 127.0 (8)  | C19—C20—N4    | 108.1 (10) |
| C13—N5—Fe1 | 112.3 (8)  | H00f—C20—N4   | 110.1 (6)  |
| C13—N5—C9  | 120.8 (10) | H00f—C20—C19  | 110.1 (6)  |
| C7—N3—Fe1  | 115.4 (9)  | H00j—C20—N4   | 110.1 (6)  |
| C3—N3—Fe1  | 127.2 (9)  | H00j—C20—C19  | 110.1 (7)  |
| C3—N3—C7   | 117.3 (12) | H00j—C20—H00f | 108.4      |
| C8—N4—Fe1  | 109.7 (7)  | C5—C4—C3      | 117.4 (13) |
| C14—N4—Fe1 | 104.9 (7)  | H00P—C4—C3    | 121.3 (8)  |
| C14—N4—C8  | 112.1 (9)  | H00P—C4—C5    | 121.3 (9)  |
| C20—N4—Fe1 | 105.6 (7)  | C10—C11—C12   | 120.3 (12) |
| C20—N4—C8  | 111.7 (9)  | H00Q—C11—C12  | 119.8 (7)  |
| C20—N4—C14 | 112.4 (9)  | H00Q—C11—C10  | 119.8 (8)  |
| C1—N1—Fe1  | 164.5 (11) | C11—C12—C13   | 118.8 (11) |
| C19—N6—Fe1 | 113.3 (8)  | H00R—C12—C13  | 120.6 (7)  |
| C15—N6—Fe1 | 128.0 (9)  | H00R—C12—C11  | 120.6 (7)  |
| C15—N6—C19 | 118.7 (11) | C16—C17—C18   | 119.1 (13) |
| C2—N2—Fe1  | 168.8 (11) | H00S—C17—C18  | 120.5 (8)  |
| C7—C8—N4   | 114.4 (10) | H00S—C17—C16  | 120.5 (8)  |
| H00a—C8—N4 | 108.7 (6)  | C22—C21—C26   | 120.0      |
| H00a—C8—C7 | 108.7 (6)  | H00T—C21—C26  | 120.0 (4)  |
| H00b—C8—N4 | 108.7 (6)  | H00T—C21—C22  | 120.0 (4)  |

|                |             |                 |              |
|----------------|-------------|-----------------|--------------|
| H00b—C8—C7     | 108.7 (7)   | C4—C5—C6        | 118.0 (14)   |
| H00b—C8—H00a   | 107.6       | H00U—C5—C6      | 121.0 (8)    |
| C18—C19—N6     | 122.2 (10)  | H00U—C5—C4      | 121.0 (9)    |
| C20—C19—N6     | 115.0 (10)  | C21—C26—I1      | 118.9        |
| C20—C19—C18    | 122.7 (11)  | C25—C26—I1      | 121.0        |
| C8—C7—N3       | 115.1 (11)  | C25—C26—C21     | 120.0        |
| C6—C7—N3       | 122.5 (12)  | C11—C10—C9      | 119.2 (13)   |
| C6—C7—C8       | 122.3 (11)  | H00W—C10—C9     | 120.4 (8)    |
| C10—C9—N5      | 120.0 (11)  | H00W—C10—C11    | 120.4 (8)    |
| H00E—C9—N5     | 120.0 (7)   | C24—C25—C26     | 120.0000 (1) |
| H00E—C9—C10    | 120.0 (8)   | H00X—C25—C26    | 120.0 (4)    |
| C14—C13—N5     | 116.5 (10)  | H00X—C25—C24    | 120.0 (4)    |
| C12—C13—N5     | 120.9 (11)  | C23—C24—C25     | 120.0        |
| C12—C13—C14    | 122.5 (11)  | H00Y—C24—C25    | 120.0 (4)    |
| C4—C3—N3       | 124.3 (13)  | H00Y—C24—C23    | 120.0 (4)    |
| H00G—C3—N3     | 117.8 (8)   | C22—C23—C24     | 120.0        |
| H00G—C3—C4     | 117.8 (8)   | H00Z—C23—C24    | 120.0 (4)    |
| C17—C18—C19    | 118.6 (12)  | H00Z—C23—C22    | 120.0 (4)    |
| H00H—C18—C19   | 120.7 (7)   | C23—C22—C21     | 120.0        |
| H00H—C18—C17   | 120.7 (8)   | H010—C22—C21    | 120.0 (4)    |
| C17—C16—C15    | 119.2 (12)  | H010—C22—C23    | 120.0 (4)    |
| H00I—C16—C15   | 120.4 (8)   |                 |              |
| I1—C26—C21—C22 | −176.7 (3)  | C8—C7—N3—C3     | 179.9 (11)   |
| I1—C26—C25—C24 | 176.6 (3)   | C8—C7—C6—C5     | 179.1 (12)   |
| Fe1—N5—C9—C10  | −177.5 (11) | C19—N6—C15—C16  | 1.6 (13)     |
| Fe1—N5—C13—C14 | −6.0 (10)   | C19—C18—C17—C16 | −0.9 (14)    |
| Fe1—N5—C13—C12 | 176.7 (8)   | C19—C20—N4—C14  | 154.9 (9)    |
| Fe1—N3—C7—C8   | 0.9 (9)     | C7—N3—C3—C4     | 3.0 (13)     |
| Fe1—N3—C7—C6   | 177.1 (8)   | C7—C8—N4—C14    | −114.3 (10)  |
| Fe1—N3—C3—C4   | −178.1 (10) | C7—C8—N4—C20    | 118.5 (10)   |
| Fe1—N4—C8—C7   | 1.8 (8)     | C7—C6—C5—C4     | −1.5 (14)    |
| Fe1—N4—C14—C13 | −37.1 (7)   | C9—N5—C13—C14   | 174.6 (12)   |
| Fe1—N4—C20—C19 | 41.1 (8)    | C9—N5—C13—C12   | −2.6 (14)    |
| Fe1—N6—C19—C18 | −178.4 (8)  | C9—C10—C11—C12  | 0.7 (16)     |
| Fe1—N6—C19—C20 | 3.4 (9)     | C13—N5—C9—C10   | 1.7 (14)     |
| Fe1—N6—C15—C16 | 178.7 (10)  | C13—C14—N4—C20  | −151.4 (9)   |
| N5—C9—C10—C11  | −0.7 (15)   | C13—C12—C11—C10 | −1.6 (15)    |
| N5—C13—C14—N4  | 29.8 (12)   | C3—N3—C7—C6     | −3.9 (13)    |
| N5—C13—C12—C11 | 2.6 (14)    | C3—C4—C5—C6     | 0.6 (14)     |
| N3—C7—C8—N4    | −1.8 (11)   | C18—C19—N6—C15  | −0.9 (15)    |
| N3—C7—C6—C5    | 3.2 (14)    | C18—C17—C16—C15 | 1.6 (16)     |
| N3—C3—C4—C5    | −1.4 (15)   | C14—C13—C12—C11 | −174.5 (13)  |
| N4—C8—C7—C6    | −178.0 (10) | C15—N6—C19—C20  | −179.1 (11)  |
| N4—C14—C13—C12 | −153.0 (9)  | C20—C19—C18—C17 | 178.7 (13)   |
| N4—C20—C19—N6  | −30.5 (11)  | C21—C26—C25—C24 | −0.0 (8)     |
| N4—C20—C19—C18 | 151.3 (10)  | C21—C22—C23—C24 | 0.0 (8)      |
| N6—C19—C18—C17 | 0.6 (14)    | C26—C21—C22—C23 | −0.0 (8)     |

|                |            |                 |         |
|----------------|------------|-----------------|---------|
| N6—C15—C16—C17 | −2.0 (15)  | C26—C25—C24—C23 | 0.0 (8) |
| C8—N4—C14—C13  | 81.8 (10)  | C25—C26—C21—C22 | 0.0 (8) |
| C8—N4—C20—C19  | −78.1 (10) | C25—C24—C23—C22 | 0.0 (8) |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) iodobenzene monosolvate (I\_145K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 14.175$  (2) Å

$b = 12.8369$  (13) Å

$c = 15.305$  (2) Å

$\beta = 109.157$  (16)°

$V = 2630.6$  (7) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.683$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3428 reflections

$\theta = 2.1$ – $24.0^\circ$

$\mu = 1.93$  mm<sup>−1</sup>

$T = 145$  K

Plate, red

$0.17 \times 0.1 \times 0.03$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.740$ ,  $T_{\max} = 1.000$

41127 measured reflections

4647 independent reflections

2700 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.239$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 1.7^\circ$

$h = -16 \rightarrow 16$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.106$

$wR(F^2) = 0.289$

$S = 1.49$

4647 reflections

313 parameters

0 restraints

44 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 2.23$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -2.35$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| I1  | 0.63199 (11) | 0.03501 (10) | 0.56325 (10) | 0.0773 (5)                       |
| Fe1 | 0.66320 (12) | 0.34019 (12) | 0.32132 (11) | 0.0202 (4)                       |
| S2  | 0.4772 (2)   | 0.0539 (3)   | 0.1688 (3)   | 0.0377 (8)                       |
| S1  | 0.7444 (3)   | 0.3039 (3)   | 0.0484 (2)   | 0.0409 (9)                       |
| N5  | 0.7722 (7)   | 0.2471 (7)   | 0.3926 (6)   | 0.020 (2)                        |
| N6  | 0.5783 (7)   | 0.4580 (7)   | 0.2635 (6)   | 0.025 (2)                        |
| N3  | 0.6118 (7)   | 0.3494 (7)   | 0.4235 (6)   | 0.024 (2)                        |
| N4  | 0.7566 (6)   | 0.4535 (7)   | 0.3867 (6)   | 0.025 (2)                        |
| N1  | 0.7151 (7)   | 0.3370 (7)   | 0.2165 (7)   | 0.029 (2)                        |
| N2  | 0.5739 (7)   | 0.2292 (8)   | 0.2604 (7)   | 0.030 (2)                        |
| C9  | 0.7677 (10)  | 0.1435 (8)   | 0.4060 (8)   | 0.029 (3)                        |

|      |             |             |             |            |
|------|-------------|-------------|-------------|------------|
| C19  | 0.6298 (8)  | 0.5493 (9)  | 0.2735 (7)  | 0.023 (3)  |
| C1   | 0.7276 (8)  | 0.3251 (8)  | 0.1469 (8)  | 0.024 (3)  |
| C2   | 0.5331 (9)  | 0.1578 (9)  | 0.2202 (8)  | 0.025 (3)  |
| C14  | 0.8588 (8)  | 0.4082 (8)  | 0.4078 (8)  | 0.024 (3)  |
| C3   | 0.5377 (9)  | 0.2929 (9)  | 0.4353 (8)  | 0.030 (3)  |
| C18  | 0.5819 (8)  | 0.6417 (10) | 0.2391 (8)  | 0.030 (3)  |
| C7   | 0.6533 (9)  | 0.4213 (9)  | 0.4875 (8)  | 0.024 (3)  |
| C13  | 0.8597 (9)  | 0.2967 (9)  | 0.4324 (8)  | 0.025 (3)  |
| C17  | 0.4781 (10) | 0.6410 (11) | 0.1928 (9)  | 0.037 (3)  |
| C15  | 0.4784 (8)  | 0.4585 (10) | 0.2203 (8)  | 0.030 (3)  |
| C16  | 0.4270 (8)  | 0.5478 (10) | 0.1825 (8)  | 0.030 (3)  |
| C11  | 0.9389 (9)  | 0.1410 (9)  | 0.5025 (8)  | 0.032 (3)  |
| C8   | 0.7366 (10) | 0.4823 (9)  | 0.4706 (8)  | 0.032 (3)  |
| C10  | 0.8492 (11) | 0.0872 (10) | 0.4622 (8)  | 0.039 (3)  |
| C20  | 0.7386 (8)  | 0.5403 (8)  | 0.3203 (8)  | 0.024 (3)  |
| C6   | 0.6222 (9)  | 0.4411 (9)  | 0.5636 (8)  | 0.030 (3)  |
| C4   | 0.5038 (10) | 0.3057 (10) | 0.5095 (8)  | 0.034 (3)  |
| C5   | 0.5464 (10) | 0.3844 (10) | 0.5757 (8)  | 0.035 (3)  |
| C12  | 0.9435 (9)  | 0.2454 (9)  | 0.4878 (9)  | 0.033 (3)  |
| C26  | 0.6978 (8)  | 0.1326 (7)  | 0.6694 (7)  | 0.059 (5)  |
| C21  | 0.6804 (7)  | 0.1173 (7)  | 0.7527 (9)  | 0.057 (4)  |
| C25  | 0.7560 (8)  | 0.2160 (9)  | 0.6594 (7)  | 0.064 (5)  |
| C24  | 0.7968 (7)  | 0.2840 (7)  | 0.7328 (10) | 0.067 (5)  |
| C23  | 0.7794 (8)  | 0.2688 (8)  | 0.8161 (8)  | 0.075 (6)  |
| C22  | 0.7212 (9)  | 0.1854 (9)  | 0.8261 (7)  | 0.075 (5)  |
| H00X | 0.7698 (8)  | 0.2281 (9)  | 0.5933 (7)  | 0.077 (6)* |
| H00Y | 0.8430 (7)  | 0.3502 (7)  | 0.7248 (10) | 0.080 (6)* |
| H00Z | 0.8118 (8)  | 0.3228 (8)  | 0.8743 (8)  | 0.090 (7)* |
| H010 | 0.7074 (9)  | 0.1733 (9)  | 0.8922 (7)  | 0.090 (7)* |
| H00W | 0.6342 (7)  | 0.0512 (7)  | 0.7606 (9)  | 0.068 (5)* |
| H00B | 0.6971 (10) | 0.1020 (8)  | 0.3714 (8)  | 0.035 (3)* |
| H00U | 1.0136 (9)  | 0.2885 (9)  | 0.5201 (9)  | 0.039 (4)* |
| H00N | 1.0058 (9)  | 0.0991 (9)  | 0.5460 (8)  | 0.039 (4)* |
| H00P | 0.8431 (11) | 0.0031 (10) | 0.4744 (8)  | 0.046 (4)* |
| H00L | 0.4366 (8)  | 0.3851 (10) | 0.2152 (8)  | 0.036 (3)* |
| H00M | 0.3461 (8)  | 0.5448 (10) | 0.1445 (8)  | 0.036 (3)* |
| H00K | 0.4382 (10) | 0.7138 (11) | 0.1651 (9)  | 0.045 (4)* |
| H00H | 0.6244 (8)  | 0.7151 (10) | 0.2480 (8)  | 0.036 (3)* |
| H00G | 0.5018 (9)  | 0.2332 (9)  | 0.3832 (8)  | 0.036 (3)* |
| H00R | 0.6589 (9)  | 0.5026 (9)  | 0.6136 (8)  | 0.036 (3)* |
| H00T | 0.5193 (10) | 0.3989 (10) | 0.6344 (8)  | 0.042 (4)* |
| H00S | 0.4441 (10) | 0.2551 (10) | 0.5172 (8)  | 0.040 (4)* |
| H00a | 0.8836 (8)  | 0.4167 (8)  | 0.3464 (8)  | 0.029 (3)* |
| H00c | 0.9120 (8)  | 0.4515 (8)  | 0.4661 (8)  | 0.029 (3)* |
| H00d | 0.8059 (10) | 0.4710 (9)  | 0.5304 (8)  | 0.039 (3)* |
| H00e | 0.7174 (10) | 0.5664 (9)  | 0.4670 (8)  | 0.039 (3)* |
| H00f | 0.7684 (8)  | 0.6140 (8)  | 0.3571 (8)  | 0.028 (3)* |
| H00i | 0.7767 (8)  | 0.5251 (8)  | 0.2687 (8)  | 0.028 (3)* |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| II  | 0.0903 (10) | 0.0652 (9)  | 0.0683 (9)  | 0.0017 (7)   | 0.0149 (7)  | 0.0017 (7)   |
| Fe1 | 0.0221 (8)  | 0.0221 (8)  | 0.0152 (8)  | −0.0023 (7)  | 0.0044 (7)  | 0.0003 (7)   |
| S2  | 0.0342 (19) | 0.0354 (18) | 0.042 (2)   | −0.0072 (14) | 0.0100 (16) | −0.0151 (15) |
| S1  | 0.064 (2)   | 0.0388 (18) | 0.0269 (18) | 0.0128 (17)  | 0.0242 (17) | 0.0056 (15)  |
| N5  | 0.027 (6)   | 0.018 (5)   | 0.014 (5)   | −0.000 (4)   | 0.005 (4)   | −0.000 (4)   |
| N6  | 0.030 (6)   | 0.029 (5)   | 0.013 (5)   | 0.000 (4)    | 0.005 (4)   | −0.007 (4)   |
| N3  | 0.027 (5)   | 0.026 (5)   | 0.019 (5)   | −0.001 (4)   | 0.007 (4)   | −0.006 (4)   |
| N4  | 0.015 (5)   | 0.034 (6)   | 0.021 (5)   | 0.000 (4)    | −0.002 (4)  | 0.004 (4)    |
| N1  | 0.034 (6)   | 0.029 (5)   | 0.018 (6)   | 0.004 (4)    | 0.001 (5)   | 0.004 (4)    |
| N2  | 0.030 (6)   | 0.032 (6)   | 0.026 (6)   | −0.003 (5)   | 0.005 (5)   | −0.002 (5)   |
| C9  | 0.046 (8)   | 0.017 (6)   | 0.027 (7)   | −0.014 (5)   | 0.014 (6)   | −0.002 (5)   |
| C19 | 0.023 (6)   | 0.028 (6)   | 0.014 (6)   | −0.003 (5)   | −0.001 (5)  | 0.003 (5)    |
| C1  | 0.032 (7)   | 0.027 (6)   | 0.016 (7)   | 0.010 (5)    | 0.011 (5)   | 0.008 (5)    |
| C2  | 0.031 (6)   | 0.022 (6)   | 0.017 (6)   | 0.004 (5)    | 0.003 (5)   | 0.000 (5)    |
| C14 | 0.020 (6)   | 0.022 (6)   | 0.028 (7)   | −0.002 (5)   | 0.005 (5)   | 0.009 (5)    |
| C3  | 0.027 (7)   | 0.038 (7)   | 0.021 (6)   | 0.001 (5)    | 0.003 (5)   | −0.011 (5)   |
| C18 | 0.017 (6)   | 0.044 (8)   | 0.029 (7)   | 0.009 (5)    | 0.006 (5)   | 0.001 (6)    |
| C7  | 0.033 (7)   | 0.026 (6)   | 0.017 (6)   | 0.004 (5)    | 0.011 (5)   | 0.007 (5)    |
| C13 | 0.032 (7)   | 0.026 (6)   | 0.017 (6)   | −0.003 (5)   | 0.007 (5)   | 0.004 (5)    |
| C17 | 0.033 (7)   | 0.051 (9)   | 0.028 (7)   | −0.003 (6)   | 0.011 (6)   | −0.005 (6)   |
| C15 | 0.016 (6)   | 0.049 (8)   | 0.020 (6)   | −0.009 (6)   | 0.000 (5)   | −0.013 (6)   |
| C16 | 0.014 (6)   | 0.053 (8)   | 0.013 (6)   | 0.007 (6)    | −0.008 (5)  | −0.003 (5)   |
| C11 | 0.034 (7)   | 0.030 (7)   | 0.026 (7)   | 0.013 (6)    | 0.002 (6)   | 0.006 (5)    |
| C8  | 0.043 (8)   | 0.031 (7)   | 0.022 (7)   | 0.003 (6)    | 0.009 (6)   | −0.002 (5)   |
| C10 | 0.067 (10)  | 0.026 (7)   | 0.024 (7)   | 0.008 (7)    | 0.017 (7)   | 0.012 (5)    |
| C20 | 0.018 (6)   | 0.020 (6)   | 0.033 (7)   | −0.004 (5)   | 0.007 (5)   | 0.001 (5)    |
| C6  | 0.031 (7)   | 0.039 (7)   | 0.020 (6)   | −0.007 (5)   | 0.006 (5)   | −0.005 (5)   |
| C4  | 0.040 (8)   | 0.040 (7)   | 0.023 (7)   | 0.002 (6)    | 0.013 (6)   | 0.007 (6)    |
| C5  | 0.042 (8)   | 0.047 (8)   | 0.020 (7)   | 0.004 (6)    | 0.016 (6)   | 0.007 (6)    |
| C12 | 0.023 (7)   | 0.028 (7)   | 0.037 (7)   | 0.001 (5)    | −0.004 (6)  | −0.009 (6)   |
| C26 | 0.037 (8)   | 0.037 (9)   | 0.099 (15)  | 0.005 (6)    | 0.019 (9)   | 0.014 (8)    |
| C21 | 0.052 (9)   | 0.042 (9)   | 0.079 (13)  | 0.015 (7)    | 0.024 (9)   | 0.007 (9)    |
| C25 | 0.062 (11)  | 0.053 (10)  | 0.084 (14)  | 0.008 (9)    | 0.033 (10)  | 0.010 (10)   |
| C24 | 0.037 (9)   | 0.067 (12)  | 0.089 (14)  | 0.014 (8)    | 0.011 (10)  | 0.022 (11)   |
| C23 | 0.047 (11)  | 0.076 (13)  | 0.095 (16)  | 0.011 (9)    | 0.012 (10)  | 0.021 (11)   |
| C22 | 0.088 (14)  | 0.056 (11)  | 0.077 (14)  | 0.027 (10)   | 0.020 (11)  | 0.014 (10)   |

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

|        |            |          |            |
|--------|------------|----------|------------|
| II—C26 | 2.021 (9)  | C7—C6    | 1.397 (15) |
| Fe1—N5 | 1.975 (9)  | C13—C12  | 1.379 (16) |
| Fe1—N6 | 1.954 (9)  | C17—C16  | 1.380 (17) |
| Fe1—N3 | 1.934 (9)  | C17—H00K | 1.1030     |
| Fe1—N4 | 2.001 (9)  | C15—C16  | 1.380 (16) |
| Fe1—N1 | 1.973 (10) | C15—H00L | 1.1030     |



|            |            |              |            |
|------------|------------|--------------|------------|
| Fe1—N2     | 1.930 (11) | C16—H00M     | 1.1030     |
| S2—C2      | 1.618 (12) | C11—C10      | 1.401 (18) |
| S1—C1      | 1.625 (12) | C11—C12      | 1.364 (16) |
| N5—C9      | 1.350 (14) | C11—H00N     | 1.1030     |
| N5—C13     | 1.348 (14) | C8—H00d      | 1.1110     |
| N6—C19     | 1.362 (14) | C8—H00e      | 1.1110     |
| N6—C15     | 1.352 (14) | C10—H00P     | 1.1030     |
| N3—C3      | 1.336 (15) | C20—H00f     | 1.1110     |
| N3—C7      | 1.334 (14) | C20—H00i     | 1.1110     |
| N4—C14     | 1.494 (13) | C6—C5        | 1.360 (17) |
| N4—C8      | 1.450 (15) | C6—H00R      | 1.1030     |
| N4—C20     | 1.473 (14) | C4—C5        | 1.418 (18) |
| N1—C1      | 1.146 (14) | C4—H00S      | 1.1030     |
| N2—C2      | 1.148 (14) | C5—H00T      | 1.1030     |
| C9—C10     | 1.394 (17) | C12—H00U     | 1.1030     |
| C9—H00B    | 1.1030     | C26—C21      | 1.3900     |
| C19—C18    | 1.382 (16) | C26—C25      | 1.3900     |
| C19—C20    | 1.476 (15) | C21—C22      | 1.3900     |
| C14—C13    | 1.479 (15) | C21—H00W     | 1.1030     |
| C14—H00a   | 1.1110     | C25—C24      | 1.3900     |
| C14—H00c   | 1.1110     | C25—H00X     | 1.1030     |
| C3—C4      | 1.381 (16) | C24—C23      | 1.3900     |
| C3—H00G    | 1.1030     | C24—H00Y     | 1.1030     |
| C18—C17    | 1.407 (17) | C23—C22      | 1.3900     |
| C18—H00H   | 1.1030     | C23—H00Z     | 1.1030     |
| C7—C8      | 1.509 (16) | C22—H010     | 1.1030     |
|            |            |              |            |
| N6—Fe1—N5  | 166.5 (4)  | C16—C17—C18  | 119.1 (12) |
| N3—Fe1—N5  | 91.4 (4)   | H00K—C17—C18 | 120.5 (8)  |
| N3—Fe1—N6  | 88.9 (4)   | H00K—C17—C16 | 120.5 (7)  |
| N4—Fe1—N5  | 83.9 (4)   | C16—C15—N6   | 122.2 (11) |
| N4—Fe1—N6  | 82.7 (4)   | H00L—C15—N6  | 118.9 (7)  |
| N4—Fe1—N3  | 85.1 (4)   | H00L—C15—C16 | 118.9 (7)  |
| N1—Fe1—N5  | 90.2 (4)   | C15—C16—C17  | 119.1 (11) |
| N1—Fe1—N6  | 89.2 (4)   | H00M—C16—C17 | 120.4 (7)  |
| N1—Fe1—N3  | 177.7 (4)  | H00M—C16—C15 | 120.4 (7)  |
| N1—Fe1—N4  | 93.4 (4)   | C12—C11—C10  | 119.8 (11) |
| N2—Fe1—N5  | 95.2 (4)   | H00N—C11—C10 | 120.1 (7)  |
| N2—Fe1—N6  | 98.2 (4)   | H00N—C11—C12 | 120.1 (7)  |
| N2—Fe1—N3  | 94.2 (4)   | C7—C8—N4     | 114.3 (10) |
| N2—Fe1—N4  | 178.9 (4)  | H00d—C8—N4   | 108.7 (6)  |
| N2—Fe1—N1  | 87.3 (4)   | H00d—C8—C7   | 108.7 (6)  |
| C9—N5—Fe1  | 127.8 (8)  | H00e—C8—N4   | 108.7 (6)  |
| C13—N5—Fe1 | 113.6 (7)  | H00e—C8—C7   | 108.7 (6)  |
| C13—N5—C9  | 118.6 (10) | H00e—C8—H00d | 107.6      |
| C19—N6—Fe1 | 112.8 (7)  | C11—C10—C9   | 117.7 (11) |
| C15—N6—Fe1 | 128.0 (8)  | H00P—C10—C9  | 121.2 (7)  |
| C15—N6—C19 | 119.1 (10) | H00P—C10—C11 | 121.2 (7)  |

|                |             |                 |             |
|----------------|-------------|-----------------|-------------|
| C3—N3—Fe1      | 126.7 (8)   | C19—C20—N4      | 108.2 (8)   |
| C7—N3—Fe1      | 116.3 (8)   | H00f—C20—N4     | 110.1 (6)   |
| C7—N3—C3       | 117.0 (10)  | H00f—C20—C19    | 110.1 (6)   |
| C14—N4—Fe1     | 105.1 (7)   | H00i—C20—N4     | 110.1 (6)   |
| C8—N4—Fe1      | 109.8 (7)   | H00i—C20—C19    | 110.1 (6)   |
| C8—N4—C14      | 111.5 (9)   | H00i—C20—H00f   | 108.4       |
| C20—N4—Fe1     | 105.5 (6)   | C5—C6—C7        | 119.7 (11)  |
| C20—N4—C14     | 112.3 (8)   | H00R—C6—C7      | 120.2 (7)   |
| C20—N4—C8      | 112.2 (9)   | H00R—C6—C5      | 120.2 (7)   |
| C1—N1—Fe1      | 166.2 (10)  | C5—C4—C3        | 119.4 (12)  |
| C2—N2—Fe1      | 169.2 (10)  | H00S—C4—C3      | 120.3 (8)   |
| C10—C9—N5      | 122.2 (11)  | H00S—C4—C5      | 120.3 (7)   |
| H00B—C9—N5     | 118.9 (7)   | C4—C5—C6        | 117.1 (11)  |
| H00B—C9—C10    | 118.9 (7)   | H00T—C5—C6      | 121.5 (8)   |
| C18—C19—N6     | 121.2 (10)  | H00T—C5—C4      | 121.5 (7)   |
| C20—C19—N6     | 115.0 (9)   | C11—C12—C13     | 119.5 (11)  |
| C20—C19—C18    | 123.8 (10)  | H00U—C12—C13    | 120.3 (7)   |
| N1—C1—S1       | 177.9 (11)  | H00U—C12—C11    | 120.3 (7)   |
| N2—C2—S2       | 176.9 (11)  | C21—C26—I1      | 118.4       |
| C13—C14—N4     | 111.0 (9)   | C25—C26—I1      | 121.5       |
| H00a—C14—N4    | 109.4 (6)   | C25—C26—C21     | 120.0       |
| H00a—C14—C13   | 109.4 (6)   | C22—C21—C26     | 120.0       |
| H00c—C14—N4    | 109.4 (6)   | H00W—C21—C26    | 120.0 (4)   |
| H00c—C14—C13   | 109.4 (6)   | H00W—C21—C22    | 120.0 (4)   |
| H00c—C14—H00a  | 108.0       | C24—C25—C26     | 120.0       |
| C4—C3—N3       | 123.2 (11)  | H00X—C25—C26    | 120.0 (4)   |
| H00G—C3—N3     | 118.4 (6)   | H00X—C25—C24    | 120.0 (4)   |
| H00G—C3—C4     | 118.4 (8)   | C23—C24—C25     | 120.0       |
| C17—C18—C19    | 119.2 (12)  | H00Y—C24—C25    | 120.0 (4)   |
| H00H—C18—C19   | 120.4 (7)   | H00Y—C24—C23    | 120.0 (4)   |
| H00H—C18—C17   | 120.4 (8)   | C22—C23—C24     | 120.0       |
| C8—C7—N3       | 114.5 (10)  | H00Z—C23—C24    | 120.0 (4)   |
| C6—C7—N3       | 123.7 (11)  | H00Z—C23—C22    | 120.0 (4)   |
| C6—C7—C8       | 121.8 (11)  | C23—C22—C21     | 120.0       |
| C14—C13—N5     | 114.3 (10)  | H010—C22—C21    | 120.0 (4)   |
| C12—C13—N5     | 122.1 (10)  | H010—C22—C23    | 120.0 (4)   |
| C12—C13—C14    | 123.5 (11)  |                 |             |
| I1—C26—C21—C22 | −177.5 (2)  | C9—C10—C11—C12  | 1.5 (14)    |
| I1—C26—C25—C24 | 177.4 (2)   | C19—N6—C15—C16  | 2.8 (12)    |
| Fe1—N5—C9—C10  | −176.5 (10) | C19—C18—C17—C16 | −0.3 (13)   |
| Fe1—N5—C13—C14 | −5.3 (8)    | C19—C20—N4—C14  | 155.6 (9)   |
| Fe1—N5—C13—C12 | 177.5 (8)   | C19—C20—N4—C8   | −77.9 (10)  |
| Fe1—N6—C19—C18 | −178.3 (7)  | C14—N4—C8—C7    | −115.7 (9)  |
| Fe1—N6—C19—C20 | 3.5 (8)     | C14—C13—C12—C11 | −176.1 (12) |
| Fe1—N6—C15—C16 | 179.7 (9)   | C3—N3—C7—C8     | 179.9 (10)  |
| Fe1—N3—C3—C4   | −178.6 (10) | C3—N3—C7—C6     | −1.3 (12)   |
| Fe1—N3—C7—C8   | −1.6 (8)    | C3—C4—C5—C6     | −2.4 (13)   |

|                |             |                 |             |
|----------------|-------------|-----------------|-------------|
| Fe1—N3—C7—C6   | 177.2 (8)   | C18—C19—N6—C15  | −1.0 (13)   |
| Fe1—N4—C14—C13 | −37.0 (7)   | C18—C17—C16—C15 | 2.0 (14)    |
| Fe1—N4—C8—C7   | 0.3 (7)     | C7—N3—C3—C4     | −0.3 (12)   |
| Fe1—N4—C20—C19 | 41.6 (7)    | C7—C8—N4—C20    | 117.3 (10)  |
| N5—C9—C10—C11  | −2.4 (14)   | C7—C6—C5—C4     | 0.9 (13)    |
| N5—C13—C14—N4  | 29.1 (11)   | C13—N5—C9—C10   | 2.4 (12)    |
| N5—C13—C12—C11 | 0.8 (13)    | C13—C14—N4—C8   | 81.9 (10)   |
| N6—C19—C18—C17 | −0.2 (13)   | C13—C14—N4—C20  | −151.2 (9)  |
| N6—C19—C20—N4  | −30.8 (10)  | C13—C12—C11—C10 | −0.8 (15)   |
| N6—C15—C16—C17 | −3.3 (13)   | C17—C18—C19—C20 | 177.8 (10)  |
| N3—C3—C4—C5    | 2.2 (14)    | C15—N6—C19—C20  | −179.2 (10) |
| N3—C7—C8—N4    | 0.8 (11)    | C8—C7—C6—C5     | 179.7 (12)  |
| N3—C7—C6—C5    | 0.9 (13)    | C26—C21—C22—C23 | 0.0 (8)     |
| N4—C14—C13—C12 | −153.8 (10) | C26—C25—C24—C23 | 0.0 (8)     |
| N4—C8—C7—C6    | −178.0 (10) | C21—C26—C25—C24 | 0.0 (8)     |
| N4—C20—C19—C18 | 151.0 (9)   | C21—C22—C23—C24 | 0.0 (8)     |
| C9—N5—C13—C14  | 175.6 (10)  | C25—C26—C21—C22 | −0.0 (8)    |
| C9—N5—C13—C12  | −1.5 (12)   | C25—C24—C23—C22 | −0.0 (8)    |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) iodobenzene monosolvate (I\_150K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 14.191$  (2) Å

$b = 12.8422$  (13) Å

$c = 15.309$  (2) Å

$\beta = 109.185$  (16)°

$V = 2635.1$  (7) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.680$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3360 reflections

$\theta = 2.1$ – $22.9^\circ$

$\mu = 1.93$  mm<sup>−1</sup>

$T = 150$  K

Plate, red

$0.17 \times 0.1 \times 0.03$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.758$ ,  $T_{\max} = 1.000$

41057 measured reflections

4650 independent reflections

2669 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.258$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.1^\circ$

$h = -16 \rightarrow 16$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.120$

$wR(F^2) = 0.323$

$S = 1.58$

4650 reflections

313 parameters

0 restraints

44 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 2.72$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -2.55$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| I1   | 0.63202 (12) | 0.03494 (11) | 0.56321 (11) | 0.0788 (6)                       |
| Fe1  | 0.66313 (14) | 0.34008 (13) | 0.32119 (12) | 0.0211 (5)                       |
| S2   | 0.4771 (3)   | 0.0535 (3)   | 0.1686 (3)   | 0.0387 (10)                      |
| S1   | 0.7446 (3)   | 0.3037 (3)   | 0.0480 (3)   | 0.0418 (10)                      |
| N3   | 0.6112 (8)   | 0.3499 (7)   | 0.4233 (7)   | 0.023 (2)                        |
| N5   | 0.7721 (8)   | 0.2470 (8)   | 0.3934 (7)   | 0.025 (2)                        |
| N4   | 0.7568 (7)   | 0.4516 (8)   | 0.3856 (7)   | 0.022 (2)                        |
| N6   | 0.5787 (8)   | 0.4581 (8)   | 0.2637 (7)   | 0.025 (2)                        |
| N1   | 0.7156 (8)   | 0.3374 (8)   | 0.2175 (8)   | 0.027 (2)                        |
| N2   | 0.5737 (9)   | 0.2290 (8)   | 0.2609 (8)   | 0.031 (3)                        |
| C9   | 0.7679 (10)  | 0.1421 (9)   | 0.4075 (9)   | 0.026 (3)                        |
| C7   | 0.6543 (10)  | 0.4202 (10)  | 0.4877 (8)   | 0.026 (3)                        |
| C2   | 0.5334 (9)   | 0.1566 (10)  | 0.2219 (9)   | 0.026 (3)                        |
| C3   | 0.5375 (10)  | 0.2925 (10)  | 0.4359 (9)   | 0.030 (3)                        |
| C8   | 0.7353 (10)  | 0.4830 (9)   | 0.4705 (9)   | 0.026 (3)                        |
| C13  | 0.8587 (9)   | 0.2963 (9)   | 0.4336 (9)   | 0.024 (3)                        |
| C1   | 0.7266 (9)   | 0.3248 (9)   | 0.1460 (8)   | 0.022 (3)                        |
| C19  | 0.6297 (9)   | 0.5489 (9)   | 0.2733 (8)   | 0.023 (3)                        |
| C14  | 0.8590 (9)   | 0.4078 (10)  | 0.4067 (9)   | 0.025 (3)                        |
| C18  | 0.5829 (10)  | 0.6417 (11)  | 0.2390 (8)   | 0.029 (3)                        |
| C10  | 0.8485 (11)  | 0.0874 (10)  | 0.4623 (10)  | 0.036 (4)                        |
| C15  | 0.4779 (9)   | 0.4593 (10)  | 0.2188 (9)   | 0.028 (3)                        |
| C6   | 0.6225 (10)  | 0.4423 (11)  | 0.5632 (9)   | 0.031 (3)                        |
| C11  | 0.9377 (10)  | 0.1414 (10)  | 0.5008 (9)   | 0.031 (3)                        |
| C20  | 0.7379 (9)   | 0.5405 (9)   | 0.3205 (9)   | 0.026 (3)                        |
| C16  | 0.4261 (10)  | 0.5474 (10)  | 0.1817 (8)   | 0.028 (3)                        |
| C4   | 0.5029 (11)  | 0.3060 (10)  | 0.5099 (10)  | 0.036 (3)                        |
| C5   | 0.5470 (11)  | 0.3844 (11)  | 0.5745 (10)  | 0.037 (4)                        |
| C17  | 0.4773 (11)  | 0.6404 (11)  | 0.1918 (10)  | 0.038 (4)                        |
| C12  | 0.9430 (10)  | 0.2464 (10)  | 0.4881 (9)   | 0.029 (3)                        |
| C26  | 0.6977 (8)   | 0.1321 (8)   | 0.6709 (8)   | 0.059 (5)                        |
| C21  | 0.6807 (8)   | 0.1172 (8)   | 0.7544 (10)  | 0.059 (5)                        |
| C25  | 0.7561 (9)   | 0.2152 (10)  | 0.6606 (8)   | 0.060 (5)                        |
| C24  | 0.7973 (8)   | 0.2832 (8)   | 0.7338 (11)  | 0.068 (6)                        |
| C22  | 0.7219 (10)  | 0.1853 (11)  | 0.8276 (8)   | 0.076 (6)                        |
| C23  | 0.7803 (10)  | 0.2683 (10)  | 0.8173 (9)   | 0.091 (8)                        |
| H00X | 0.7696 (9)   | 0.2270 (10)  | 0.5944 (8)   | 0.072 (6)*                       |
| H00Y | 0.8436 (8)   | 0.3491 (8)   | 0.7257 (11)  | 0.082 (7)*                       |
| H010 | 0.8130 (10)  | 0.3223 (10)  | 0.8754 (9)   | 0.109 (9)*                       |
| H00Z | 0.7084 (10)  | 0.1734 (11)  | 0.8938 (8)   | 0.091 (7)*                       |
| H00W | 0.6344 (8)   | 0.0513 (8)   | 0.7625 (10)  | 0.071 (6)*                       |
| H00E | 0.5028 (10)  | 0.2315 (10)  | 0.3846 (9)   | 0.036 (4)*                       |
| H00N | 0.6577 (10)  | 0.5051 (11)  | 0.6122 (9)   | 0.037 (4)*                       |
| H00S | 0.5208 (11)  | 0.3989 (11)  | 0.6337 (10)  | 0.045 (4)*                       |
| H00R | 0.4426 (11)  | 0.2567 (10)  | 0.5179 (10)  | 0.043 (4)*                       |

|      |             |             |             |            |
|------|-------------|-------------|-------------|------------|
| H00B | 0.6974 (10) | 0.1004 (9)  | 0.3737 (9)  | 0.031 (4)* |
| H00U | 1.0129 (10) | 0.2896 (10) | 0.5208 (9)  | 0.035 (4)* |
| H00O | 1.0051 (10) | 0.0988 (10) | 0.5422 (9)  | 0.038 (4)* |
| H00L | 0.8429 (11) | 0.0036 (10) | 0.4755 (10) | 0.043 (4)* |
| H00M | 0.4364 (9)  | 0.3855 (10) | 0.2123 (9)  | 0.034 (4)* |
| H00Q | 0.3452 (10) | 0.5442 (10) | 0.1445 (8)  | 0.034 (4)* |
| H00T | 0.4373 (11) | 0.7130 (11) | 0.1638 (10) | 0.045 (4)* |
| H00K | 0.6256 (10) | 0.7149 (11) | 0.2476 (8)  | 0.035 (4)* |
| H00a | 0.8045 (10) | 0.4750 (9)  | 0.5309 (9)  | 0.031 (3)* |
| H00c | 0.7133 (10) | 0.5664 (9)  | 0.4646 (9)  | 0.031 (3)* |
| H00d | 0.8833 (9)  | 0.4144 (10) | 0.3449 (9)  | 0.030 (3)* |
| H00f | 0.9123 (9)  | 0.4523 (10) | 0.4641 (9)  | 0.030 (3)* |
| H00g | 0.7669 (9)  | 0.6136 (9)  | 0.3588 (9)  | 0.031 (3)* |
| H00h | 0.7766 (9)  | 0.5277 (9)  | 0.2690 (9)  | 0.031 (3)* |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| I1  | 0.0953 (12) | 0.0654 (10) | 0.0691 (11) | 0.0012 (8)   | 0.0181 (9)  | 0.0014 (7)   |
| Fe1 | 0.0255 (10) | 0.0231 (9)  | 0.0150 (9)  | −0.0020 (8)  | 0.0069 (7)  | 0.0004 (8)   |
| S2  | 0.035 (2)   | 0.036 (2)   | 0.045 (2)   | −0.0085 (16) | 0.0131 (18) | −0.0176 (17) |
| S1  | 0.066 (3)   | 0.039 (2)   | 0.028 (2)   | 0.0132 (19)  | 0.026 (2)   | 0.0050 (16)  |
| N3  | 0.030 (6)   | 0.021 (5)   | 0.018 (6)   | −0.001 (4)   | 0.007 (5)   | −0.001 (4)   |
| N5  | 0.029 (6)   | 0.024 (6)   | 0.019 (6)   | 0.003 (5)    | 0.005 (5)   | −0.004 (4)   |
| N4  | 0.015 (5)   | 0.029 (6)   | 0.020 (6)   | 0.002 (4)    | 0.002 (4)   | −0.000 (4)   |
| N6  | 0.032 (6)   | 0.027 (6)   | 0.014 (5)   | −0.000 (5)   | 0.005 (5)   | −0.008 (4)   |
| N1  | 0.030 (6)   | 0.027 (6)   | 0.024 (6)   | 0.006 (5)    | 0.010 (5)   | 0.004 (5)    |
| N2  | 0.037 (7)   | 0.026 (6)   | 0.025 (6)   | −0.003 (5)   | 0.002 (5)   | −0.002 (5)   |
| C9  | 0.040 (8)   | 0.013 (6)   | 0.024 (7)   | −0.012 (5)   | 0.011 (6)   | −0.003 (5)   |
| C7  | 0.030 (7)   | 0.036 (7)   | 0.012 (6)   | 0.008 (6)    | 0.008 (6)   | 0.001 (6)    |
| C2  | 0.029 (7)   | 0.025 (7)   | 0.023 (7)   | −0.002 (6)   | 0.008 (6)   | −0.006 (6)   |
| C3  | 0.038 (8)   | 0.034 (7)   | 0.012 (7)   | −0.003 (6)   | 0.001 (6)   | 0.001 (5)    |
| C8  | 0.033 (7)   | 0.022 (6)   | 0.021 (7)   | −0.006 (5)   | 0.005 (6)   | 0.001 (5)    |
| C13 | 0.025 (7)   | 0.026 (6)   | 0.018 (7)   | 0.004 (6)    | 0.004 (6)   | 0.001 (5)    |
| C1  | 0.039 (8)   | 0.019 (6)   | 0.015 (7)   | 0.013 (5)    | 0.017 (6)   | 0.011 (5)    |
| C19 | 0.026 (7)   | 0.025 (7)   | 0.014 (6)   | 0.003 (5)    | 0.002 (5)   | 0.013 (5)    |
| C14 | 0.022 (7)   | 0.036 (7)   | 0.017 (7)   | −0.005 (6)   | 0.007 (6)   | −0.001 (6)   |
| C18 | 0.029 (7)   | 0.040 (8)   | 0.013 (6)   | 0.005 (6)    | −0.001 (6)  | 0.003 (6)    |
| C10 | 0.059 (10)  | 0.012 (6)   | 0.032 (8)   | 0.006 (6)    | 0.007 (7)   | 0.000 (6)    |
| C15 | 0.016 (7)   | 0.040 (8)   | 0.024 (7)   | −0.010 (6)   | 0.000 (6)   | −0.005 (6)   |
| C6  | 0.032 (8)   | 0.042 (8)   | 0.018 (7)   | −0.006 (6)   | 0.006 (6)   | −0.007 (6)   |
| C11 | 0.039 (8)   | 0.025 (7)   | 0.032 (8)   | 0.015 (6)    | 0.013 (7)   | 0.010 (6)    |
| C20 | 0.028 (7)   | 0.023 (6)   | 0.025 (7)   | −0.006 (5)   | 0.010 (6)   | −0.002 (5)   |
| C16 | 0.023 (7)   | 0.043 (8)   | 0.010 (6)   | 0.010 (6)    | −0.007 (5)  | −0.005 (6)   |
| C4  | 0.053 (9)   | 0.033 (7)   | 0.030 (8)   | −0.002 (6)   | 0.026 (7)   | 0.008 (6)    |
| C5  | 0.047 (9)   | 0.052 (9)   | 0.020 (8)   | 0.013 (7)    | 0.021 (7)   | −0.002 (7)   |
| C17 | 0.044 (9)   | 0.040 (9)   | 0.037 (9)   | 0.007 (7)    | 0.025 (8)   | −0.007 (7)   |
| C12 | 0.027 (8)   | 0.030 (7)   | 0.033 (8)   | −0.005 (6)   | 0.011 (6)   | −0.003 (6)   |

|     |            |            |            |            |            |            |
|-----|------------|------------|------------|------------|------------|------------|
| C26 | 0.042 (10) | 0.044 (10) | 0.092 (16) | 0.006 (7)  | 0.025 (10) | 0.006 (9)  |
| C21 | 0.053 (11) | 0.054 (11) | 0.066 (13) | 0.015 (8)  | 0.014 (10) | 0.017 (10) |
| C25 | 0.064 (12) | 0.054 (11) | 0.074 (14) | 0.013 (9)  | 0.038 (11) | 0.016 (10) |
| C24 | 0.036 (10) | 0.062 (12) | 0.098 (17) | 0.009 (8)  | 0.009 (11) | 0.022 (12) |
| C22 | 0.082 (15) | 0.054 (12) | 0.096 (18) | 0.025 (10) | 0.033 (13) | 0.002 (12) |
| C23 | 0.061 (14) | 0.107 (19) | 0.10 (2)   | 0.041 (13) | 0.024 (13) | 0.043 (16) |

*Geometric parameters (Å, °)*

|                    |            |               |            |
|--------------------|------------|---------------|------------|
| I1—I1 <sup>i</sup> | 3.718 (3)  | C14—H00d      | 1.1110     |
| I1—C26             | 2.034 (10) | C14—H00f      | 1.1110     |
| Fe1—N3             | 1.939 (11) | C18—C17       | 1.43 (2)   |
| Fe1—N5             | 1.979 (10) | C18—H00K      | 1.1030     |
| Fe1—N4             | 1.982 (10) | C10—C11       | 1.393 (19) |
| Fe1—N6             | 1.953 (10) | C10—H00L      | 1.1030     |
| Fe1—N1             | 1.963 (11) | C15—C16       | 1.367 (17) |
| Fe1—N2             | 1.931 (11) | C15—H00M      | 1.1030     |
| S2—C2              | 1.621 (13) | C6—C5         | 1.361 (19) |
| S1—C1              | 1.624 (12) | C6—H00N       | 1.1030     |
| N3—C7              | 1.328 (16) | C11—C12       | 1.368 (17) |
| N3—C3              | 1.344 (16) | C11—H00O      | 1.1030     |
| N5—C9              | 1.369 (15) | C20—H00g      | 1.1110     |
| N5—C13             | 1.338 (16) | C20—H00h      | 1.1110     |
| N4—C8              | 1.485 (16) | C16—C17       | 1.379 (19) |
| N4—C14             | 1.489 (15) | C16—H00Q      | 1.1030     |
| N4—C20             | 1.480 (15) | C4—C5         | 1.41 (2)   |
| N6—C19             | 1.354 (15) | C4—H00R       | 1.1030     |
| N6—C15             | 1.368 (16) | C5—H00S       | 1.1030     |
| N1—C1              | 1.167 (16) | C17—H00T      | 1.1030     |
| N2—C2              | 1.150 (16) | C12—H00U      | 1.1030     |
| C9—C10             | 1.369 (18) | C26—C21       | 1.3900     |
| C9—H00B            | 1.1030     | C26—C25       | 1.3900     |
| C7—C8              | 1.497 (18) | C21—C22       | 1.3900     |
| C7—C6              | 1.401 (17) | C21—H00W      | 1.1030     |
| C3—C4              | 1.386 (18) | C25—C24       | 1.3900     |
| C3—H00E            | 1.1030     | C25—H00X      | 1.1030     |
| C8—H00a            | 1.1110     | C24—C23       | 1.3900     |
| C8—H00c            | 1.1110     | C24—H00Y      | 1.1030     |
| C13—C14            | 1.490 (17) | C22—C23       | 1.3900     |
| C13—C12            | 1.373 (18) | C22—H00Z      | 1.1030     |
| C19—C18            | 1.381 (17) | C23—H010      | 1.1030     |
| C19—C20            | 1.471 (17) |               |            |
| N5—Fe1—N3          | 91.3 (4)   | H00f—C14—N4   | 109.8 (6)  |
| N4—Fe1—N3          | 85.5 (4)   | H00f—C14—C13  | 109.8 (7)  |
| N4—Fe1—N5          | 83.4 (4)   | H00f—C14—H00d | 108.2      |
| N6—Fe1—N3          | 88.5 (4)   | C17—C18—C19   | 118.3 (13) |
| N6—Fe1—N5          | 166.2 (4)  | H00K—C18—C19  | 120.8 (8)  |

|              |            |               |            |
|--------------|------------|---------------|------------|
| N6—Fe1—N4    | 82.8 (4)   | H00K—C18—C17  | 120.8 (8)  |
| N1—Fe1—N3    | 177.3 (4)  | C11—C10—C9    | 117.6 (12) |
| N1—Fe1—N5    | 90.4 (4)   | H00L—C10—C9   | 121.2 (8)  |
| N1—Fe1—N4    | 92.5 (4)   | H00L—C10—C11  | 121.2 (7)  |
| N1—Fe1—N6    | 89.4 (4)   | C16—C15—N6    | 123.2 (12) |
| N2—Fe1—N3    | 94.0 (5)   | H00M—C15—N6   | 118.4 (7)  |
| N2—Fe1—N5    | 95.2 (4)   | H00M—C15—C16  | 118.4 (8)  |
| N2—Fe1—N4    | 178.5 (4)  | C5—C6—C7      | 118.3 (13) |
| N2—Fe1—N6    | 98.5 (4)   | H00N—C6—C7    | 120.8 (8)  |
| N2—Fe1—N1    | 88.0 (5)   | H00N—C6—C5    | 120.8 (8)  |
| C7—N3—Fe1    | 116.0 (8)  | C12—C11—C10   | 121.0 (12) |
| C3—N3—Fe1    | 126.8 (8)  | H00O—C11—C10  | 119.5 (7)  |
| C3—N3—C7     | 117.2 (11) | H00O—C11—C12  | 119.5 (8)  |
| C9—N5—Fe1    | 128.0 (9)  | C19—C20—N4    | 108.4 (9)  |
| C13—N5—Fe1   | 113.7 (8)  | H00g—C20—N4   | 110.0 (6)  |
| C13—N5—C9    | 118.3 (11) | H00g—C20—C19  | 110.0 (7)  |
| C8—N4—Fe1    | 109.5 (7)  | H00h—C20—N4   | 110.0 (6)  |
| C14—N4—Fe1   | 106.3 (7)  | H00h—C20—C19  | 110.0 (7)  |
| C14—N4—C8    | 112.2 (9)  | H00h—C20—H00g | 108.4      |
| C20—N4—Fe1   | 106.2 (7)  | C17—C16—C15   | 118.6 (12) |
| C20—N4—C8    | 109.7 (9)  | H00Q—C16—C15  | 120.7 (8)  |
| C20—N4—C14   | 112.6 (10) | H00Q—C16—C17  | 120.7 (8)  |
| C19—N6—Fe1   | 113.1 (8)  | C5—C4—C3      | 118.0 (13) |
| C15—N6—Fe1   | 128.3 (9)  | H00R—C4—C3    | 121.0 (8)  |
| C15—N6—C19   | 118.6 (11) | H00R—C4—C5    | 121.0 (8)  |
| C1—N1—Fe1    | 164.7 (11) | C4—C5—C6      | 119.2 (12) |
| C2—N2—Fe1    | 169.0 (11) | H00S—C5—C6    | 120.4 (8)  |
| C10—C9—N5    | 122.1 (12) | H00S—C5—C4    | 120.4 (8)  |
| H00B—C9—N5   | 119.0 (7)  | C16—C17—C18   | 119.4 (13) |
| H00B—C9—C10  | 119.0 (8)  | H00T—C17—C18  | 120.3 (8)  |
| C8—C7—N3     | 115.4 (11) | H00T—C17—C16  | 120.3 (8)  |
| C6—C7—N3     | 123.8 (12) | C11—C12—C13   | 118.0 (12) |
| C6—C7—C8     | 120.7 (12) | H00U—C12—C13  | 121.0 (8)  |
| N2—C2—S2     | 179.0 (12) | H00U—C12—C11  | 121.0 (8)  |
| C4—C3—N3     | 123.4 (12) | C21—C26—I1    | 119.2 (8)  |
| H00E—C3—N3   | 118.3 (7)  | C25—C26—I1    | 120.7 (8)  |
| H00E—C3—C4   | 118.3 (8)  | C25—C26—C21   | 120.0      |
| C7—C8—N4     | 113.5 (10) | C22—C21—C26   | 120.0      |
| H00a—C8—N4   | 108.9 (6)  | H00W—C21—C26  | 120.0      |
| H00a—C8—C7   | 108.9 (7)  | H00W—C21—C22  | 120.0      |
| H00c—C8—N4   | 108.9 (6)  | C24—C25—C26   | 120.0      |
| H00c—C8—C7   | 108.9 (7)  | H00X—C25—C26  | 120.0 (5)  |
| H00c—C8—H00a | 107.7      | H00X—C25—C24  | 120.0 (5)  |
| C14—C13—N5   | 114.4 (11) | C23—C24—C25   | 120.0      |
| C12—C13—N5   | 123.0 (12) | H00Y—C24—C25  | 120.0 (5)  |
| C12—C13—C14  | 122.4 (12) | H00Y—C24—C23  | 120.0 (5)  |
| N1—C1—S1     | 178.0 (11) | C23—C22—C21   | 120.0      |
| C18—C19—N6   | 121.9 (11) | H00Z—C22—C21  | 120.0 (5)  |

|                |             |                 |             |
|----------------|-------------|-----------------|-------------|
| C20—C19—N6     | 115.1 (10)  | H00Z—C22—C23    | 120.0 (5)   |
| C20—C19—C18    | 123.0 (11)  | C22—C23—C24     | 120.0       |
| C13—C14—N4     | 109.4 (10)  | H010—C23—C24    | 120.0 (5)   |
| H00d—C14—N4    | 109.8 (6)   | H010—C23—C22    | 120.0 (5)   |
| H00d—C14—C13   | 109.8 (7)   |                 |             |
|                |             |                 |             |
| I1—C26—C21—C22 | −177.6 (3)  | C9—C10—C11—C12  | 3.3 (16)    |
| I1—C26—C25—C24 | 177.5 (3)   | C7—N3—C3—C4     | 2.4 (13)    |
| Fe1—N3—C7—C8   | 1.3 (9)     | C7—C8—N4—C14    | −114.8 (11) |
| Fe1—N3—C7—C6   | 175.9 (9)   | C7—C8—N4—C20    | 119.2 (11)  |
| Fe1—N3—C3—C4   | −178.4 (11) | C7—C6—C5—C4     | −0.9 (15)   |
| Fe1—N5—C9—C10  | −177.4 (11) | C3—N3—C7—C8     | −179.4 (11) |
| Fe1—N5—C13—C14 | −7.1 (9)    | C3—N3—C7—C6     | −4.8 (13)   |
| Fe1—N5—C13—C12 | 178.8 (9)   | C3—C4—C5—C6     | −1.2 (15)   |
| Fe1—N4—C8—C7   | 3.0 (8)     | C8—N4—C14—C13   | 81.6 (10)   |
| Fe1—N4—C14—C13 | −38.1 (8)   | C8—N4—C20—C19   | −78.5 (10)  |
| Fe1—N4—C20—C19 | 39.8 (8)    | C8—C7—C6—C5     | 178.5 (13)  |
| Fe1—N6—C19—C18 | −178.7 (8)  | C13—N5—C9—C10   | 1.2 (14)    |
| Fe1—N6—C19—C20 | 2.8 (9)     | C13—C14—N4—C20  | −154.0 (10) |
| Fe1—N6—C15—C16 | 179.4 (11)  | C13—C12—C11—C10 | −2.3 (16)   |
| N3—C7—C8—N4    | −2.9 (12)   | C19—N6—C15—C16  | 1.2 (13)    |
| N3—C7—C6—C5    | 4.1 (15)    | C19—C18—C17—C16 | −0.3 (14)   |
| N3—C3—C4—C5    | 0.5 (15)    | C19—C20—N4—C14  | 155.8 (10)  |
| N5—C9—C10—C11  | −2.8 (15)   | C14—C13—C12—C11 | −173.1 (13) |
| N5—C13—C14—N4  | 30.6 (12)   | C18—C19—N6—C15  | −0.2 (15)   |
| N5—C13—C12—C11 | 0.6 (15)    | C18—C17—C16—C15 | 1.2 (15)    |
| N4—C8—C7—C6    | −177.7 (10) | C15—N6—C19—C20  | −178.7 (12) |
| N4—C14—C13—C12 | −155.2 (10) | C20—C19—C18—C17 | 178.2 (12)  |
| N4—C20—C19—N6  | −28.9 (11)  | C26—C21—C22—C23 | 0.0 (9)     |
| N4—C20—C19—C18 | 152.7 (10)  | C26—C25—C24—C23 | 0.0 (9)     |
| N6—C19—C18—C17 | −0.1 (14)   | C21—C26—C25—C24 | 0.0 (9)     |
| N6—C15—C16—C17 | −1.6 (16)   | C21—C22—C23—C24 | 0.0 (9)     |
| C9—N5—C13—C14  | 174.1 (11)  | C25—C26—C21—C22 | −0.0 (9)    |
| C9—N5—C13—C12  | −0.0 (14)   | C25—C24—C23—C22 | −0.0 (9)    |

Symmetry code: (i)  $-x+1, -y, -z+1$ .

### Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) iodobenzene monosolvate (I\_155K)

#### Crystal data

$[\text{Fe}(\text{NCS})_2(\text{C}_{18}\text{H}_{18}\text{N}_4)] \cdot \text{C}_6\text{H}_5\text{I}$

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 15.209$  (3) Å

$b = 12.1403$  (14) Å

$c = 15.353$  (2) Å

$\beta = 110.897$  (18)°

$V = 2648.3$  (8) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.671$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4171 reflections

$\theta = 2.1$ – $22.4^\circ$

$\mu = 1.92$  mm<sup>−1</sup>

$T = 155$  K

Plate, yellow

$0.17 \times 0.1 \times 0.03$  mm



*Data collection*

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector  
Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source  
Mirror monochromator  
Detector resolution: 10.0000 pixels mm<sup>-1</sup>  
 $\omega$  scans  
Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.718$ ,  $T_{\max} = 1.000$   
40897 measured reflections  
4681 independent reflections  
2669 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.175$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -18 \rightarrow 18$   
 $k = -14 \rightarrow 14$   
 $l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.121$   
 $wR(F^2) = 0.320$   
 $S = 1.66$   
4681 reflections  
262 parameters  
0 restraints

33 constraints  
Primary atom site location: dual  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = -0.001$   
 $\Delta\rho_{\max} = 1.36 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -1.00 \text{ e } \text{\AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|-------------|----------------------------------|
| Fe1  | 0.65529 (10) | 0.32751 (12) | 0.30784 (9) | 0.0572 (5)                       |
| S2   | 0.4732 (2)   | 0.0082 (3)   | 0.1706 (3)  | 0.0857 (10)                      |
| S1   | 0.7518 (3)   | 0.3247 (3)   | 0.0414 (3)  | 0.1157 (15)                      |
| N3   | 0.6105 (5)   | 0.3409 (6)   | 0.4238 (5)  | 0.0534 (19)                      |
| N4   | 0.7438 (5)   | 0.4640 (6)   | 0.3846 (5)  | 0.055 (2)                        |
| N6   | 0.5676 (5)   | 0.4677 (6)   | 0.2580 (5)  | 0.061 (2)                        |
| N5   | 0.7834 (5)   | 0.2456 (6)   | 0.3924 (5)  | 0.0511 (19)                      |
| C7   | 0.6514 (6)   | 0.4195 (8)   | 0.4854 (6)  | 0.054 (2)                        |
| N1   | 0.7048 (6)   | 0.3434 (7)   | 0.1995 (7)  | 0.072 (3)                        |
| N2   | 0.5731 (6)   | 0.1947 (8)   | 0.2520 (6)  | 0.073 (3)                        |
| C13  | 0.8547 (6)   | 0.3136 (7)   | 0.4283 (5)  | 0.049 (2)                        |
| C19  | 0.6131 (7)   | 0.5637 (8)   | 0.2726 (6)  | 0.056 (2)                        |
| C8   | 0.7281 (7)   | 0.4842 (8)   | 0.4730 (6)  | 0.061 (3)                        |
| H00a | 0.7944 (7)   | 0.4657 (8)   | 0.5319 (6)  | 0.073 (3)*                       |
| H00b | 0.7126 (7)   | 0.5731 (8)   | 0.4769 (6)  | 0.073 (3)*                       |
| C3   | 0.5420 (8)   | 0.2831 (9)   | 0.4356 (7)  | 0.068 (3)                        |
| H00G | 0.5090 (8)   | 0.2171 (9)   | 0.3852 (7)  | 0.082 (3)*                       |
| C9   | 0.7923 (8)   | 0.1399 (8)   | 0.4108 (6)  | 0.063 (3)                        |
| H00H | 0.7302 (8)   | 0.0866 (8)   | 0.3794 (6)  | 0.075 (3)*                       |
| C15  | 0.4740 (7)   | 0.4676 (9)   | 0.2098 (6)  | 0.066 (3)                        |
| H00I | 0.4360 (7)   | 0.3884 (9)   | 0.1936 (6)  | 0.079 (3)*                       |
| C6   | 0.6226 (7)   | 0.4449 (9)   | 0.5604 (7)  | 0.065 (3)                        |
| H00J | 0.6553 (7)   | 0.5128 (9)   | 0.6089 (7)  | 0.078 (3)*                       |
| C14  | 0.8402 (7)   | 0.4297 (8)   | 0.3983 (7)  | 0.060 (3)                        |
| H00c | 0.8534 (7)   | 0.4397 (8)   | 0.3321 (7)  | 0.072 (3)*                       |
| H00d | 0.8909 (7)   | 0.4827 (8)   | 0.4519 (7)  | 0.072 (3)*                       |
| C16  | 0.4250 (8)   | 0.5640 (10)  | 0.1806 (6)  | 0.072 (3)                        |

|      |            |             |            |            |
|------|------------|-------------|------------|------------|
| H00L | 0.3480 (8) | 0.5614 (10) | 0.1444 (6) | 0.087 (4)* |
| C18  | 0.5675 (8) | 0.6657 (9)  | 0.2456 (7) | 0.068 (3)  |
| H00M | 0.6072 (8) | 0.7435 (9)  | 0.2635 (7) | 0.082 (3)* |
| C10  | 0.8768 (9) | 0.0909 (9)  | 0.4685 (7) | 0.076 (3)  |
| H00N | 0.8827 (9) | 0.0017 (9)  | 0.4830 (7) | 0.091 (4)* |
| C20  | 0.7167 (7) | 0.5579 (8)  | 0.3186 (7) | 0.067 (3)  |
| H00e | 0.7431 (7) | 0.6359 (8)  | 0.3569 (7) | 0.080 (3)* |
| H00f | 0.7495 (7) | 0.5480 (8)  | 0.2650 (7) | 0.080 (3)* |
| C1   | 0.7241 (8) | 0.3374 (9)  | 0.1362 (9) | 0.075 (3)  |
| C2   | 0.5300 (8) | 0.1155 (10) | 0.2158 (8) | 0.067 (3)  |
| C17  | 0.4719 (8) | 0.6660 (10) | 0.1963 (7) | 0.079 (3)  |
| H00R | 0.4337 (8) | 0.7434 (10) | 0.1701 (7) | 0.095 (4)* |
| C11  | 0.9528 (8) | 0.1626 (8)  | 0.5062 (7) | 0.071 (3)  |
| H00S | 1.0214 (8) | 0.1299 (8)  | 0.5518 (7) | 0.085 (4)* |
| C4   | 0.5093 (8) | 0.3012 (10) | 0.5082 (7) | 0.071 (3)  |
| H00T | 0.4510 (8) | 0.2518 (10) | 0.5144 (7) | 0.085 (4)* |
| C12  | 0.9429 (7) | 0.2725 (8)  | 0.4872 (6) | 0.064 (3)  |
| H00U | 1.0031 (7) | 0.3288 (8)  | 0.5173 (6) | 0.077 (3)* |
| C5   | 0.5510 (7) | 0.3811 (10) | 0.5711 (8) | 0.071 (3)  |
| H00V | 0.5282 (7) | 0.3955 (10) | 0.6308 (8) | 0.086 (4)* |

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

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|------------|------------|------------|--------------|-------------|--------------|
| Fe1 | 0.0551 (9) | 0.0637 (9) | 0.0453 (8) | 0.0016 (7)   | 0.0088 (6)  | 0.0114 (7)   |
| S2  | 0.070 (2)  | 0.095 (2)  | 0.093 (2)  | −0.0114 (17) | 0.0309 (18) | −0.0109 (18) |
| S1  | 0.153 (4)  | 0.123 (3)  | 0.081 (2)  | 0.062 (3)    | 0.054 (2)   | 0.046 (2)    |
| N3  | 0.051 (5)  | 0.056 (5)  | 0.047 (4)  | −0.005 (4)   | 0.010 (4)   | −0.001 (4)   |
| N4  | 0.045 (4)  | 0.056 (5)  | 0.054 (5)  | −0.003 (4)   | 0.006 (4)   | 0.011 (4)    |
| N6  | 0.062 (5)  | 0.046 (5)  | 0.060 (5)  | 0.001 (4)    | 0.002 (4)   | −0.018 (4)   |
| N5  | 0.059 (5)  | 0.043 (4)  | 0.043 (4)  | −0.001 (4)   | 0.008 (4)   | 0.007 (3)    |
| C7  | 0.050 (6)  | 0.062 (6)  | 0.041 (5)  | 0.002 (5)    | 0.006 (4)   | 0.001 (4)    |
| N1  | 0.074 (6)  | 0.077 (6)  | 0.069 (6)  | 0.014 (5)    | 0.031 (5)   | 0.019 (5)    |
| N2  | 0.068 (6)  | 0.086 (7)  | 0.059 (6)  | −0.003 (5)   | 0.016 (5)   | 0.008 (5)    |
| C13 | 0.058 (6)  | 0.043 (5)  | 0.032 (4)  | −0.000 (5)   | 0.001 (4)   | 0.006 (4)    |
| C19 | 0.057 (6)  | 0.063 (6)  | 0.033 (5)  | 0.014 (5)    | −0.002 (4)  | 0.009 (4)    |
| C8  | 0.062 (6)  | 0.065 (6)  | 0.047 (6)  | −0.008 (5)   | 0.007 (5)   | −0.004 (5)   |
| C3  | 0.068 (7)  | 0.068 (7)  | 0.057 (6)  | −0.003 (6)   | 0.008 (5)   | 0.006 (5)    |
| C9  | 0.081 (8)  | 0.056 (6)  | 0.041 (5)  | −0.019 (5)   | 0.009 (5)   | 0.004 (4)    |
| C15 | 0.053 (6)  | 0.086 (8)  | 0.035 (5)  | −0.002 (6)   | −0.012 (4)  | −0.022 (5)   |
| C6  | 0.057 (6)  | 0.078 (7)  | 0.049 (6)  | −0.001 (5)   | 0.004 (5)   | −0.006 (5)   |
| C14 | 0.065 (7)  | 0.066 (6)  | 0.046 (5)  | −0.013 (5)   | 0.015 (5)   | −0.005 (5)   |
| C16 | 0.064 (7)  | 0.081 (8)  | 0.042 (6)  | 0.010 (6)    | −0.019 (5)  | −0.007 (5)   |
| C18 | 0.074 (7)  | 0.069 (7)  | 0.052 (6)  | 0.010 (6)    | 0.011 (5)   | 0.013 (5)    |
| C10 | 0.126 (10) | 0.046 (6)  | 0.042 (5)  | 0.023 (7)    | 0.013 (6)   | 0.006 (5)    |
| C20 | 0.072 (7)  | 0.057 (6)  | 0.061 (6)  | −0.007 (5)   | 0.012 (6)   | 0.013 (5)    |
| C1  | 0.075 (8)  | 0.066 (7)  | 0.082 (9)  | 0.015 (6)    | 0.026 (7)   | 0.019 (6)    |
| C2  | 0.064 (7)  | 0.069 (7)  | 0.066 (7)  | 0.006 (6)    | 0.020 (6)   | 0.022 (6)    |

|     |           |           |           |            |            |            |
|-----|-----------|-----------|-----------|------------|------------|------------|
| C17 | 0.079 (8) | 0.088 (9) | 0.052 (6) | 0.039 (7)  | 0.002 (6)  | 0.016 (6)  |
| C11 | 0.083 (8) | 0.061 (7) | 0.053 (6) | 0.024 (6)  | 0.005 (5)  | 0.000 (5)  |
| C4  | 0.064 (7) | 0.097 (8) | 0.059 (7) | −0.005 (6) | 0.029 (6)  | 0.006 (6)  |
| C12 | 0.063 (7) | 0.059 (6) | 0.050 (6) | 0.023 (5)  | −0.004 (5) | −0.003 (5) |
| C5  | 0.063 (7) | 0.095 (8) | 0.053 (6) | −0.006 (6) | 0.017 (6)  | −0.006 (6) |

*Geometric parameters (Å, °)*

|           |            |              |            |
|-----------|------------|--------------|------------|
| Fe1—N3    | 2.128 (8)  | C8—H00b      | 1.1110     |
| Fe1—N4    | 2.194 (8)  | C3—H00G      | 1.1030     |
| Fe1—N6    | 2.129 (8)  | C3—C4        | 1.390 (14) |
| Fe1—N5    | 2.160 (7)  | C9—H00H      | 1.1030     |
| Fe1—N1    | 2.065 (10) | C9—C10       | 1.407 (14) |
| Fe1—N2    | 2.032 (10) | C15—H00I     | 1.1030     |
| S2—C2     | 1.581 (13) | C15—C16      | 1.374 (14) |
| S1—C1     | 1.660 (14) | C6—H00J      | 1.1030     |
| N3—C7     | 1.330 (11) | C6—C5        | 1.394 (14) |
| N3—C3     | 1.321 (12) | C14—H00c     | 1.1110     |
| N4—C8     | 1.480 (12) | C14—H00d     | 1.1110     |
| N4—C14    | 1.466 (12) | C16—H00L     | 1.1030     |
| N4—C20    | 1.482 (11) | C16—C17      | 1.407 (15) |
| N6—C19    | 1.333 (12) | C18—H00M     | 1.1030     |
| N6—C15    | 1.351 (12) | C18—C17      | 1.378 (15) |
| N5—C13    | 1.317 (11) | C10—H00N     | 1.1030     |
| N5—C9     | 1.310 (11) | C10—C11      | 1.396 (15) |
| C7—C8     | 1.474 (12) | C20—H00e     | 1.1110     |
| C7—C6     | 1.405 (13) | C20—H00f     | 1.1110     |
| N1—C1     | 1.114 (13) | C17—H00R     | 1.1030     |
| N2—C2     | 1.184 (13) | C11—H00S     | 1.1030     |
| C13—C14   | 1.475 (12) | C11—C12      | 1.363 (13) |
| C13—C12   | 1.415 (12) | C4—H00T      | 1.1030     |
| C19—C18   | 1.407 (13) | C4—C5        | 1.356 (15) |
| C19—C20   | 1.480 (13) | C12—H00U     | 1.1030     |
| C8—H00a   | 1.1110     | C5—H00V      | 1.1030     |
| N4—Fe1—N3 | 79.1 (3)   | C4—C3—H00G   | 118.3 (7)  |
| N6—Fe1—N3 | 83.9 (3)   | H00H—C9—N5   | 118.3 (6)  |
| N6—Fe1—N4 | 77.0 (3)   | C10—C9—N5    | 123.4 (10) |
| N5—Fe1—N3 | 91.2 (3)   | C10—C9—H00H  | 118.3 (7)  |
| N5—Fe1—N4 | 77.1 (3)   | H00I—C15—N6  | 119.3 (6)  |
| N5—Fe1—N6 | 154.1 (3)  | C16—C15—N6   | 121.4 (10) |
| N1—Fe1—N3 | 169.9 (3)  | C16—C15—H00I | 119.3 (6)  |
| N1—Fe1—N4 | 91.8 (3)   | H00J—C6—C7   | 121.3 (6)  |
| N1—Fe1—N6 | 89.9 (3)   | C5—C6—C7     | 117.4 (10) |
| N1—Fe1—N5 | 91.0 (3)   | C5—C6—H00J   | 121.3 (6)  |
| N2—Fe1—N3 | 94.7 (3)   | C13—C14—N4   | 110.3 (8)  |
| N2—Fe1—N4 | 173.0 (3)  | H00c—C14—N4  | 109.6 (5)  |
| N2—Fe1—N6 | 105.7 (3)  | H00c—C14—C13 | 109.6 (5)  |

|                |            |                 |            |
|----------------|------------|-----------------|------------|
| N2—Fe1—N5      | 100.1 (3)  | H00d—C14—N4     | 109.6 (5)  |
| N2—Fe1—N1      | 94.6 (4)   | H00d—C14—C13    | 109.6 (5)  |
| C7—N3—Fe1      | 116.2 (6)  | H00d—C14—H00c   | 108.1      |
| C3—N3—Fe1      | 125.2 (7)  | H00L—C16—C15    | 119.6 (6)  |
| C3—N3—C7       | 118.4 (9)  | C17—C16—C15     | 120.7 (9)  |
| C8—N4—Fe1      | 110.4 (5)  | C17—C16—H00L    | 119.6 (6)  |
| C14—N4—Fe1     | 104.7 (6)  | H00M—C18—C19    | 120.8 (6)  |
| C14—N4—C8      | 113.3 (7)  | C17—C18—C19     | 118.5 (10) |
| C20—N4—Fe1     | 104.2 (6)  | C17—C18—H00M    | 120.8 (7)  |
| C20—N4—C8      | 113.8 (8)  | H00N—C10—C9     | 122.2 (7)  |
| C20—N4—C14     | 109.7 (7)  | C11—C10—C9      | 115.6 (9)  |
| C19—N6—Fe1     | 114.7 (6)  | C11—C10—H00N    | 122.2 (6)  |
| C15—N6—Fe1     | 126.8 (7)  | C19—C20—N4      | 110.6 (8)  |
| C15—N6—C19     | 118.5 (8)  | H00e—C20—N4     | 109.5 (5)  |
| C13—N5—Fe1     | 113.2 (6)  | H00e—C20—C19    | 109.5 (5)  |
| C9—N5—Fe1      | 125.6 (7)  | H00f—C20—N4     | 109.5 (5)  |
| C9—N5—C13      | 121.2 (8)  | H00f—C20—C19    | 109.5 (5)  |
| C8—C7—N3       | 118.8 (8)  | H00f—C20—H00e   | 108.1      |
| C6—C7—N3       | 122.4 (9)  | N1—C1—S1        | 178.3 (12) |
| C6—C7—C8       | 118.7 (9)  | N2—C2—S2        | 178.2 (10) |
| C1—N1—Fe1      | 169.2 (10) | C18—C17—C16     | 117.6 (10) |
| C2—N2—Fe1      | 174.7 (9)  | H00R—C17—C16    | 121.2 (6)  |
| C14—C13—N5     | 117.4 (8)  | H00R—C17—C18    | 121.2 (7)  |
| C12—C13—N5     | 119.9 (8)  | H00S—C11—C10    | 119.7 (6)  |
| C12—C13—C14    | 122.6 (9)  | C12—C11—C10     | 120.7 (10) |
| C18—C19—N6     | 123.2 (9)  | C12—C11—H00S    | 119.7 (7)  |
| C20—C19—N6     | 116.0 (8)  | H00T—C4—C3      | 120.8 (7)  |
| C20—C19—C18    | 120.8 (9)  | C5—C4—C3        | 118.3 (10) |
| C7—C8—N4       | 115.1 (8)  | C5—C4—H00T      | 120.8 (7)  |
| H00a—C8—N4     | 108.5 (5)  | C11—C12—C13     | 119.3 (10) |
| H00a—C8—C7     | 108.5 (5)  | H00U—C12—C13    | 120.4 (6)  |
| H00b—C8—N4     | 108.5 (5)  | H00U—C12—C11    | 120.4 (7)  |
| H00b—C8—C7     | 108.5 (5)  | C4—C5—C6        | 120.0 (10) |
| H00b—C8—H00a   | 107.5      | H00V—C5—C6      | 120.0 (6)  |
| H00G—C3—N3     | 118.3 (6)  | H00V—C5—C4      | 120.0 (7)  |
| C4—C3—N3       | 123.4 (10) |                 |            |
| Fe1—N3—C7—C8   | −4.6 (7)   | C7—N3—C3—C4     | 1.0 (10)   |
| Fe1—N3—C7—C6   | 173.7 (6)  | C7—C8—N4—C14    | −123.6 (8) |
| Fe1—N3—C3—C4   | −174.1 (8) | C7—C8—N4—C20    | 110.3 (8)  |
| Fe1—N4—C8—C7   | −6.5 (6)   | C7—C6—C5—C4     | −2.7 (11)  |
| Fe1—N4—C14—C13 | −44.5 (5)  | C13—N5—C9—C10   | −0.5 (10)  |
| Fe1—N4—C20—C19 | 44.1 (6)   | C13—C14—N4—C8   | 75.8 (8)   |
| Fe1—N6—C19—C18 | −179.4 (6) | C13—C14—N4—C20  | −155.8 (8) |
| Fe1—N6—C19—C20 | 1.4 (7)    | C13—C12—C11—C10 | −0.2 (11)  |
| Fe1—N6—C15—C16 | −179.4 (8) | C19—N6—C15—C16  | −3.5 (10)  |
| Fe1—N5—C13—C14 | −6.4 (7)   | C19—C18—C17—C16 | 3.5 (11)   |
| Fe1—N5—C13—C12 | 178.5 (6)  | C19—C20—N4—C8   | −76.2 (9)  |

|                |            |                 |             |
|----------------|------------|-----------------|-------------|
| Fe1—N5—C9—C10  | −178.2 (8) | C19—C20—N4—C14  | 155.7 (8)   |
| N3—C7—C8—N4    | 7.7 (9)    | C8—C7—N3—C3     | 179.8 (9)   |
| N3—C7—C6—C5    | 2.7 (10)   | C8—C7—C6—C5     | −178.9 (9)  |
| N3—C3—C4—C5    | −1.1 (12)  | C3—N3—C7—C6     | −1.8 (10)   |
| N4—C8—C7—C6    | −170.8 (8) | C3—C4—C5—C6     | 2.0 (12)    |
| N4—C14—C13—N5  | 36.0 (9)   | C9—N5—C13—C14   | 175.6 (9)   |
| N4—C14—C13—C12 | −149.0 (7) | C9—N5—C13—C12   | 0.5 (10)    |
| N4—C20—C19—N6  | −32.3 (10) | C9—C10—C11—C12  | 0.3 (11)    |
| N4—C20—C19—C18 | 148.5 (8)  | C15—N6—C19—C18  | 4.3 (11)    |
| N6—C19—C18—C17 | −4.4 (11)  | C15—N6—C19—C20  | −174.9 (9)  |
| N6—C15—C16—C17 | 3.0 (12)   | C15—C16—C17—C18 | −3.0 (12)   |
| N5—C13—C12—C11 | −0.2 (10)  | C14—C13—C12—C11 | −175.0 (10) |
| N5—C9—C10—C11  | 0.0 (11)   | C20—C19—C18—C17 | 174.8 (10)  |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) iodobenzene monosolvate (I\_160K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 15.3161$  (11) Å

$b = 12.2026$  (7) Å

$c = 15.4429$  (11) Å

$\beta = 110.918$  (8)°

$V = 2696.0$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 1329.481$

$D_x = 1.642$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 10309 reflections

$\theta = 2.2$ – $24.2^\circ$

$\mu = 1.89$  mm<sup>−1</sup>

$T = 160$  K

Plate, yellow

$0.18 \times 0.1 \times 0.03$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.718$ ,  $T_{\max} = 1.000$

42395 measured reflections

4771 independent reflections

3574 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.056$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -18 \rightarrow 18$

$k = -14 \rightarrow 14$

$l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.130$

$S = 1.01$

4771 reflections

272 parameters

0 restraints

33 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 3.1937P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.0003$

$\Delta\rho_{\max} = 0.80$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.87$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | $x$         | $y$         | $z$         | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Fe1 | 0.65484 (3) | 0.32633 (4) | 0.30691 (3) | 0.04497 (15)                     |

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|      |              |              |              |              |
|------|--------------|--------------|--------------|--------------|
| S2   | 0.47479 (17) | 0.0081 (2)   | 0.1727 (2)   | 0.0715 (4)   |
| S1   | 0.75154 (12) | 0.32371 (13) | 0.04114 (10) | 0.1028 (5)   |
| N3   | 0.60929 (19) | 0.3409 (2)   | 0.42387 (18) | 0.0428 (6)   |
| N6   | 0.5673 (2)   | 0.4695 (2)   | 0.25601 (18) | 0.0452 (7)   |
| N4   | 0.74349 (19) | 0.4625 (2)   | 0.38373 (18) | 0.0418 (6)   |
| N5   | 0.78251 (19) | 0.2452 (2)   | 0.39199 (18) | 0.0432 (7)   |
| N1   | 0.7046 (2)   | 0.3424 (3)   | 0.1981 (2)   | 0.0578 (8)   |
| N2   | 0.5730 (2)   | 0.1928 (3)   | 0.2516 (2)   | 0.0614 (9)   |
| C7   | 0.6501 (2)   | 0.4199 (3)   | 0.4850 (2)   | 0.0418 (8)   |
| C19  | 0.6130 (2)   | 0.5657 (3)   | 0.2736 (2)   | 0.0449 (8)   |
| C13  | 0.8555 (2)   | 0.3117 (3)   | 0.4289 (2)   | 0.0424 (8)   |
| C18  | 0.5675 (3)   | 0.6645 (3)   | 0.2456 (3)   | 0.0549 (9)   |
| C8   | 0.7291 (2)   | 0.4827 (3)   | 0.4723 (2)   | 0.0508 (9)   |
| C14  | 0.8404 (2)   | 0.4295 (3)   | 0.3998 (3)   | 0.0463 (8)   |
| C10  | 0.8784 (3)   | 0.0925 (3)   | 0.4679 (3)   | 0.0604 (10)  |
| C3   | 0.5406 (2)   | 0.2841 (3)   | 0.4356 (3)   | 0.0522 (9)   |
| C9   | 0.7936 (3)   | 0.1379 (3)   | 0.4108 (2)   | 0.0514 (9)   |
| C6   | 0.6226 (3)   | 0.4434 (3)   | 0.5600 (2)   | 0.0537 (9)   |
| C2   | 0.5318 (3)   | 0.1143 (4)   | 0.2179 (3)   | 0.0539 (9)   |
| C15  | 0.4750 (3)   | 0.4694 (3)   | 0.2113 (2)   | 0.0552 (10)  |
| C1   | 0.7241 (3)   | 0.3357 (3)   | 0.1325 (3)   | 0.0580 (10)  |
| C16  | 0.4250 (3)   | 0.5649 (4)   | 0.1800 (3)   | 0.0631 (11)  |
| C20  | 0.7171 (2)   | 0.5577 (3)   | 0.3209 (3)   | 0.0494 (9)   |
| C12  | 0.9428 (3)   | 0.2735 (3)   | 0.4870 (2)   | 0.0507 (9)   |
| C11  | 0.9536 (3)   | 0.1618 (3)   | 0.5060 (3)   | 0.0603 (10)  |
| C17  | 0.4721 (3)   | 0.6645 (3)   | 0.1963 (3)   | 0.0617 (11)  |
| C4   | 0.5092 (3)   | 0.3026 (4)   | 0.5077 (3)   | 0.0617 (10)  |
| C5   | 0.5510 (3)   | 0.3830 (4)   | 0.5711 (3)   | 0.0618 (10)  |
| H00J | 0.5070 (2)   | 0.2188 (3)   | 0.3853 (3)   | 0.0626 (11)* |
| H00U | 0.4513 (3)   | 0.2536 (4)   | 0.5143 (3)   | 0.0741 (12)* |
| H00V | 0.5278 (3)   | 0.3990 (4)   | 0.6299 (3)   | 0.0742 (12)* |
| H00L | 0.6574 (3)   | 0.5090 (3)   | 0.6094 (2)   | 0.0644 (11)* |
| H00N | 0.4373 (3)   | 0.3906 (3)   | 0.1988 (2)   | 0.0663 (12)* |
| H00P | 0.3488 (3)   | 0.5621 (4)   | 0.1427 (3)   | 0.0757 (13)* |
| H00T | 0.4345 (3)   | 0.7418 (3)   | 0.1705 (3)   | 0.0740 (13)* |
| H00F | 0.6066 (3)   | 0.7423 (3)   | 0.2623 (3)   | 0.0659 (11)* |
| H00K | 0.7332 (3)   | 0.0830 (3)   | 0.3800 (2)   | 0.0617 (11)* |
| H00I | 0.8851 (3)   | 0.0037 (3)   | 0.4820 (3)   | 0.0725 (12)* |
| H00S | 1.0217 (3)   | 0.1289 (3)   | 0.5512 (3)   | 0.0724 (12)* |
| H00R | 1.0017 (3)   | 0.3306 (3)   | 0.5171 (2)   | 0.0608 (11)* |
| H00c | 0.8555 (2)   | 0.4413 (3)   | 0.3352 (3)   | 0.0556 (10)* |
| H00d | 0.8892 (2)   | 0.4820 (3)   | 0.4547 (3)   | 0.0556 (10)* |
| H00a | 0.7947 (2)   | 0.4618 (3)   | 0.5303 (2)   | 0.0609 (10)* |
| H00b | 0.7162 (2)   | 0.5716 (3)   | 0.4774 (2)   | 0.0609 (10)* |
| H00e | 0.7437 (2)   | 0.6338 (3)   | 0.3611 (3)   | 0.0592 (11)* |
| H00g | 0.7502 (2)   | 0.5502 (3)   | 0.2678 (3)   | 0.0592 (11)* |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Fe1 | 0.0430 (3)  | 0.0481 (3)  | 0.0397 (3)  | 0.0020 (2)   | 0.0097 (2)   | 0.0095 (2)   |
| S2  | 0.0562 (6)  | 0.0765 (8)  | 0.0881 (9)  | −0.0108 (6)  | 0.0335 (6)   | −0.0114 (7)  |
| S1  | 0.1426 (13) | 0.1101 (11) | 0.0693 (8)  | 0.0635 (10)  | 0.0545 (9)   | 0.0419 (8)   |
| N3  | 0.0431 (15) | 0.0470 (16) | 0.0355 (14) | −0.0041 (13) | 0.0107 (12)  | −0.0040 (12) |
| N6  | 0.0492 (17) | 0.0429 (17) | 0.0355 (15) | 0.0023 (13)  | 0.0052 (13)  | −0.0019 (12) |
| N4  | 0.0439 (15) | 0.0379 (15) | 0.0378 (15) | −0.0038 (12) | 0.0075 (12)  | 0.0061 (12)  |
| N5  | 0.0494 (17) | 0.0411 (16) | 0.0331 (14) | −0.0023 (13) | 0.0072 (12)  | 0.0036 (12)  |
| N1  | 0.063 (2)   | 0.0523 (19) | 0.057 (2)   | 0.0085 (15)  | 0.0204 (17)  | 0.0121 (16)  |
| N2  | 0.056 (2)   | 0.072 (2)   | 0.0525 (19) | −0.0029 (18) | 0.0151 (16)  | 0.0025 (17)  |
| C7  | 0.0401 (18) | 0.0463 (19) | 0.0340 (17) | 0.0021 (15)  | 0.0071 (14)  | 0.0001 (14)  |
| C19 | 0.052 (2)   | 0.0382 (19) | 0.0377 (18) | 0.0023 (16)  | 0.0075 (15)  | 0.0049 (14)  |
| C13 | 0.0416 (19) | 0.0413 (19) | 0.0390 (17) | −0.0039 (15) | 0.0080 (15)  | 0.0007 (14)  |
| C18 | 0.064 (2)   | 0.042 (2)   | 0.051 (2)   | 0.0100 (18)  | 0.0125 (18)  | 0.0073 (16)  |
| C8  | 0.051 (2)   | 0.052 (2)   | 0.044 (2)   | −0.0070 (17) | 0.0107 (16)  | −0.0051 (16) |
| C14 | 0.0417 (19) | 0.0437 (19) | 0.050 (2)   | −0.0081 (15) | 0.0121 (16)  | 0.0039 (16)  |
| C10 | 0.085 (3)   | 0.041 (2)   | 0.046 (2)   | 0.009 (2)    | 0.012 (2)    | 0.0043 (17)  |
| C3  | 0.046 (2)   | 0.062 (2)   | 0.048 (2)   | −0.0132 (18) | 0.0160 (17)  | −0.0047 (17) |
| C9  | 0.071 (3)   | 0.040 (2)   | 0.0404 (19) | −0.0070 (18) | 0.0157 (18)  | −0.0017 (15) |
| C6  | 0.053 (2)   | 0.061 (2)   | 0.043 (2)   | 0.0011 (18)  | 0.0127 (17)  | −0.0063 (17) |
| C2  | 0.047 (2)   | 0.066 (3)   | 0.048 (2)   | −0.006 (2)   | 0.0168 (17)  | −0.0035 (19) |
| C15 | 0.049 (2)   | 0.060 (2)   | 0.042 (2)   | 0.0035 (18)  | −0.0022 (16) | −0.0096 (17) |
| C1  | 0.064 (2)   | 0.054 (2)   | 0.050 (2)   | 0.0138 (19)  | 0.013 (2)    | 0.0165 (19)  |
| C16 | 0.056 (2)   | 0.074 (3)   | 0.044 (2)   | 0.016 (2)    | −0.0004 (18) | −0.0056 (19) |
| C20 | 0.051 (2)   | 0.0390 (19) | 0.051 (2)   | −0.0063 (16) | 0.0100 (17)  | 0.0069 (15)  |
| C12 | 0.049 (2)   | 0.051 (2)   | 0.045 (2)   | 0.0085 (17)  | 0.0090 (16)  | −0.0003 (16) |
| C11 | 0.066 (3)   | 0.056 (2)   | 0.048 (2)   | 0.016 (2)    | 0.0069 (19)  | −0.0022 (18) |
| C17 | 0.064 (3)   | 0.063 (3)   | 0.047 (2)   | 0.019 (2)    | 0.0066 (19)  | 0.0053 (19)  |
| C4  | 0.054 (2)   | 0.072 (3)   | 0.066 (3)   | −0.005 (2)   | 0.031 (2)    | 0.002 (2)    |
| C5  | 0.061 (2)   | 0.078 (3)   | 0.053 (2)   | 0.003 (2)    | 0.028 (2)    | −0.002 (2)   |

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

|        |           |          |           |
|--------|-----------|----------|-----------|
| Fe1—N3 | 2.163 (3) | C18—H00F | 1.1030    |
| Fe1—N6 | 2.172 (3) | C8—H00a  | 1.1110    |
| Fe1—N4 | 2.205 (3) | C8—H00b  | 1.1110    |
| Fe1—N5 | 2.167 (3) | C14—H00c | 1.1110    |
| Fe1—N1 | 2.088 (4) | C14—H00d | 1.1110    |
| Fe1—N2 | 2.048 (4) | C10—C9   | 1.398 (5) |
| S2—C2  | 1.580 (5) | C10—C11  | 1.379 (6) |
| S1—C1  | 1.616 (5) | C10—H00I | 1.1030    |
| N3—C7  | 1.338 (4) | C3—C4    | 1.381 (5) |
| N3—C3  | 1.325 (4) | C3—H00J  | 1.1030    |
| N6—C19 | 1.344 (4) | C9—H00K  | 1.1030    |
| N6—C15 | 1.333 (4) | C6—C5    | 1.384 (5) |
| N4—C8  | 1.482 (4) | C6—H00L  | 1.1030    |

|            |             |               |             |
|------------|-------------|---------------|-------------|
| N4—C14     | 1.471 (4)   | C15—C16       | 1.384 (5)   |
| N4—C20     | 1.474 (4)   | C15—H00N      | 1.1030      |
| N5—C13     | 1.334 (4)   | C16—C17       | 1.390 (6)   |
| N5—C9      | 1.339 (4)   | C16—H00P      | 1.1030      |
| N1—C1      | 1.156 (5)   | C20—H00e      | 1.1110      |
| N2—C2      | 1.162 (5)   | C20—H00g      | 1.1110      |
| C7—C8      | 1.503 (5)   | C12—C11       | 1.392 (5)   |
| C7—C6      | 1.396 (5)   | C12—H00R      | 1.1030      |
| C19—C18    | 1.383 (5)   | C11—H00S      | 1.1030      |
| C19—C20    | 1.501 (5)   | C17—H00T      | 1.1030      |
| C13—C14    | 1.499 (5)   | C4—C5         | 1.372 (6)   |
| C13—C12    | 1.397 (5)   | C4—H00U       | 1.1030      |
| C18—C17    | 1.385 (5)   | C5—H00V       | 1.1030      |
| N6—Fe1—N3  | 83.93 (10)  | H00b—C8—H00a  | 107.5       |
| N4—Fe1—N3  | 78.98 (10)  | C13—C14—N4    | 110.6 (3)   |
| N4—Fe1—N6  | 76.72 (10)  | H00c—C14—N4   | 109.53 (17) |
| N5—Fe1—N3  | 91.24 (10)  | H00c—C14—C13  | 109.53 (19) |
| N5—Fe1—N6  | 153.37 (11) | H00d—C14—N4   | 109.53 (18) |
| N5—Fe1—N4  | 76.65 (10)  | H00d—C14—C13  | 109.53 (18) |
| N1—Fe1—N3  | 169.58 (12) | H00d—C14—H00c | 108.1       |
| N1—Fe1—N6  | 89.35 (11)  | C11—C10—C9    | 118.2 (4)   |
| N1—Fe1—N4  | 91.74 (12)  | H00I—C10—C9   | 120.9 (2)   |
| N1—Fe1—N5  | 91.22 (11)  | H00I—C10—C11  | 120.9 (2)   |
| N2—Fe1—N3  | 94.68 (12)  | C4—C3—N3      | 123.1 (4)   |
| N2—Fe1—N6  | 106.40 (12) | H00J—C3—N3    | 118.4 (2)   |
| N2—Fe1—N4  | 172.71 (12) | H00J—C3—C4    | 118.4 (2)   |
| N2—Fe1—N5  | 100.07 (12) | C10—C9—N5     | 122.8 (4)   |
| N2—Fe1—N1  | 94.86 (13)  | H00K—C9—N5    | 118.6 (2)   |
| C7—N3—Fe1  | 116.1 (2)   | H00K—C9—C10   | 118.6 (2)   |
| C3—N3—Fe1  | 125.4 (2)   | C5—C6—C7      | 118.8 (4)   |
| C3—N3—C7   | 118.4 (3)   | H00L—C6—C7    | 120.6 (2)   |
| C19—N6—Fe1 | 114.8 (2)   | H00L—C6—C5    | 120.6 (2)   |
| C15—N6—Fe1 | 126.2 (2)   | N2—C2—S2      | 179.2 (4)   |
| C15—N6—C19 | 119.0 (3)   | C16—C15—N6    | 122.2 (4)   |
| C8—N4—Fe1  | 110.9 (2)   | H00N—C15—N6   | 118.9 (2)   |
| C14—N4—Fe1 | 105.9 (2)   | H00N—C15—C16  | 118.9 (2)   |
| C14—N4—C8  | 111.3 (3)   | N1—C1—S1      | 178.9 (4)   |
| C20—N4—Fe1 | 105.54 (19) | C17—C16—C15   | 119.1 (4)   |
| C20—N4—C8  | 112.4 (3)   | H00P—C16—C15  | 120.4 (2)   |
| C20—N4—C14 | 110.5 (3)   | H00P—C16—C17  | 120.4 (2)   |
| C13—N5—Fe1 | 114.8 (2)   | C19—C20—N4    | 111.5 (3)   |
| C9—N5—Fe1  | 126.6 (2)   | H00e—C20—N4   | 109.33 (18) |
| C9—N5—C13  | 118.7 (3)   | H00e—C20—C19  | 109.33 (19) |
| C1—N1—Fe1  | 168.8 (3)   | H00g—C20—N4   | 109.33 (19) |
| C2—N2—Fe1  | 174.9 (3)   | H00g—C20—C19  | 109.3 (2)   |
| C8—C7—N3   | 118.1 (3)   | H00g—C20—H00e | 108.0       |
| C6—C7—N3   | 122.0 (3)   | C11—C12—C13   | 118.4 (4)   |



|                |             |                 |            |
|----------------|-------------|-----------------|------------|
| C6—C7—C8       | 119.9 (3)   | H00R—C12—C13    | 120.8 (2)  |
| C18—C19—N6     | 122.0 (3)   | H00R—C12—C11    | 120.8 (2)  |
| C20—C19—N6     | 115.3 (3)   | C12—C11—C10     | 119.5 (4)  |
| C20—C19—C18    | 122.6 (3)   | H00S—C11—C10    | 120.3 (2)  |
| C14—C13—N5     | 116.0 (3)   | H00S—C11—C12    | 120.3 (2)  |
| C12—C13—N5     | 122.4 (3)   | C16—C17—C18     | 118.5 (4)  |
| C12—C13—C14    | 121.5 (3)   | H00T—C17—C18    | 120.8 (2)  |
| C17—C18—C19    | 119.1 (4)   | H00T—C17—C16    | 120.8 (2)  |
| H00F—C18—C19   | 120.4 (2)   | C5—C4—C3        | 119.0 (4)  |
| H00F—C18—C17   | 120.4 (2)   | H00U—C4—C3      | 120.5 (2)  |
| C7—C8—N4       | 115.3 (3)   | H00U—C4—C5      | 120.5 (2)  |
| H00a—C8—N4     | 108.45 (18) | C4—C5—C6        | 118.8 (4)  |
| H00a—C8—C7     | 108.45 (18) | H00V—C5—C6      | 120.6 (2)  |
| H00b—C8—N4     | 108.45 (18) | H00V—C5—C4      | 120.6 (2)  |
| H00b—C8—C7     | 108.45 (19) |                 |            |
| Fe1—N3—C7—C8   | −5.9 (3)    | C7—N3—C3—C4     | 0.1 (4)    |
| Fe1—N3—C7—C6   | 175.3 (2)   | C7—C8—N4—C14    | −125.9 (3) |
| Fe1—N3—C3—C4   | −174.6 (3)  | C7—C8—N4—C20    | 109.6 (3)  |
| Fe1—N6—C19—C18 | 179.8 (2)   | C7—C6—C5—C4     | −0.4 (4)   |
| Fe1—N6—C19—C20 | 3.0 (2)     | C19—N6—C15—C16  | 1.9 (4)    |
| Fe1—N6—C15—C16 | −178.9 (3)  | C19—C18—C17—C16 | 2.6 (4)    |
| Fe1—N4—C8—C7   | −8.3 (2)    | C19—C20—N4—C8   | −77.7 (3)  |
| Fe1—N4—C14—C13 | −43.0 (2)   | C19—C20—N4—C14  | 157.3 (3)  |
| Fe1—N4—C20—C19 | 43.2 (2)    | C13—N5—C9—C10   | 0.3 (4)    |
| Fe1—N5—C13—C14 | −4.9 (2)    | C13—C14—N4—C8   | 77.6 (3)   |
| Fe1—N5—C13—C12 | 178.7 (2)   | C13—C14—N4—C20  | −156.8 (3) |
| Fe1—N5—C9—C10  | −178.9 (3)  | C13—C12—C11—C10 | −0.5 (4)   |
| N3—C7—C8—N4    | 9.7 (3)     | C18—C19—N6—C15  | −0.9 (4)   |
| N3—C7—C6—C5    | 0.0 (4)     | C18—C17—C16—C15 | −1.7 (5)   |
| N3—C3—C4—C5    | −0.5 (5)    | C8—C7—N3—C3     | 178.9 (3)  |
| N6—C19—C18—C17 | −1.4 (4)    | C8—C7—C6—C5     | −178.7 (3) |
| N6—C19—C20—N4  | −32.3 (3)   | C14—C13—N5—C9   | 175.8 (3)  |
| N6—C15—C16—C17 | −0.6 (4)    | C14—C13—C12—C11 | −175.5 (4) |
| N4—C8—C7—C6    | −171.5 (3)  | C3—N3—C7—C6     | 0.1 (4)    |
| N4—C14—C13—N5  | 33.5 (3)    | C3—C4—C5—C6     | 0.6 (5)    |
| N4—C14—C13—C12 | −150.1 (3)  | C9—N5—C13—C12   | −0.6 (4)   |
| N4—C20—C19—C18 | 150.8 (3)   | C9—C10—C11—C12  | 0.2 (4)    |
| N5—C13—C12—C11 | 0.7 (4)     | C15—N6—C19—C20  | −177.7 (3) |
| N5—C9—C10—C11  | −0.1 (4)    | C20—C19—C18—C17 | 175.3 (4)  |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) iodobenzene monosolvate (I\_170K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I

$M_r = 666.40$

Monoclinic,  $P2_1/n$

$a = 15.3571$  (5) Å

$b = 12.2072$  (3) Å

$c = 15.4611$  (5) Å

$\beta = 110.885$  (4)°

$V = 2708.01$  (16) Å<sup>3</sup>

$Z = 4$   
 $F(000) = 1329.481$   
 $D_x = 1.635 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 11957 reflections  
 $\theta = 2.2\text{--}25.0^\circ$   
 $\mu = 1.88 \text{ mm}^{-1}$   
 $T = 170 \text{ K}$   
 Plate, yellow  
 $0.17 \times 0.1 \times 0.03 \text{ mm}$

*Data collection*

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)  
 $T_{\min} = 0.781$ ,  $T_{\max} = 1.000$   
 42884 measured reflections  
 4803 independent reflections  
 3777 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.043$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -18 \rightarrow 18$   
 $k = -14 \rightarrow 14$   
 $l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.073$   
 $S = 1.01$   
 4803 reflections  
 282 parameters  
 0 restraints  
 33 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 1.9164P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.007$   
 $\Delta\rho_{\max} = 0.67 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.64 \text{ e \AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.34580 (2)  | 0.32506 (3)  | 0.69406 (2)  | 0.03880 (9)                      |
| S2  | 0.52589 (11) | 0.00588 (13) | 0.82839 (13) | 0.0645 (2)                       |
| S1  | 0.24948 (15) | 0.32347 (17) | 0.95958 (12) | 0.0999 (4)                       |
| N3  | 0.39070 (12) | 0.34079 (15) | 0.57594 (12) | 0.0389 (4)                       |
| N6  | 0.43298 (13) | 0.46954 (15) | 0.74386 (12) | 0.0404 (4)                       |
| N4  | 0.25694 (12) | 0.46236 (14) | 0.61651 (12) | 0.0368 (4)                       |
| N5  | 0.21726 (12) | 0.24420 (14) | 0.60833 (12) | 0.0379 (4)                       |
| N2  | 0.42752 (15) | 0.19089 (19) | 0.74845 (14) | 0.0560 (6)                       |
| N1  | 0.29609 (15) | 0.34192 (16) | 0.80323 (15) | 0.0519 (5)                       |
| C7  | 0.35016 (15) | 0.41977 (18) | 0.51545 (15) | 0.0379 (5)                       |
| C19 | 0.38730 (16) | 0.56551 (18) | 0.72611 (15) | 0.0409 (5)                       |
| C13 | 0.14427 (15) | 0.31134 (18) | 0.57170 (15) | 0.0384 (5)                       |
| C18 | 0.43221 (18) | 0.6648 (2)   | 0.75372 (16) | 0.0496 (6)                       |
| C8  | 0.27114 (16) | 0.4822 (2)   | 0.52803 (16) | 0.0463 (6)                       |
| C9  | 0.20515 (18) | 0.13717 (19) | 0.58899 (16) | 0.0462 (6)                       |
| C14 | 0.16008 (15) | 0.42965 (18) | 0.60028 (17) | 0.0440 (6)                       |
| C10 | 0.12093 (19) | 0.0932 (2)   | 0.53230 (17) | 0.0538 (7)                       |
| C2  | 0.46900 (17) | 0.1129 (2)   | 0.78267 (17) | 0.0481 (6)                       |
| C6  | 0.37735 (17) | 0.4435 (2)   | 0.44079 (17) | 0.0510 (6)                       |
| C15 | 0.52547 (17) | 0.4705 (2)   | 0.78910 (16) | 0.0519 (6)                       |
| C1  | 0.27660 (17) | 0.3349 (2)   | 0.86876 (18) | 0.0509 (6)                       |
| C20 | 0.28341 (16) | 0.55805 (18) | 0.67894 (17) | 0.0468 (6)                       |

|      |               |              |              |             |
|------|---------------|--------------|--------------|-------------|
| C12  | 0.05762 (16)  | 0.2737 (2)   | 0.51324 (16) | 0.0463 (6)  |
| C16  | 0.57470 (19)  | 0.5665 (2)   | 0.81989 (17) | 0.0592 (7)  |
| C3   | 0.46012 (17)  | 0.2834 (2)   | 0.56456 (17) | 0.0487 (6)  |
| C11  | 0.04587 (18)  | 0.1629 (2)   | 0.49424 (17) | 0.0536 (6)  |
| C17  | 0.52732 (19)  | 0.6654 (2)   | 0.80256 (17) | 0.0576 (7)  |
| C4   | 0.49136 (19)  | 0.3018 (2)   | 0.49247 (19) | 0.0598 (7)  |
| C5   | 0.44883 (19)  | 0.3832 (2)   | 0.42905 (19) | 0.0597 (7)  |
| H00R | 0.49382 (17)  | 0.2185 (2)   | 0.61502 (17) | 0.0584 (7)* |
| H00U | 0.54889 (19)  | 0.2529 (2)   | 0.48551 (19) | 0.0717 (9)* |
| H00V | 0.47148 (19)  | 0.3998 (2)   | 0.37017 (19) | 0.0716 (9)* |
| H00L | 0.34257 (17)  | 0.5092 (2)   | 0.39168 (17) | 0.0612 (8)* |
| H00M | 0.56362 (17)  | 0.3921 (2)   | 0.80222 (16) | 0.0623 (8)* |
| H00Q | 0.65064 (19)  | 0.5643 (2)   | 0.85772 (17) | 0.0710 (9)* |
| H00T | 0.56463 (19)  | 0.7431 (2)   | 0.82718 (17) | 0.0691 (8)* |
| H00F | 0.39279 (18)  | 0.7423 (2)   | 0.73706 (16) | 0.0595 (7)* |
| H00H | 0.26485 (18)  | 0.08149 (19) | 0.61953 (16) | 0.0554 (7)* |
| H00J | 0.11386 (19)  | 0.0045 (2)   | 0.51781 (17) | 0.0646 (8)* |
| H00S | −0.02234 (18) | 0.1306 (2)   | 0.44938 (17) | 0.0643 (8)* |
| H00P | −0.00061 (16) | 0.3313 (2)   | 0.48265 (16) | 0.0555 (7)* |
| H00e | 0.25719 (16)  | 0.63401 (18) | 0.63844 (17) | 0.0561 (7)* |
| H00g | 0.25035 (16)  | 0.55126 (18) | 0.73199 (17) | 0.0561 (7)* |
| H00a | 0.20579 (16)  | 0.4608 (2)   | 0.47025 (16) | 0.0556 (7)* |
| H00b | 0.28366 (16)  | 0.5711 (2)   | 0.52261 (16) | 0.0556 (7)* |
| H00c | 0.14477 (15)  | 0.44231 (18) | 0.66459 (17) | 0.0528 (7)* |
| H00d | 0.11174 (15)  | 0.48199 (18) | 0.54517 (17) | 0.0528 (7)* |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.03765 (17) | 0.03971 (18) | 0.03556 (17) | −0.00027 (15) | 0.00880 (13) | −0.00604 (15) |
| S2  | 0.0509 (4)   | 0.0626 (5)   | 0.0863 (6)   | 0.0126 (3)    | 0.0321 (4)   | 0.0158 (4)    |
| S1  | 0.1429 (9)   | 0.1084 (7)   | 0.0660 (5)   | −0.0670 (7)   | 0.0588 (6)   | −0.0423 (5)   |
| N3  | 0.0372 (10)  | 0.0411 (11)  | 0.0363 (10)  | 0.0024 (8)    | 0.0107 (8)   | −0.0006 (9)   |
| N6  | 0.0430 (11)  | 0.0372 (11)  | 0.0343 (10)  | −0.0016 (9)   | 0.0056 (9)   | 0.0020 (8)    |
| N4  | 0.0344 (10)  | 0.0344 (10)  | 0.0378 (10)  | 0.0031 (8)    | 0.0083 (8)   | −0.0029 (8)   |
| N5  | 0.0429 (11)  | 0.0321 (10)  | 0.0340 (10)  | 0.0012 (8)    | 0.0077 (8)   | −0.0030 (8)   |
| N2  | 0.0534 (13)  | 0.0610 (15)  | 0.0479 (12)  | 0.0079 (11)   | 0.0111 (10)  | 0.0030 (11)   |
| N1  | 0.0594 (13)  | 0.0478 (12)  | 0.0508 (13)  | −0.0068 (10)  | 0.0222 (11)  | −0.0107 (10)  |
| C7  | 0.0363 (12)  | 0.0392 (12)  | 0.0329 (12)  | −0.0018 (10)  | 0.0058 (10)  | −0.0002 (10)  |
| C19 | 0.0458 (13)  | 0.0357 (13)  | 0.0358 (12)  | −0.0036 (10)  | 0.0080 (10)  | −0.0043 (10)  |
| C13 | 0.0378 (12)  | 0.0358 (12)  | 0.0372 (12)  | 0.0002 (10)   | 0.0080 (10)  | −0.0025 (10)  |
| C18 | 0.0581 (16)  | 0.0395 (14)  | 0.0465 (14)  | −0.0093 (12)  | 0.0129 (12)  | −0.0095 (11)  |
| C8  | 0.0456 (14)  | 0.0449 (14)  | 0.0430 (14)  | 0.0080 (11)   | 0.0091 (11)  | 0.0042 (11)   |
| C9  | 0.0604 (16)  | 0.0359 (13)  | 0.0374 (13)  | 0.0056 (11)   | 0.0116 (12)  | 0.0026 (10)   |
| C14 | 0.0361 (12)  | 0.0402 (13)  | 0.0529 (15)  | 0.0064 (10)   | 0.0126 (11)  | −0.0051 (11)  |
| C10 | 0.0702 (18)  | 0.0384 (14)  | 0.0439 (14)  | −0.0094 (13)  | 0.0094 (13)  | −0.0025 (11)  |
| C2  | 0.0410 (14)  | 0.0566 (17)  | 0.0467 (14)  | 0.0041 (12)   | 0.0157 (11)  | 0.0046 (13)   |
| C6  | 0.0507 (15)  | 0.0595 (16)  | 0.0407 (14)  | −0.0037 (12)  | 0.0137 (12)  | 0.0068 (12)   |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C15 | 0.0429 (14) | 0.0551 (16) | 0.0462 (14) | −0.0015 (12) | 0.0018 (11)  | 0.0100 (12)  |
| C1  | 0.0552 (15) | 0.0513 (15) | 0.0450 (15) | −0.0136 (12) | 0.0166 (12)  | −0.0141 (12) |
| C20 | 0.0463 (14) | 0.0352 (12) | 0.0530 (15) | 0.0029 (10)  | 0.0107 (12)  | −0.0106 (11) |
| C12 | 0.0410 (13) | 0.0445 (14) | 0.0460 (14) | −0.0066 (11) | 0.0067 (11)  | 0.0003 (11)  |
| C16 | 0.0498 (16) | 0.0679 (19) | 0.0457 (15) | −0.0171 (14) | −0.0003 (12) | 0.0024 (13)  |
| C3  | 0.0445 (14) | 0.0516 (15) | 0.0505 (15) | 0.0082 (11)  | 0.0177 (12)  | −0.0001 (12) |
| C11 | 0.0539 (16) | 0.0502 (16) | 0.0469 (14) | −0.0152 (13) | 0.0060 (12)  | −0.0011 (12) |
| C17 | 0.0597 (17) | 0.0549 (17) | 0.0487 (15) | −0.0215 (14) | 0.0075 (13)  | −0.0078 (13) |
| C4  | 0.0525 (16) | 0.0706 (19) | 0.0635 (18) | 0.0050 (14)  | 0.0296 (14)  | −0.0038 (15) |
| C5  | 0.0587 (17) | 0.0764 (19) | 0.0513 (16) | −0.0025 (15) | 0.0287 (14)  | 0.0019 (14)  |

*Geometric parameters (Å, °)*

|           |             |              |             |
|-----------|-------------|--------------|-------------|
| Fe1—N3    | 2.1783 (19) | C18—H00F     | 1.1030      |
| Fe1—N6    | 2.1820 (18) | C8—H00a      | 1.1110      |
| Fe1—N4    | 2.2232 (18) | C8—H00b      | 1.1110      |
| Fe1—N5    | 2.1824 (18) | C9—C10       | 1.386 (3)   |
| Fe1—N2    | 2.052 (2)   | C9—H00H      | 1.1030      |
| Fe1—N1    | 2.096 (2)   | C14—H00c     | 1.1110      |
| S2—C2     | 1.590 (3)   | C14—H00d     | 1.1110      |
| S1—C1     | 1.607 (3)   | C10—C11      | 1.384 (4)   |
| N3—C7     | 1.333 (3)   | C10—H00J     | 1.1030      |
| N3—C3     | 1.339 (3)   | C6—C5        | 1.386 (4)   |
| N6—C19    | 1.343 (3)   | C6—H00L      | 1.1030      |
| N6—C15    | 1.341 (3)   | C15—C16      | 1.384 (3)   |
| N4—C8     | 1.480 (3)   | C15—H00M     | 1.1030      |
| N4—C14    | 1.473 (3)   | C20—H00e     | 1.1110      |
| N4—C20    | 1.477 (3)   | C20—H00g     | 1.1110      |
| N5—C13    | 1.340 (3)   | C12—C11      | 1.381 (3)   |
| N5—C9     | 1.338 (3)   | C12—H00P     | 1.1030      |
| N2—C2     | 1.163 (3)   | C16—C17      | 1.386 (4)   |
| N1—C1     | 1.157 (3)   | C16—H00Q     | 1.1030      |
| C7—C8     | 1.503 (3)   | C3—C4        | 1.380 (4)   |
| C7—C6     | 1.391 (3)   | C3—H00R      | 1.1030      |
| C19—C18   | 1.385 (3)   | C11—H00S     | 1.1030      |
| C19—C20   | 1.502 (3)   | C17—H00T     | 1.1030      |
| C13—C14   | 1.505 (3)   | C4—C5        | 1.385 (4)   |
| C13—C12   | 1.394 (3)   | C4—H00U      | 1.1030      |
| C18—C17   | 1.385 (3)   | C5—H00V      | 1.1030      |
| N6—Fe1—N3 | 83.50 (7)   | H00b—C8—H00a | 107.5       |
| N4—Fe1—N3 | 78.36 (7)   | C10—C9—N5    | 122.9 (2)   |
| N4—Fe1—N6 | 76.25 (7)   | H00H—C9—N5   | 118.55 (13) |
| N5—Fe1—N3 | 91.08 (7)   | H00H—C9—C10  | 118.55 (15) |
| N5—Fe1—N6 | 152.66 (7)  | C13—C14—N4   | 110.96 (18) |
| N5—Fe1—N4 | 76.41 (6)   | H00c—C14—N4  | 109.45 (12) |
| N2—Fe1—N3 | 94.81 (8)   | H00c—C14—C13 | 109.45 (13) |
| N2—Fe1—N6 | 106.97 (8)  | H00d—C14—N4  | 109.45 (12) |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| N2—Fe1—N4      | 172.19 (8)   | H00d—C14—C13    | 109.45 (12)  |
| N2—Fe1—N5      | 100.17 (8)   | H00d—C14—H00c   | 108.0        |
| N1—Fe1—N3      | 168.96 (7)   | C11—C10—C9      | 118.6 (2)    |
| N1—Fe1—N6      | 89.44 (7)    | H00J—C10—C9     | 120.70 (15)  |
| N1—Fe1—N4      | 91.75 (8)    | H00J—C10—C11    | 120.70 (14)  |
| N1—Fe1—N5      | 91.33 (7)    | N2—C2—S2        | 179.3 (2)    |
| N1—Fe1—N2      | 95.36 (8)    | C5—C6—C7        | 119.0 (2)    |
| C7—N3—Fe1      | 116.35 (15)  | H00L—C6—C7      | 120.51 (14)  |
| C3—N3—Fe1      | 124.67 (16)  | H00L—C6—C5      | 120.51 (16)  |
| C3—N3—C7       | 118.8 (2)    | C16—C15—N6      | 122.3 (2)    |
| C19—N6—Fe1     | 115.03 (14)  | H00M—C15—N6     | 118.87 (14)  |
| C15—N6—Fe1     | 126.41 (16)  | H00M—C15—C16    | 118.87 (16)  |
| C15—N6—C19     | 118.6 (2)    | N1—C1—S1        | 179.2 (2)    |
| C8—N4—Fe1      | 110.95 (13)  | C19—C20—N4      | 111.25 (18)  |
| C14—N4—Fe1     | 106.02 (13)  | H00e—C20—N4     | 109.38 (12)  |
| C14—N4—C8      | 111.08 (18)  | H00e—C20—C19    | 109.38 (13)  |
| C20—N4—Fe1     | 105.71 (13)  | H00g—C20—N4     | 109.38 (13)  |
| C20—N4—C8      | 112.20 (18)  | H00g—C20—C19    | 109.38 (13)  |
| C20—N4—C14     | 110.58 (17)  | H00g—C20—H00e   | 108.0        |
| C13—N5—Fe1     | 114.79 (14)  | C11—C12—C13     | 118.9 (2)    |
| C9—N5—Fe1      | 126.92 (16)  | H00P—C12—C13    | 120.56 (14)  |
| C9—N5—C13      | 118.3 (2)    | H00P—C12—C11    | 120.56 (15)  |
| C2—N2—Fe1      | 174.7 (2)    | C17—C16—C15     | 119.1 (2)    |
| C1—N1—Fe1      | 168.5 (2)    | H00Q—C16—C15    | 120.44 (16)  |
| C8—C7—N3       | 118.2 (2)    | H00Q—C16—C17    | 120.44 (15)  |
| C6—C7—N3       | 122.0 (2)    | C4—C3—N3        | 122.9 (2)    |
| C6—C7—C8       | 119.8 (2)    | H00R—C3—N3      | 118.55 (14)  |
| C18—C19—N6     | 122.3 (2)    | H00R—C3—C4      | 118.55 (16)  |
| C20—C19—N6     | 115.69 (19)  | C12—C11—C10     | 119.1 (2)    |
| C20—C19—C18    | 121.9 (2)    | H00S—C11—C10    | 120.47 (14)  |
| C14—C13—N5     | 116.09 (19)  | H00S—C11—C12    | 120.47 (15)  |
| C12—C13—N5     | 122.3 (2)    | C16—C17—C18     | 118.7 (2)    |
| C12—C13—C14    | 121.6 (2)    | H00T—C17—C18    | 120.65 (15)  |
| C17—C18—C19    | 119.0 (2)    | H00T—C17—C16    | 120.65 (15)  |
| H00F—C18—C19   | 120.49 (14)  | C5—C4—C3        | 118.5 (3)    |
| H00F—C18—C17   | 120.49 (15)  | H00U—C4—C3      | 120.76 (16)  |
| C7—C8—N4       | 115.37 (18)  | H00U—C4—C5      | 120.76 (16)  |
| H00a—C8—N4     | 108.43 (11)  | C4—C5—C6        | 118.9 (2)    |
| H00a—C8—C7     | 108.43 (12)  | H00V—C5—C6      | 120.55 (16)  |
| H00b—C8—N4     | 108.43 (12)  | H00V—C5—C4      | 120.55 (16)  |
| H00b—C8—C7     | 108.43 (13)  |                 |              |
| Fe1—N3—C7—C8   | −6.35 (17)   | C7—N3—C3—C4     | −0.4 (2)     |
| Fe1—N3—C7—C6   | 175.28 (15)  | C7—C8—N4—C14    | −126.33 (19) |
| Fe1—N3—C3—C4   | −174.70 (19) | C7—C8—N4—C20    | 109.3 (2)    |
| Fe1—N6—C19—C18 | 179.44 (16)  | C7—C6—C5—C4     | −0.5 (3)     |
| Fe1—N6—C19—C20 | 2.69 (17)    | C19—N6—C15—C16  | 1.6 (2)      |
| Fe1—N6—C15—C16 | −178.5 (2)   | C19—C18—C17—C16 | 1.7 (3)      |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| Fe1—N4—C8—C7   | −8.66 (14)   | C19—C20—N4—C8   | −78.0 (2)    |
| Fe1—N4—C14—C13 | −42.64 (13)  | C19—C20—N4—C14  | 157.4 (2)    |
| Fe1—N4—C20—C19 | 43.03 (14)   | C13—N5—C9—C10   | 0.6 (2)      |
| Fe1—N5—C13—C14 | −4.08 (16)   | C13—C14—N4—C8   | 78.0 (2)     |
| Fe1—N5—C13—C12 | 178.23 (16)  | C13—C14—N4—C20  | −156.77 (19) |
| Fe1—N5—C9—C10  | −178.87 (19) | C13—C12—C11—C10 | −1.3 (3)     |
| N3—C7—C8—N4    | 10.3 (2)     | C18—C19—N6—C15  | −0.7 (3)     |
| N3—C7—C6—C5    | −0.1 (3)     | C18—C17—C16—C15 | −0.9 (3)     |
| N3—C3—C4—C5    | −0.1 (3)     | C8—C7—N3—C3     | 178.9 (2)    |
| N6—C19—C18—C17 | −1.0 (3)     | C8—C7—C6—C5     | −178.4 (2)   |
| N6—C19—C20—N4  | −32.1 (2)    | C9—N5—C13—C14   | 176.4 (2)    |
| N6—C15—C16—C17 | −0.8 (3)     | C9—N5—C13—C12   | −1.3 (2)     |
| N4—C8—C7—C6    | −171.31 (19) | C9—C10—C11—C12  | 0.6 (3)      |
| N4—C14—C13—N5  | 32.8 (2)     | C14—C13—C12—C11 | −175.9 (2)   |
| N4—C14—C13—C12 | −149.53 (19) | C6—C7—N3—C3     | 0.5 (3)      |
| N4—C20—C19—C18 | 151.16 (19)  | C6—C5—C4—C3     | 0.6 (3)      |
| N5—C13—C12—C11 | 1.7 (3)      | C15—N6—C19—C20  | −177.4 (2)   |
| N5—C9—C10—C11  | −0.3 (3)     | C20—C19—C18—C17 | 175.6 (2)    |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II)–iodobenzene (4/1) (I\_200K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]<sub>1.33</sub>·0.33C<sub>6</sub>H<sub>5</sub>I

$M_r = 616.51$

Monoclinic,  $P2_1/n$

$a = 15.4065$  (6) Å

$b = 12.2289$  (3) Å

$c = 15.5023$  (6) Å

$\beta = 110.846$  (4)°

$V = 2729.52$  (18) Å<sup>3</sup>

$Z = 3$

$F(000) = 954.913$

$D_x = 1.125$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 11403 reflections

$\theta = 2.2$ – $24.9^\circ$

$\mu = 0.72$  mm<sup>−1</sup>

$T = 200$  K

Block, yellow

$0.17 \times 0.09 \times 0.02 \times 0.04$  (radius) mm

## Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: for a sphere

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.892$ ,  $T_{\max} = 0.892$

43400 measured reflections

4841 independent reflections

3722 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -18 \rightarrow 18$

$k = -14 \rightarrow 14$

$l = -18 \rightarrow 18$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.080$

$S = 0.98$

4841 reflections

282 parameters

0 restraints

33 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 1.1665P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.54$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.45$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| Fe1  | 0.65372 (2)  | 0.32324 (2)  | 0.30482 (2)  | 0.04062 (9)                      |
| S2   | 0.47378 (11) | 0.00468 (13) | 0.17127 (13) | 0.0693 (2)                       |
| S1   | 0.74835 (15) | 0.32131 (17) | 0.03888 (12) | 0.1100 (4)                       |
| N3   | 0.60904 (12) | 0.33955 (14) | 0.42357 (12) | 0.0432 (4)                       |
| N6   | 0.56705 (12) | 0.46971 (14) | 0.25586 (12) | 0.0446 (4)                       |
| N4   | 0.74282 (12) | 0.46101 (14) | 0.38261 (12) | 0.0412 (4)                       |
| N5   | 0.78329 (12) | 0.24326 (14) | 0.39122 (11) | 0.0420 (4)                       |
| N2   | 0.57195 (14) | 0.18934 (17) | 0.25126 (14) | 0.0593 (5)                       |
| N1   | 0.70317 (14) | 0.34048 (16) | 0.19508 (14) | 0.0558 (5)                       |
| C7   | 0.65028 (14) | 0.41873 (17) | 0.48393 (14) | 0.0429 (5)                       |
| C19  | 0.61302 (16) | 0.56469 (17) | 0.27406 (15) | 0.0440 (5)                       |
| C13  | 0.85578 (14) | 0.31071 (18) | 0.42743 (14) | 0.0427 (5)                       |
| C18  | 0.56850 (17) | 0.66442 (19) | 0.24740 (16) | 0.0549 (6)                       |
| C9   | 0.79547 (17) | 0.13632 (18) | 0.41096 (15) | 0.0488 (6)                       |
| C8   | 0.72898 (16) | 0.48040 (19) | 0.47098 (16) | 0.0508 (6)                       |
| C14  | 0.83909 (14) | 0.42868 (18) | 0.39817 (17) | 0.0480 (6)                       |
| C2   | 0.53086 (15) | 0.1119 (2)   | 0.21720 (16) | 0.0491 (6)                       |
| C10  | 0.87959 (18) | 0.0928 (2)   | 0.46740 (16) | 0.0577 (6)                       |
| C1   | 0.72187 (18) | 0.3331 (2)   | 0.12968 (17) | 0.0555 (6)                       |
| C15  | 0.47531 (16) | 0.4707 (2)   | 0.21110 (16) | 0.0552 (6)                       |
| C3   | 0.54015 (16) | 0.2834 (2)   | 0.43552 (17) | 0.0539 (6)                       |
| C20  | 0.71658 (15) | 0.55671 (17) | 0.32094 (17) | 0.0507 (6)                       |
| C12  | 0.94201 (16) | 0.2736 (2)   | 0.48568 (16) | 0.0516 (6)                       |
| C11  | 0.95380 (18) | 0.1630 (2)   | 0.50540 (17) | 0.0589 (6)                       |
| C6   | 0.62304 (18) | 0.4427 (2)   | 0.55827 (16) | 0.0580 (6)                       |
| C16  | 0.42642 (18) | 0.5673 (2)   | 0.18098 (17) | 0.0652 (7)                       |
| C17  | 0.47409 (19) | 0.6655 (2)   | 0.19884 (17) | 0.0627 (7)                       |
| C4   | 0.50923 (19) | 0.3024 (2)   | 0.50749 (19) | 0.0665 (7)                       |
| C5   | 0.5515 (2)   | 0.3833 (3)   | 0.56988 (19) | 0.0698 (8)                       |
| H00N | 0.50613 (16) | 0.2186 (2)   | 0.38545 (17) | 0.0646 (7)*                      |
| H00U | 0.45194 (19) | 0.2538 (2)   | 0.51480 (19) | 0.0797 (9)*                      |
| H00V | 0.5287 (2)   | 0.4007 (3)   | 0.62835 (19) | 0.0838 (9)*                      |
| H00R | 0.65799 (18) | 0.5081 (2)   | 0.60731 (16) | 0.0696 (8)*                      |
| H00M | 0.43715 (16) | 0.3925 (2)   | 0.19761 (16) | 0.0662 (7)*                      |
| H00S | 0.35072 (18) | 0.5658 (2)   | 0.14338 (17) | 0.0782 (9)*                      |
| H00T | 0.43727 (19) | 0.7434 (2)   | 0.17468 (17) | 0.0753 (8)*                      |
| H00F | 0.60800 (17) | 0.74159 (19) | 0.26477 (16) | 0.0659 (7)*                      |
| H00G | 0.73600 (17) | 0.08059 (18) | 0.38101 (15) | 0.0586 (7)*                      |
| H00K | 0.88716 (18) | 0.0043 (2)   | 0.48167 (16) | 0.0692 (8)*                      |
| H00Q | 1.02164 (18) | 0.1313 (2)   | 0.55094 (17) | 0.0707 (8)*                      |
| H00P | 1.00002 (16) | 0.3312 (2)   | 0.51566 (16) | 0.0619 (7)*                      |
| H00a | 0.79414 (16) | 0.45850 (19) | 0.52830 (16) | 0.0610 (7)*                      |
| H00b | 0.71700 (16) | 0.56924 (19) | 0.47681 (16) | 0.0610 (7)*                      |
| H00c | 0.85370 (14) | 0.44088 (18) | 0.33363 (17) | 0.0576 (7)*                      |
| H00d | 0.88736 (14) | 0.48152 (18) | 0.45250 (17) | 0.0576 (7)*                      |

|      |              |              |              |             |
|------|--------------|--------------|--------------|-------------|
| H00e | 0.74295 (15) | 0.63226 (17) | 0.36164 (17) | 0.0608 (7)* |
| H00h | 0.74941 (15) | 0.55023 (17) | 0.26790 (17) | 0.0608 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|---------------|--------------|--------------|
| Fe1 | 0.04087 (17) | 0.03969 (17) | 0.03710 (17) | −0.00138 (14) | 0.00873 (13) | 0.00196 (14) |
| S2  | 0.0577 (4)   | 0.0603 (4)   | 0.0965 (6)   | −0.0171 (3)   | 0.0355 (4)   | −0.0223 (4)  |
| S1  | 0.1633 (10)  | 0.1192 (8)   | 0.0696 (5)   | 0.0763 (7)    | 0.0686 (6)   | 0.0436 (5)   |
| N3  | 0.0398 (10)  | 0.0462 (10)  | 0.0420 (10)  | −0.0040 (8)   | 0.0124 (8)   | −0.0002 (8)  |
| N6  | 0.0457 (11)  | 0.0402 (10)  | 0.0399 (10)  | 0.0027 (8)    | 0.0053 (8)   | −0.0002 (8)  |
| N4  | 0.0388 (10)  | 0.0376 (9)   | 0.0426 (10)  | −0.0037 (8)   | 0.0089 (8)   | 0.0041 (8)   |
| N5  | 0.0445 (11)  | 0.0383 (10)  | 0.0382 (10)  | −0.0005 (8)   | 0.0085 (8)   | 0.0024 (8)   |
| N2  | 0.0609 (13)  | 0.0539 (13)  | 0.0568 (13)  | −0.0151 (11)  | 0.0131 (10)  | −0.0089 (10) |
| N1  | 0.0677 (13)  | 0.0555 (12)  | 0.0475 (12)  | 0.0080 (10)   | 0.0246 (11)  | 0.0076 (10)  |
| C7  | 0.0411 (12)  | 0.0448 (12)  | 0.0382 (12)  | 0.0018 (10)   | 0.0086 (10)  | −0.0005 (10) |
| C19 | 0.0497 (13)  | 0.0365 (12)  | 0.0414 (12)  | 0.0043 (10)   | 0.0108 (10)  | 0.0041 (9)   |
| C13 | 0.0385 (12)  | 0.0429 (12)  | 0.0421 (12)  | 0.0004 (10)   | 0.0087 (9)   | 0.0020 (10)  |
| C18 | 0.0626 (16)  | 0.0425 (13)  | 0.0528 (14)  | 0.0124 (11)   | 0.0120 (12)  | 0.0110 (11)  |
| C9  | 0.0598 (15)  | 0.0390 (12)  | 0.0428 (12)  | −0.0003 (11)  | 0.0123 (11)  | −0.0001 (10) |
| C8  | 0.0489 (13)  | 0.0513 (14)  | 0.0473 (13)  | −0.0103 (11)  | 0.0109 (11)  | −0.0079 (11) |
| C14 | 0.0388 (12)  | 0.0426 (12)  | 0.0587 (15)  | −0.0046 (10)  | 0.0126 (11)  | 0.0051 (11)  |
| C2  | 0.0433 (13)  | 0.0529 (15)  | 0.0502 (14)  | −0.0075 (11)  | 0.0157 (11)  | −0.0072 (11) |
| C10 | 0.0707 (17)  | 0.0440 (13)  | 0.0493 (14)  | 0.0111 (12)   | 0.0104 (13)  | 0.0034 (11)  |
| C1  | 0.0657 (16)  | 0.0554 (14)  | 0.0437 (14)  | 0.0187 (12)   | 0.0174 (12)  | 0.0136 (12)  |
| C15 | 0.0459 (14)  | 0.0587 (15)  | 0.0482 (14)  | 0.0042 (12)   | 0.0011 (11)  | −0.0070 (11) |
| C3  | 0.0483 (14)  | 0.0592 (15)  | 0.0559 (15)  | −0.0100 (11)  | 0.0209 (12)  | −0.0016 (12) |
| C20 | 0.0484 (13)  | 0.0378 (12)  | 0.0592 (15)  | −0.0034 (10)  | 0.0108 (12)  | 0.0096 (10)  |
| C12 | 0.0403 (13)  | 0.0529 (14)  | 0.0531 (14)  | 0.0060 (11)   | 0.0061 (11)  | 0.0012 (11)  |
| C11 | 0.0552 (15)  | 0.0556 (16)  | 0.0544 (14)  | 0.0159 (13)   | 0.0054 (12)  | 0.0023 (12)  |
| C6  | 0.0621 (16)  | 0.0673 (16)  | 0.0446 (14)  | 0.0039 (13)   | 0.0190 (12)  | −0.0098 (12) |
| C16 | 0.0540 (16)  | 0.0753 (19)  | 0.0509 (15)  | 0.0157 (14)   | −0.0002 (12) | −0.0012 (13) |
| C17 | 0.0631 (17)  | 0.0588 (17)  | 0.0563 (15)  | 0.0214 (14)   | 0.0090 (13)  | 0.0081 (13)  |
| C4  | 0.0587 (16)  | 0.0787 (19)  | 0.0705 (18)  | −0.0079 (14)  | 0.0335 (14)  | 0.0016 (15)  |
| C5  | 0.0700 (18)  | 0.092 (2)    | 0.0589 (17)  | 0.0020 (16)   | 0.0367 (15)  | −0.0006 (15) |

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

|        |             |          |           |
|--------|-------------|----------|-----------|
| Fe1—N3 | 2.1920 (18) | C18—H00F | 1.1030    |
| Fe1—N6 | 2.2013 (18) | C9—C10   | 1.386 (3) |
| Fe1—N4 | 2.2359 (17) | C9—H00G  | 1.1030    |
| Fe1—N5 | 2.1983 (17) | C8—H00a  | 1.1110    |
| Fe1—N2 | 2.053 (2)   | C8—H00b  | 1.1110    |
| Fe1—N1 | 2.107 (2)   | C14—H00c | 1.1110    |
| S2—C2  | 1.598 (3)   | C14—H00d | 1.1110    |
| S1—C1  | 1.605 (3)   | C10—C11  | 1.383 (3) |
| N3—C7  | 1.339 (3)   | C10—H00K | 1.1030    |
| N3—C3  | 1.331 (3)   | C15—C16  | 1.390 (3) |



|            |             |               |             |
|------------|-------------|---------------|-------------|
| N6—C19     | 1.337 (3)   | C15—H00M      | 1.1030      |
| N6—C15     | 1.335 (3)   | C3—C4         | 1.380 (4)   |
| N4—C8      | 1.479 (3)   | C3—H00N       | 1.1030      |
| N4—C14     | 1.469 (3)   | C20—H00e      | 1.1110      |
| N4—C20     | 1.474 (3)   | C20—H00h      | 1.1110      |
| N5—C13     | 1.340 (3)   | C12—C11       | 1.383 (3)   |
| N5—C9      | 1.341 (3)   | C12—H00P      | 1.1030      |
| N2—C2      | 1.157 (3)   | C11—H00Q      | 1.1030      |
| N1—C1      | 1.152 (3)   | C6—C5         | 1.384 (4)   |
| C7—C8      | 1.501 (3)   | C6—H00R       | 1.1030      |
| C7—C6      | 1.391 (3)   | C16—C17       | 1.383 (4)   |
| C19—C18    | 1.388 (3)   | C16—H00S      | 1.1030      |
| C19—C20    | 1.503 (3)   | C17—H00T      | 1.1030      |
| C13—C14    | 1.507 (3)   | C4—C5         | 1.375 (4)   |
| C13—C12    | 1.390 (3)   | C4—H00U       | 1.1030      |
| C18—C17    | 1.380 (3)   | C5—H00V       | 1.1030      |
|            |             |               |             |
| N6—Fe1—N3  | 83.24 (7)   | H00a—C8—C7    | 108.36 (12) |
| N4—Fe1—N3  | 78.14 (6)   | H00b—C8—N4    | 108.36 (11) |
| N4—Fe1—N6  | 75.73 (6)   | H00b—C8—C7    | 108.36 (12) |
| N5—Fe1—N3  | 90.99 (6)   | H00b—C8—H00a  | 107.4       |
| N5—Fe1—N6  | 151.64 (7)  | C13—C14—N4    | 111.18 (17) |
| N5—Fe1—N4  | 75.91 (6)   | H00c—C14—N4   | 109.40 (12) |
| N2—Fe1—N3  | 94.66 (7)   | H00c—C14—C13  | 109.40 (13) |
| N2—Fe1—N6  | 107.46 (7)  | H00d—C14—N4   | 109.40 (11) |
| N2—Fe1—N4  | 171.86 (7)  | H00d—C14—C13  | 109.40 (12) |
| N2—Fe1—N5  | 100.66 (7)  | H00d—C14—H00c | 108.0       |
| N1—Fe1—N3  | 168.71 (7)  | N2—C2—S2      | 179.3 (2)   |
| N1—Fe1—N6  | 89.40 (7)   | C11—C10—C9    | 118.4 (2)   |
| N1—Fe1—N4  | 91.78 (7)   | H00K—C10—C9   | 120.78 (15) |
| N1—Fe1—N5  | 91.47 (7)   | H00K—C10—C11  | 120.78 (14) |
| N1—Fe1—N2  | 95.71 (8)   | N1—C1—S1      | 179.3 (2)   |
| C7—N3—Fe1  | 116.09 (14) | C16—C15—N6    | 122.0 (2)   |
| C3—N3—Fe1  | 125.05 (16) | H00M—C15—N6   | 119.02 (13) |
| C3—N3—C7   | 118.6 (2)   | H00M—C15—C16  | 119.02 (15) |
| C19—N6—Fe1 | 115.04 (14) | C4—C3—N3      | 123.2 (2)   |
| C15—N6—Fe1 | 125.93 (16) | H00N—C3—N3    | 118.40 (14) |
| C15—N6—C19 | 119.03 (19) | H00N—C3—C4    | 118.40 (16) |
| C8—N4—Fe1  | 110.93 (12) | C19—C20—N4    | 111.37 (18) |
| C14—N4—Fe1 | 106.01 (13) | H00e—C20—N4   | 109.36 (11) |
| C14—N4—C8  | 111.16 (17) | H00e—C20—C19  | 109.36 (12) |
| C20—N4—Fe1 | 105.93 (12) | H00h—C20—N4   | 109.36 (12) |
| C20—N4—C8  | 112.15 (18) | H00h—C20—C19  | 109.36 (13) |
| C20—N4—C14 | 110.37 (17) | H00h—C20—H00e | 108.0       |
| C13—N5—Fe1 | 114.96 (14) | C11—C12—C13   | 118.9 (2)   |
| C9—N5—Fe1  | 126.58 (15) | H00P—C12—C13  | 120.57 (14) |
| C9—N5—C13  | 118.46 (19) | H00P—C12—C11  | 120.57 (14) |
| C2—N2—Fe1  | 174.3 (2)   | C12—C11—C10   | 119.3 (2)   |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C1—N1—Fe1      | 168.0 (2)    | H00Q—C11—C10    | 120.37 (14)  |
| C8—C7—N3       | 118.35 (19)  | H00Q—C11—C12    | 120.37 (14)  |
| C6—C7—N3       | 121.5 (2)    | C5—C6—C7        | 119.1 (2)    |
| C6—C7—C8       | 120.1 (2)    | H00R—C6—C7      | 120.44 (15)  |
| C18—C19—N6     | 122.1 (2)    | H00R—C6—C5      | 120.44 (16)  |
| C20—C19—N6     | 115.92 (18)  | C17—C16—C15     | 119.0 (2)    |
| C20—C19—C18    | 121.9 (2)    | H00S—C16—C15    | 120.48 (15)  |
| C14—C13—N5     | 115.83 (18)  | H00S—C16—C17    | 120.48 (14)  |
| C12—C13—N5     | 122.2 (2)    | C16—C17—C18     | 118.8 (2)    |
| C12—C13—C14    | 122.0 (2)    | H00T—C17—C18    | 120.58 (15)  |
| C17—C18—C19    | 119.0 (2)    | H00T—C17—C16    | 120.58 (14)  |
| H00F—C18—C19   | 120.51 (14)  | C5—C4—C3        | 118.5 (3)    |
| H00F—C18—C17   | 120.51 (15)  | H00U—C4—C3      | 120.77 (16)  |
| C10—C9—N5      | 122.8 (2)    | H00U—C4—C5      | 120.77 (16)  |
| H00G—C9—N5     | 118.60 (13)  | C4—C5—C6        | 119.0 (3)    |
| H00G—C9—C10    | 118.60 (15)  | H00V—C5—C6      | 120.48 (16)  |
| C7—C8—N4       | 115.67 (17)  | H00V—C5—C4      | 120.48 (16)  |
| H00a—C8—N4     | 108.36 (11)  |                 |              |
| Fe1—N3—C7—C8   | −6.29 (16)   | C7—N3—C3—C4     | −0.3 (2)     |
| Fe1—N3—C7—C6   | 175.08 (15)  | C7—C8—N4—C14    | −126.73 (19) |
| Fe1—N3—C3—C4   | −174.55 (19) | C7—C8—N4—C20    | 109.2 (2)    |
| Fe1—N6—C19—C18 | 179.69 (16)  | C7—C6—C5—C4     | −0.1 (3)     |
| Fe1—N6—C19—C20 | 2.67 (16)    | C19—N6—C15—C16  | 1.5 (2)      |
| Fe1—N6—C15—C16 | −178.6 (2)   | C19—C18—C17—C16 | 2.1 (3)      |
| Fe1—N4—C8—C7   | −9.04 (13)   | C19—C20—N4—C8   | −78.1 (2)    |
| Fe1—N4—C14—C13 | −43.28 (13)  | C19—C20—N4—C14  | 157.39 (19)  |
| Fe1—N4—C20—C19 | 43.06 (14)   | C13—N5—C9—C10   | 0.3 (2)      |
| Fe1—N5—C13—C14 | −4.08 (16)   | C13—C14—N4—C8   | 77.4 (2)     |
| Fe1—N5—C13—C12 | 178.20 (16)  | C13—C14—N4—C20  | −157.56 (19) |
| Fe1—N5—C9—C10  | −179.03 (19) | C13—C12—C11—C10 | −0.5 (3)     |
| N3—C7—C8—N4    | 10.5 (2)     | C18—C19—N6—C15  | −0.4 (3)     |
| N3—C7—C6—C5    | −0.1 (3)     | C18—C17—C16—C15 | −1.1 (3)     |
| N3—C3—C4—C5    | 0.1 (3)      | C9—N5—C13—C14   | 176.6 (2)    |
| N6—C19—C18—C17 | −1.4 (3)     | C9—N5—C13—C12   | −1.2 (2)     |
| N6—C19—C20—N4  | −32.1 (2)    | C9—C10—C11—C12  | −0.3 (3)     |
| N6—C15—C16—C17 | −0.8 (3)     | C8—C7—N3—C3     | 179.0 (2)    |
| N4—C8—C7—C6    | −170.82 (19) | C8—C7—C6—C5     | −178.7 (2)   |
| N4—C14—C13—N5  | 33.2 (2)     | C14—C13—C12—C11 | −176.3 (2)   |
| N4—C14—C13—C12 | −149.08 (19) | C15—N6—C19—C20  | −177.4 (2)   |
| N4—C20—C19—C18 | 150.88 (19)  | C3—N3—C7—C6     | 0.3 (2)      |
| N5—C13—C12—C11 | 1.3 (3)      | C3—C4—C5—C6     | 0.1 (3)      |
| N5—C9—C10—C11  | 0.5 (3)      | C20—C19—C18—C17 | 175.4 (2)    |

**Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) iodobenzene monosolvate (I\_230K)***Crystal data*[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>6</sub>H<sub>5</sub>I $M_r = 666.40$ Monoclinic,  $P2_1/n$  $a = 15.4614$  (7) Å $b = 12.2589$  (4) Å $c = 15.5469$  (7) Å $\beta = 110.864$  (5)° $V = 2753.5$  (2) Å<sup>3</sup> $Z = 4$  $F(000) = 1329.481$  $D_x = 1.607$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 10796 reflections

 $\theta = 2.2$ – $24.8^\circ$  $\mu = 1.85$  mm<sup>-1</sup> $T = 230$  K

Plate, yellow

 $0.18 \times 0.1 \times 0.03$  mm*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup> $\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

 $T_{\min} = 0.756$ ,  $T_{\max} = 1.000$ 

43523 measured reflections

4872 independent reflections

3707 reflections with  $I \geq 2\sigma(I)$  $R_{\text{int}} = 0.046$  $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$  $h = -18 \rightarrow 18$  $k = -14 \rightarrow 14$  $l = -18 \rightarrow 18$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$  $wR(F^2) = 0.110$  $S = 1.01$ 

4872 reflections

282 parameters

0 restraints

33 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 1.1461P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = -0.001$  $\Delta\rho_{\max} = 0.74$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.44$  e Å<sup>-3</sup>*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.34609 (3)  | 0.32242 (3)  | 0.69560 (3)  | 0.04610 (12)                     |
| S2  | 0.52608 (14) | 0.00536 (16) | 0.82810 (17) | 0.0790 (3)                       |
| S1  | 0.2537 (2)   | 0.3194 (2)   | 0.96173 (16) | 0.1244 (5)                       |
| N3  | 0.39054 (15) | 0.33896 (17) | 0.57664 (15) | 0.0485 (5)                       |
| N6  | 0.43260 (15) | 0.46889 (18) | 0.74458 (15) | 0.0509 (5)                       |
| N4  | 0.25690 (14) | 0.45953 (17) | 0.61779 (15) | 0.0471 (5)                       |
| N5  | 0.21631 (15) | 0.24251 (17) | 0.60923 (14) | 0.0472 (5)                       |
| N2  | 0.42790 (19) | 0.1895 (2)   | 0.74867 (18) | 0.0673 (7)                       |
| N1  | 0.29699 (18) | 0.3391 (2)   | 0.80542 (18) | 0.0633 (7)                       |
| C7  | 0.34927 (18) | 0.4178 (2)   | 0.51677 (18) | 0.0494 (6)                       |
| C19 | 0.38625 (19) | 0.5635 (2)   | 0.72524 (18) | 0.0503 (6)                       |
| C13 | 0.14475 (18) | 0.3102 (2)   | 0.57299 (18) | 0.0501 (6)                       |
| C18 | 0.4303 (2)   | 0.6634 (2)   | 0.7511 (2)   | 0.0617 (8)                       |
| C9  | 0.2043 (2)   | 0.1364 (2)   | 0.58933 (19) | 0.0559 (7)                       |
| C8  | 0.2702 (2)   | 0.4782 (2)   | 0.5293 (2)   | 0.0589 (7)                       |

|      |              |            |              |              |
|------|--------------|------------|--------------|--------------|
| C14  | 0.16074 (18) | 0.4273 (2) | 0.6022 (2)   | 0.0556 (7)   |
| C2   | 0.4692 (2)   | 0.1125 (2) | 0.7826 (2)   | 0.0567 (7)   |
| C1   | 0.2793 (2)   | 0.3315 (2) | 0.8713 (2)   | 0.0616 (8)   |
| C15  | 0.5238 (2)   | 0.4708 (3) | 0.7887 (2)   | 0.0620 (8)   |
| C10  | 0.1204 (2)   | 0.0922 (3) | 0.5331 (2)   | 0.0654 (8)   |
| C20  | 0.28279 (19) | 0.5553 (2) | 0.6784 (2)   | 0.0587 (8)   |
| C16  | 0.5722 (2)   | 0.5675 (3) | 0.8183 (2)   | 0.0733 (9)   |
| C12  | 0.0585 (2)   | 0.2729 (2) | 0.5152 (2)   | 0.0598 (7)   |
| C3   | 0.4594 (2)   | 0.2842 (3) | 0.5645 (2)   | 0.0624 (8)   |
| C6   | 0.3763 (2)   | 0.4422 (3) | 0.4427 (2)   | 0.0670 (8)   |
| C11  | 0.0463 (2)   | 0.1629 (3) | 0.4952 (2)   | 0.0672 (8)   |
| C17  | 0.5246 (2)   | 0.6651 (3) | 0.7996 (2)   | 0.0714 (9)   |
| C4   | 0.4901 (2)   | 0.3030 (3) | 0.4931 (2)   | 0.0764 (9)   |
| C5   | 0.4476 (3)   | 0.3839 (3) | 0.4312 (3)   | 0.0832 (10)  |
| H00Q | 0.4936 (2)   | 0.2206 (3) | 0.6142 (2)   | 0.0749 (9)*  |
| H00U | 0.5466 (2)   | 0.2550 (3) | 0.4857 (2)   | 0.0917 (11)* |
| H00V | 0.4701 (3)   | 0.4019 (3) | 0.3736 (3)   | 0.0998 (13)* |
| H00R | 0.3414 (2)   | 0.5067 (3) | 0.3943 (2)   | 0.0804 (10)* |
| H00L | 0.5620 (2)   | 0.3938 (3) | 0.8022 (2)   | 0.0744 (9)*  |
| H00O | 0.6469 (2)   | 0.5663 (3) | 0.8558 (2)   | 0.0880 (11)* |
| H00T | 0.5608 (2)   | 0.7423 (3) | 0.8227 (2)   | 0.0857 (11)* |
| H00F | 0.3910 (2)   | 0.7395 (2) | 0.7335 (2)   | 0.0741 (9)*  |
| H00G | 0.2632 (2)   | 0.0815 (2) | 0.61870 (19) | 0.0670 (8)*  |
| H00M | 0.1130 (2)   | 0.0047 (3) | 0.5191 (2)   | 0.0785 (10)* |
| H00S | −0.0208 (2)  | 0.1319 (3) | 0.4501 (2)   | 0.0807 (10)* |
| H00P | 0.0011 (2)   | 0.3299 (2) | 0.4860 (2)   | 0.0718 (9)*  |
| H00a | 0.2060 (2)   | 0.4550 (2) | 0.4730 (2)   | 0.0707 (9)*  |
| H00b | 0.2807 (2)   | 0.5661 (2) | 0.5225 (2)   | 0.0707 (9)*  |
| H00c | 0.14629 (18) | 0.4390 (2) | 0.6660 (2)   | 0.0667 (8)*  |
| H00d | 0.11300 (18) | 0.4795 (2) | 0.5486 (2)   | 0.0667 (8)*  |
| H00e | 0.25667 (19) | 0.6296 (2) | 0.6376 (2)   | 0.0705 (9)*  |
| H00h | 0.25034 (19) | 0.5495 (2) | 0.7308 (2)   | 0.0705 (9)*  |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|-------------|-------------|-------------|--------------|--------------|---------------|
| Fe1 | 0.0475 (2)  | 0.0444 (2)  | 0.0418 (2)  | 0.00245 (17) | 0.01019 (16) | −0.00027 (16) |
| S2  | 0.0671 (5)  | 0.0670 (5)  | 0.1102 (9)  | 0.0215 (4)   | 0.0406 (6)   | 0.0275 (5)    |
| S1  | 0.1879 (13) | 0.1330 (10) | 0.0773 (7)  | −0.0845 (10) | 0.0780 (8)   | −0.0458 (7)   |
| N3  | 0.0462 (12) | 0.0516 (13) | 0.0451 (12) | 0.0063 (10)  | 0.0129 (10)  | 0.0015 (10)   |
| N6  | 0.0525 (14) | 0.0472 (13) | 0.0453 (12) | −0.0005 (10) | 0.0079 (10)  | 0.0010 (10)   |
| N4  | 0.0442 (12) | 0.0420 (11) | 0.0502 (13) | 0.0034 (9)   | 0.0109 (10)  | −0.0042 (10)  |
| N5  | 0.0515 (13) | 0.0437 (12) | 0.0419 (12) | −0.0007 (10) | 0.0110 (10)  | −0.0041 (9)   |
| N2  | 0.0729 (17) | 0.0577 (16) | 0.0629 (16) | 0.0199 (13)  | 0.0141 (13)  | 0.0149 (13)   |
| N1  | 0.0790 (17) | 0.0639 (16) | 0.0514 (15) | −0.0098 (13) | 0.0285 (14)  | −0.0069 (12)  |
| C7  | 0.0483 (15) | 0.0527 (15) | 0.0432 (15) | −0.0020 (12) | 0.0114 (12)  | −0.0006 (12)  |
| C19 | 0.0557 (16) | 0.0441 (15) | 0.0466 (15) | −0.0026 (12) | 0.0125 (13)  | −0.0045 (12)  |
| C13 | 0.0452 (15) | 0.0496 (15) | 0.0502 (15) | 0.0019 (12)  | 0.0103 (12)  | −0.0026 (12)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C18 | 0.069 (2)   | 0.0487 (17) | 0.0604 (18) | −0.0116 (14) | 0.0145 (15) | −0.0094 (14) |
| C9  | 0.0693 (19) | 0.0442 (15) | 0.0503 (16) | −0.0019 (13) | 0.0165 (14) | −0.0010 (12) |
| C8  | 0.0569 (17) | 0.0598 (17) | 0.0552 (17) | 0.0118 (14)  | 0.0141 (14) | 0.0103 (14)  |
| C14 | 0.0467 (15) | 0.0498 (15) | 0.0666 (19) | 0.0051 (12)  | 0.0158 (14) | −0.0044 (14) |
| C2  | 0.0527 (16) | 0.0576 (18) | 0.0603 (18) | 0.0067 (14)  | 0.0209 (14) | 0.0072 (14)  |
| C1  | 0.076 (2)   | 0.0581 (17) | 0.0501 (18) | −0.0193 (15) | 0.0219 (16) | −0.0132 (14) |
| C15 | 0.0495 (17) | 0.0675 (19) | 0.0553 (17) | −0.0033 (14) | 0.0019 (14) | 0.0038 (15)  |
| C10 | 0.079 (2)   | 0.0503 (17) | 0.0579 (18) | −0.0151 (16) | 0.0129 (16) | −0.0058 (14) |
| C20 | 0.0573 (17) | 0.0435 (15) | 0.0695 (19) | 0.0031 (13)  | 0.0154 (15) | −0.0102 (13) |
| C16 | 0.060 (2)   | 0.083 (2)   | 0.0602 (19) | −0.0163 (18) | 0.0008 (15) | 0.0035 (17)  |
| C12 | 0.0470 (16) | 0.0596 (18) | 0.0629 (18) | −0.0057 (14) | 0.0073 (14) | 0.0019 (15)  |
| C3  | 0.0559 (18) | 0.0685 (19) | 0.0655 (19) | 0.0093 (15)  | 0.0250 (15) | −0.0004 (15) |
| C6  | 0.072 (2)   | 0.079 (2)   | 0.0508 (18) | −0.0025 (17) | 0.0229 (16) | 0.0081 (15)  |
| C11 | 0.0615 (19) | 0.066 (2)   | 0.0615 (19) | −0.0198 (16) | 0.0064 (15) | −0.0033 (15) |
| C17 | 0.074 (2)   | 0.066 (2)   | 0.065 (2)   | −0.0222 (17) | 0.0132 (17) | −0.0086 (16) |
| C4  | 0.070 (2)   | 0.094 (3)   | 0.076 (2)   | 0.0053 (19)  | 0.0390 (18) | −0.003 (2)   |
| C5  | 0.087 (3)   | 0.111 (3)   | 0.067 (2)   | −0.008 (2)   | 0.048 (2)   | 0.000 (2)    |

*Geometric parameters (Å, °)*

|           |           |            |             |
|-----------|-----------|------------|-------------|
| Fe1—N3    | 2.200 (2) | C18—H00F   | 1.0930      |
| Fe1—N6    | 2.206 (2) | C9—C10     | 1.390 (4)   |
| Fe1—N4    | 2.237 (2) | C9—H00G    | 1.0930      |
| Fe1—N5    | 2.207 (2) | C8—H00a    | 1.1010      |
| Fe1—N2    | 2.048 (2) | C8—H00b    | 1.1010      |
| Fe1—N1    | 2.111 (3) | C14—H00c   | 1.1010      |
| S2—C2     | 1.599 (4) | C14—H00d   | 1.1010      |
| S1—C1     | 1.598 (4) | C15—C16    | 1.390 (4)   |
| N3—C7     | 1.336 (3) | C15—H00L   | 1.0930      |
| N3—C3     | 1.327 (3) | C10—C11    | 1.389 (4)   |
| N6—C19    | 1.340 (3) | C10—H00M   | 1.0930      |
| N6—C15    | 1.331 (3) | C20—H00e   | 1.1010      |
| N4—C8     | 1.480 (4) | C20—H00h   | 1.1010      |
| N4—C14    | 1.473 (3) | C16—C17    | 1.380 (5)   |
| N4—C20    | 1.469 (3) | C16—H00O   | 1.0930      |
| N5—C13    | 1.337 (3) | C12—C11    | 1.382 (4)   |
| N5—C9     | 1.335 (3) | C12—H00P   | 1.0930      |
| N2—C2     | 1.156 (4) | C3—C4      | 1.374 (4)   |
| N1—C1     | 1.154 (4) | C3—H00Q    | 1.0930      |
| C7—C8     | 1.500 (4) | C6—C5      | 1.377 (5)   |
| C7—C6     | 1.389 (4) | C6—H00R    | 1.0930      |
| C19—C18   | 1.389 (4) | C11—H00S   | 1.0930      |
| C19—C20   | 1.507 (4) | C17—H00T   | 1.0930      |
| C13—C14   | 1.498 (4) | C4—C5      | 1.376 (5)   |
| C13—C12   | 1.394 (4) | C4—H00U    | 1.0930      |
| C18—C17   | 1.383 (4) | C5—H00V    | 1.0930      |
| N6—Fe1—N3 | 83.26 (8) | H00a—C8—C7 | 108.36 (15) |

|              |             |               |             |
|--------------|-------------|---------------|-------------|
| N4—Fe1—N3    | 78.06 (8)   | H00b—C8—N4    | 108.36 (14) |
| N4—Fe1—N6    | 75.89 (8)   | H00b—C8—C7    | 108.36 (16) |
| N5—Fe1—N3    | 91.03 (8)   | H00b—C8—H00a  | 107.4       |
| N5—Fe1—N6    | 151.57 (8)  | C13—C14—N4    | 110.8 (2)   |
| N5—Fe1—N4    | 75.67 (7)   | H00c—C14—N4   | 109.49 (15) |
| N2—Fe1—N3    | 94.53 (9)   | H00c—C14—C13  | 109.49 (16) |
| N2—Fe1—N6    | 107.29 (10) | H00d—C14—N4   | 109.49 (14) |
| N2—Fe1—N4    | 171.67 (9)  | H00d—C14—C13  | 109.49 (15) |
| N2—Fe1—N5    | 100.90 (9)  | H00d—C14—H00c | 108.1       |
| N1—Fe1—N3    | 168.84 (9)  | N2—C2—S2      | 179.1 (3)   |
| N1—Fe1—N6    | 89.46 (9)   | N1—C1—S1      | 179.1 (3)   |
| N1—Fe1—N4    | 91.99 (9)   | C16—C15—N6    | 122.1 (3)   |
| N1—Fe1—N5    | 91.38 (9)   | H00L—C15—N6   | 118.95 (17) |
| N1—Fe1—N2    | 95.71 (11)  | H00L—C15—C16  | 118.95 (19) |
| C7—N3—Fe1    | 115.95 (18) | C11—C10—C9    | 117.9 (3)   |
| C3—N3—Fe1    | 125.4 (2)   | H00M—C10—C9   | 121.05 (18) |
| C3—N3—C7     | 118.4 (3)   | H00M—C10—C11  | 121.05 (17) |
| C19—N6—Fe1   | 114.64 (17) | C19—C20—N4    | 111.3 (2)   |
| C15—N6—Fe1   | 126.4 (2)   | H00e—C20—N4   | 109.37 (15) |
| C15—N6—C19   | 119.0 (2)   | H00e—C20—C19  | 109.37 (15) |
| C8—N4—Fe1    | 110.87 (15) | H00h—C20—N4   | 109.37 (16) |
| C14—N4—Fe1   | 106.20 (16) | H00h—C20—C19  | 109.37 (16) |
| C14—N4—C8    | 110.8 (2)   | H00h—C20—H00e | 108.0       |
| C20—N4—Fe1   | 106.20 (16) | C17—C16—C15   | 119.1 (3)   |
| C20—N4—C8    | 112.1 (2)   | H00O—C16—C15  | 120.45 (19) |
| C20—N4—C14   | 110.4 (2)   | H00O—C16—C17  | 120.45 (18) |
| C13—N5—Fe1   | 114.67 (17) | C11—C12—C13   | 119.2 (3)   |
| C9—N5—Fe1    | 126.53 (19) | H00P—C12—C13  | 120.39 (17) |
| C9—N5—C13    | 118.8 (2)   | H00P—C12—C11  | 120.39 (18) |
| C2—N2—Fe1    | 174.3 (3)   | C4—C3—N3      | 123.7 (3)   |
| C1—N1—Fe1    | 167.7 (3)   | H00Q—C3—N3    | 118.13 (17) |
| C8—C7—N3     | 118.4 (2)   | H00Q—C3—C4    | 118.1 (2)   |
| C6—C7—N3     | 121.5 (3)   | C5—C6—C7      | 119.2 (3)   |
| C6—C7—C8     | 120.0 (3)   | H00R—C6—C7    | 120.42 (18) |
| C18—C19—N6   | 122.0 (3)   | H00R—C6—C5    | 120.4 (2)   |
| C20—C19—N6   | 116.2 (2)   | C12—C11—C10   | 119.2 (3)   |
| C20—C19—C18  | 121.7 (3)   | H00S—C11—C10  | 120.41 (17) |
| C14—C13—N5   | 116.4 (2)   | H00S—C11—C12  | 120.41 (18) |
| C12—C13—N5   | 121.7 (3)   | C16—C17—C18   | 118.8 (3)   |
| C12—C13—C14  | 121.8 (2)   | H00T—C17—C18  | 120.59 (19) |
| C17—C18—C19  | 118.9 (3)   | H00T—C17—C16  | 120.59 (18) |
| H00F—C18—C19 | 120.55 (17) | C5—C4—C3      | 118.0 (3)   |
| H00F—C18—C17 | 120.55 (19) | H00U—C4—C3    | 121.0 (2)   |
| C10—C9—N5    | 123.2 (3)   | H00U—C4—C5    | 121.0 (2)   |
| H00G—C9—N5   | 118.41 (16) | C4—C5—C6      | 119.2 (3)   |
| H00G—C9—C10  | 118.41 (18) | H00V—C5—C6    | 120.4 (2)   |
| C7—C8—N4     | 115.7 (2)   | H00V—C5—C4    | 120.4 (2)   |
| H00a—C8—N4   | 108.36 (14) |               |             |

|                |              |                 |            |
|----------------|--------------|-----------------|------------|
| Fe1—N3—C7—C8   | −7.0 (2)     | C7—N3—C3—C4     | −0.9 (3)   |
| Fe1—N3—C7—C6   | 174.92 (19)  | C7—C8—N4—C14    | −127.6 (2) |
| Fe1—N3—C3—C4   | −174.5 (2)   | C7—C8—N4—C20    | 108.5 (3)  |
| Fe1—N6—C19—C18 | −179.98 (19) | C7—C6—C5—C4     | 0.2 (4)    |
| Fe1—N6—C19—C20 | 3.3 (2)      | C19—N6—C15—C16  | 2.3 (3)    |
| Fe1—N6—C15—C16 | −178.9 (3)   | C19—C18—C17—C16 | 1.8 (4)    |
| Fe1—N4—C8—C7   | −9.97 (17)   | C19—C20—N4—C8   | −78.5 (3)  |
| Fe1—N4—C14—C13 | −43.50 (16)  | C19—C20—N4—C14  | 157.5 (2)  |
| Fe1—N4—C20—C19 | 42.77 (17)   | C13—N5—C9—C10   | −0.5 (3)   |
| Fe1—N5—C13—C14 | −4.9 (2)     | C13—C14—N4—C8   | 77.0 (3)   |
| Fe1—N5—C13—C12 | 178.6 (2)    | C13—C14—N4—C20  | −158.2 (2) |
| Fe1—N5—C9—C10  | −179.0 (2)   | C13—C12—C11—C10 | −0.1 (4)   |
| N3—C7—C8—N4    | 11.7 (3)     | C18—C19—N6—C15  | −1.1 (3)   |
| N3—C7—C6—C5    | −0.4 (3)     | C18—C17—C16—C15 | −0.6 (4)   |
| N3—C3—C4—C5    | 0.7 (4)      | C9—N5—C13—C14   | 176.5 (3)  |
| N6—C19—C18—C17 | −1.0 (3)     | C9—N5—C13—C12   | −0.1 (3)   |
| N6—C19—C20—N4  | −32.3 (3)    | C9—C10—C11—C12  | −0.5 (4)   |
| N6—C15—C16—C17 | −1.5 (4)     | C8—C7—N3—C3     | 178.7 (3)  |
| N4—C8—C7—C6    | −170.2 (2)   | C8—C7—C6—C5     | −178.4 (3) |
| N4—C14—C13—N5  | 33.8 (3)     | C14—C13—C12—C11 | −176.0 (3) |
| N4—C14—C13—C12 | −149.6 (2)   | C15—N6—C19—C20  | −177.8 (3) |
| N4—C20—C19—C18 | 151.0 (2)    | C20—C19—C18—C17 | 175.6 (3)  |
| N5—C13—C12—C11 | 0.4 (3)      | C3—N3—C7—C6     | 0.7 (3)    |
| N5—C9—C10—C11  | 0.8 (3)      | C3—C4—C5—C6     | −0.3 (4)   |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_095K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.6890$  (9) Å

$b = 11.9899$  (5) Å

$c = 14.8537$  (7) Å

$\beta = 110.623$  (6)°

$V = 2448.4$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.469$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9867 reflections

$\theta = 2.1$ – $30.9$ °

$\mu = 0.82$  mm<sup>−1</sup>

$T = 95$  K

Block, red

$0.21 \times 0.08 \times 0.05$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.815$ ,  $T_{\max} = 1.000$

27140 measured reflections

4997 independent reflections

3900 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 26.4$ °,  $\theta_{\min} = 2.2$ °

$h = -18 \rightarrow 18$

$k = -14 \rightarrow 14$

$l = -18 \rightarrow 18$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$  $wR(F^2) = 0.079$  $S = 1.05$ 

4997 reflections

316 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 2.2665P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = -0.001$  $\Delta\rho_{\max} = 0.69 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.67059 (2)  | 0.36000 (3)  | 0.32185 (2)  | 0.01177 (9)                      |
| S2  | 0.48130 (5)  | 0.03731 (5)  | 0.20003 (5)  | 0.02146 (15)                     |
| S1  | 0.78287 (5)  | 0.36386 (6)  | 0.05697 (4)  | 0.02693 (16)                     |
| N6  | 0.57795 (14) | 0.48101 (15) | 0.26124 (12) | 0.0132 (4)                       |
| N1  | 0.72287 (14) | 0.37096 (16) | 0.21694 (13) | 0.0159 (4)                       |
| N5  | 0.78419 (14) | 0.26873 (16) | 0.39774 (12) | 0.0137 (4)                       |
| N4  | 0.75271 (14) | 0.48654 (15) | 0.39418 (13) | 0.0134 (4)                       |
| N3  | 0.61860 (13) | 0.35697 (16) | 0.42768 (12) | 0.0142 (4)                       |
| N2  | 0.58999 (14) | 0.23421 (16) | 0.25674 (13) | 0.0171 (4)                       |
| C18 | 0.56532 (17) | 0.6791 (2)   | 0.23821 (16) | 0.0167 (5)                       |
| H18 | 0.60045 (17) | 0.7618 (2)   | 0.25135 (16) | 0.0201 (6)*                      |
| C19 | 0.61916 (17) | 0.58330 (19) | 0.27420 (15) | 0.0146 (5)                       |
| C12 | 0.95284 (17) | 0.28236 (19) | 0.49641 (16) | 0.0159 (5)                       |
| H12 | 1.01747 (17) | 0.33504 (19) | 0.53023 (16) | 0.0191 (6)*                      |
| C13 | 0.86525 (17) | 0.33001 (18) | 0.43951 (15) | 0.0140 (5)                       |
| C1  | 0.74727 (17) | 0.36952 (19) | 0.15049 (16) | 0.0157 (5)                       |
| N7  | 0.69871 (17) | 0.1482 (2)   | 0.78453 (16) | 0.0332 (6)                       |
| C15 | 0.48202 (17) | 0.4720 (2)   | 0.21258 (16) | 0.0169 (5)                       |
| H15 | 0.44793 (17) | 0.3888 (2)   | 0.20367 (16) | 0.0203 (6)*                      |
| C9  | 0.78887 (17) | 0.15801 (18) | 0.41270 (15) | 0.0148 (5)                       |
| H9  | 0.72255 (17) | 0.10763 (18) | 0.38002 (15) | 0.0177 (6)*                      |
| C10 | 0.87444 (17) | 0.1047 (2)   | 0.46797 (16) | 0.0171 (5)                       |
| H10 | 0.87589 (17) | 0.0135 (2)   | 0.47813 (16) | 0.0205 (6)*                      |
| C20 | 0.72703 (17) | 0.58413 (18) | 0.32796 (16) | 0.0151 (5)                       |
| H19 | 0.74850 (17) | 0.66270 (18) | 0.36975 (16) | 0.0182 (6)*                      |
| H20 | 0.76642 (17) | 0.57928 (18) | 0.27649 (16) | 0.0182 (6)*                      |
| C14 | 0.85516 (16) | 0.45197 (19) | 0.41679 (17) | 0.0164 (5)                       |
| H14 | 0.87785 (16) | 0.46945 (19) | 0.35431 (17) | 0.0197 (6)*                      |
| H13 | 0.90309 (16) | 0.50028 (19) | 0.47937 (17) | 0.0197 (6)*                      |
| C7  | 0.65472 (17) | 0.43577 (19) | 0.49529 (15) | 0.0155 (5)                       |
| C11 | 0.95810 (18) | 0.16777 (19) | 0.51023 (16) | 0.0171 (5)                       |
| H11 | 1.02713 (18) | 0.12759 (19) | 0.55377 (16) | 0.0205 (6)*                      |
| C16 | 0.42421 (18) | 0.5640 (2)   | 0.17306 (16) | 0.0190 (5)                       |
| H16 | 0.34579 (18) | 0.5538 (2)   | 0.13244 (16) | 0.0228 (6)*                      |
| C8  | 0.73226 (18) | 0.5102 (2)   | 0.48467 (16) | 0.0201 (5)                       |
| H8  | 0.80020 (18) | 0.4987 (2)   | 0.54776 (16) | 0.0241 (6)*                      |



|     |              |            |              |             |
|-----|--------------|------------|--------------|-------------|
| H7  | 0.70917 (18) | 0.5985 (2) | 0.48397 (16) | 0.0241 (6)* |
| C6  | 0.62300 (18) | 0.4480 (2) | 0.57306 (16) | 0.0209 (5)  |
| H6  | 0.65445 (18) | 0.5130 (2) | 0.62778 (16) | 0.0251 (6)* |
| C2  | 0.54538 (17) | 0.1525 (2) | 0.23124 (16) | 0.0163 (5)  |
| C17 | 0.46678 (18) | 0.6691 (2) | 0.18553 (16) | 0.0186 (5)  |
| H17 | 0.42288 (18) | 0.7435 (2) | 0.15402 (16) | 0.0223 (6)* |
| C3  | 0.54924 (17) | 0.2881 (2) | 0.43568 (17) | 0.0194 (5)  |
| H3  | 0.51959 (17) | 0.2231 (2) | 0.38057 (17) | 0.0233 (6)* |
| C4  | 0.51375 (18) | 0.2959 (2) | 0.51045 (17) | 0.0230 (6)  |
| H4  | 0.45657 (18) | 0.2385 (2) | 0.51437 (17) | 0.0276 (7)* |
| C5  | 0.55142 (18) | 0.3771 (2) | 0.58033 (17) | 0.0232 (6)  |
| H5  | 0.52473 (18) | 0.3851 (2) | 0.64076 (17) | 0.0278 (7)* |
| C23 | 0.7842 (2)   | 0.2236 (2) | 0.6548 (2)   | 0.0280 (6)  |
| H23 | 0.8185 (2)   | 0.2532 (2) | 0.6039 (2)   | 0.0337 (7)* |
| C24 | 0.7195 (2)   | 0.1354 (2) | 0.63118 (19) | 0.0292 (6)  |
| H24 | 0.7010 (2)   | 0.0927 (2) | 0.56119 (19) | 0.0351 (7)* |
| C25 | 0.6783 (2)   | 0.1017 (2) | 0.6982 (2)   | 0.0323 (7)  |
| H25 | 0.6257 (2)   | 0.0323 (2) | 0.6785 (2)   | 0.0387 (8)* |
| C22 | 0.8050 (2)   | 0.2740 (2) | 0.7438 (2)   | 0.0292 (6)  |
| H22 | 0.8555 (2)   | 0.3454 (2) | 0.7649 (2)   | 0.0351 (7)* |
| C21 | 0.7611 (2)   | 0.2329 (2) | 0.8061 (2)   | 0.0318 (6)  |
| H21 | 0.7790 (2)   | 0.2727 (2) | 0.8771 (2)   | 0.0382 (8)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.01405 (17) | 0.01139 (16) | 0.00942 (15) | −0.00155 (14) | 0.00358 (12) | −0.00001 (13) |
| S2  | 0.0233 (3)   | 0.0185 (3)   | 0.0237 (3)   | −0.0052 (3)   | 0.0096 (3)   | −0.0044 (3)   |
| S1  | 0.0393 (4)   | 0.0269 (3)   | 0.0186 (3)   | 0.0119 (3)    | 0.0151 (3)   | 0.0062 (3)    |
| N6  | 0.0146 (10)  | 0.0131 (10)  | 0.0107 (9)   | −0.0009 (8)   | 0.0029 (8)   | −0.0011 (8)   |
| N1  | 0.0199 (10)  | 0.0139 (10)  | 0.0150 (10)  | 0.0009 (8)    | 0.0075 (8)   | 0.0018 (8)    |
| N5  | 0.0158 (10)  | 0.0131 (10)  | 0.0117 (9)   | −0.0020 (8)   | 0.0041 (8)   | 0.0006 (8)    |
| N4  | 0.0155 (10)  | 0.0105 (10)  | 0.0129 (9)   | −0.0012 (8)   | 0.0036 (8)   | −0.0003 (7)   |
| N3  | 0.0156 (10)  | 0.0171 (10)  | 0.0102 (9)   | −0.0015 (8)   | 0.0048 (8)   | −0.0007 (8)   |
| N2  | 0.0194 (11)  | 0.0145 (11)  | 0.0152 (10)  | −0.0046 (9)   | 0.0034 (8)   | −0.0018 (8)   |
| C18 | 0.0180 (13)  | 0.0146 (12)  | 0.0165 (11)  | 0.0024 (10)   | 0.0046 (10)  | 0.0030 (9)    |
| C19 | 0.0176 (12)  | 0.0133 (12)  | 0.0119 (10)  | 0.0011 (10)   | 0.0040 (9)   | 0.0008 (9)    |
| C12 | 0.0162 (12)  | 0.0145 (12)  | 0.0154 (11)  | 0.0018 (10)   | 0.0036 (9)   | 0.0007 (9)    |
| C13 | 0.0165 (12)  | 0.0106 (11)  | 0.0142 (11)  | 0.0004 (9)    | 0.0047 (9)   | 0.0007 (9)    |
| C1  | 0.0214 (12)  | 0.0130 (11)  | 0.0122 (11)  | 0.0033 (10)   | 0.0054 (9)   | 0.0015 (9)    |
| N7  | 0.0381 (14)  | 0.0361 (14)  | 0.0305 (13)  | 0.0035 (12)   | 0.0184 (11)  | 0.0062 (11)   |
| C15 | 0.0156 (12)  | 0.0173 (12)  | 0.0145 (11)  | −0.0020 (10)  | 0.0012 (9)   | −0.0026 (9)   |
| C9  | 0.0198 (13)  | 0.0118 (12)  | 0.0115 (10)  | −0.0021 (9)   | 0.0040 (9)   | 0.0005 (9)    |
| C10 | 0.0210 (13)  | 0.0132 (12)  | 0.0159 (11)  | 0.0004 (10)   | 0.0052 (10)  | 0.0021 (9)    |
| C20 | 0.0190 (13)  | 0.0094 (11)  | 0.0160 (11)  | −0.0015 (9)   | 0.0049 (10)  | 0.0018 (9)    |
| C14 | 0.0149 (12)  | 0.0119 (11)  | 0.0197 (12)  | −0.0027 (9)   | 0.0027 (10)  | 0.0006 (9)    |
| C7  | 0.0181 (12)  | 0.0173 (12)  | 0.0100 (10)  | 0.0009 (10)   | 0.0035 (9)   | −0.0002 (9)   |
| C11 | 0.0180 (13)  | 0.0166 (12)  | 0.0147 (11)  | 0.0011 (10)   | 0.0033 (10)  | 0.0018 (9)    |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C16 | 0.0168 (13) | 0.0222 (13) | 0.0146 (11) | 0.0026 (10)  | 0.0012 (10) | 0.0002 (10)  |
| C8  | 0.0237 (14) | 0.0204 (13) | 0.0152 (11) | −0.0067 (11) | 0.0056 (10) | −0.0040 (10) |
| C6  | 0.0251 (14) | 0.0253 (13) | 0.0129 (11) | 0.0009 (11)  | 0.0075 (10) | −0.0028 (10) |
| C2  | 0.0162 (12) | 0.0163 (13) | 0.0151 (11) | 0.0005 (10)  | 0.0039 (9)  | −0.0017 (10) |
| C17 | 0.0191 (13) | 0.0198 (13) | 0.0158 (11) | 0.0036 (10)  | 0.0047 (10) | 0.0033 (9)   |
| C3  | 0.0197 (13) | 0.0231 (13) | 0.0159 (12) | −0.0055 (10) | 0.0068 (10) | −0.0003 (10) |
| C4  | 0.0213 (13) | 0.0311 (15) | 0.0204 (13) | −0.0039 (11) | 0.0120 (11) | 0.0027 (11)  |
| C5  | 0.0236 (13) | 0.0334 (15) | 0.0168 (12) | 0.0006 (11)  | 0.0124 (10) | −0.0013 (11) |
| C23 | 0.0274 (15) | 0.0290 (14) | 0.0321 (15) | 0.0061 (12)  | 0.0159 (12) | 0.0092 (12)  |
| C24 | 0.0338 (15) | 0.0281 (14) | 0.0282 (14) | 0.0067 (13)  | 0.0139 (12) | 0.0036 (12)  |
| C25 | 0.0364 (17) | 0.0260 (15) | 0.0372 (16) | −0.0008 (13) | 0.0163 (13) | 0.0057 (12)  |
| C22 | 0.0259 (15) | 0.0280 (15) | 0.0348 (15) | 0.0028 (12)  | 0.0120 (12) | 0.0036 (12)  |
| C21 | 0.0330 (16) | 0.0345 (16) | 0.0301 (15) | 0.0084 (13)  | 0.0138 (13) | 0.0027 (13)  |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N6  | 1.9760 (19) | C9—C10  | 1.391 (3) |
| Fe1—N1  | 1.9694 (19) | C10—H10 | 1.1030    |
| Fe1—N5  | 1.9818 (19) | C10—C11 | 1.390 (3) |
| Fe1—N4  | 1.9997 (19) | C20—H19 | 1.1110    |
| Fe1—N3  | 1.9751 (18) | C20—H20 | 1.1110    |
| Fe1—N2  | 1.951 (2)   | C14—H14 | 1.1110    |
| S2—C2   | 1.644 (2)   | C14—H13 | 1.1110    |
| S1—C1   | 1.649 (2)   | C7—C8   | 1.498 (3) |
| N6—C19  | 1.351 (3)   | C7—C6   | 1.397 (3) |
| N6—C15  | 1.342 (3)   | C11—H11 | 1.1030    |
| N1—C1   | 1.163 (3)   | C16—H16 | 1.1030    |
| N5—C13  | 1.350 (3)   | C16—C17 | 1.390 (3) |
| N5—C9   | 1.344 (3)   | C8—H8   | 1.1110    |
| N4—C20  | 1.489 (3)   | C8—H7   | 1.1110    |
| N4—C14  | 1.481 (3)   | C6—H6   | 1.1030    |
| N4—C8   | 1.504 (3)   | C6—C5   | 1.385 (4) |
| N3—C7   | 1.344 (3)   | C17—H17 | 1.1030    |
| N3—C3   | 1.349 (3)   | C3—H3   | 1.1030    |
| N2—C2   | 1.165 (3)   | C3—C4   | 1.386 (3) |
| C18—H18 | 1.1030      | C4—H4   | 1.1030    |
| C18—C19 | 1.390 (3)   | C4—C5   | 1.389 (4) |
| C18—C17 | 1.387 (3)   | C5—H5   | 1.1030    |
| C19—C20 | 1.502 (3)   | C23—H23 | 1.1030    |
| C12—H12 | 1.1030      | C23—C24 | 1.382 (4) |
| C12—C13 | 1.389 (3)   | C23—C22 | 1.387 (4) |
| C12—C11 | 1.387 (3)   | C24—H24 | 1.1030    |
| C13—C14 | 1.496 (3)   | C24—C25 | 1.395 (4) |
| N7—C25  | 1.332 (4)   | C25—H25 | 1.1030    |
| N7—C21  | 1.329 (4)   | C22—H22 | 1.1030    |
| C15—H15 | 1.1030      | C22—C21 | 1.391 (4) |
| C15—C16 | 1.390 (3)   | C21—H21 | 1.1030    |
| C9—H9   | 1.1030      |         |           |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N1—Fe1—N6   | 89.33 (8)   | H19—C20—C19 | 109.95 (12) |
| N5—Fe1—N6   | 166.25 (8)  | H20—C20—N4  | 109.95 (12) |
| N5—Fe1—N1   | 90.11 (7)   | H20—C20—C19 | 109.95 (12) |
| N4—Fe1—N6   | 82.87 (7)   | H20—C20—H19 | 108.3       |
| N4—Fe1—N1   | 92.57 (8)   | C13—C14—N4  | 109.68 (18) |
| N4—Fe1—N5   | 83.43 (8)   | H14—C14—N4  | 109.73 (11) |
| N3—Fe1—N6   | 88.79 (8)   | H14—C14—C13 | 109.73 (12) |
| N3—Fe1—N1   | 177.22 (8)  | H13—C14—N4  | 109.73 (11) |
| N3—Fe1—N5   | 91.22 (7)   | H13—C14—C13 | 109.73 (12) |
| N3—Fe1—N4   | 85.15 (8)   | H13—C14—H14 | 108.2       |
| N2—Fe1—N6   | 98.14 (8)   | C8—C7—N3    | 117.0 (2)   |
| N2—Fe1—N1   | 89.83 (8)   | C6—C7—N3    | 122.2 (2)   |
| N2—Fe1—N5   | 95.59 (8)   | C6—C7—C8    | 120.8 (2)   |
| N2—Fe1—N4   | 177.41 (8)  | C10—C11—C12 | 118.5 (2)   |
| N2—Fe1—N3   | 92.47 (8)   | H11—C11—C12 | 120.73 (14) |
| C19—N6—Fe1  | 113.55 (15) | H11—C11—C10 | 120.73 (14) |
| C15—N6—Fe1  | 127.60 (15) | H16—C16—C15 | 120.52 (14) |
| C15—N6—C19  | 118.8 (2)   | C17—C16—C15 | 119.0 (2)   |
| C1—N1—Fe1   | 173.38 (19) | C17—C16—H16 | 120.52 (14) |
| C13—N5—Fe1  | 112.99 (15) | C7—C8—N4    | 112.36 (18) |
| C9—N5—Fe1   | 128.56 (16) | H8—C8—N4    | 109.13 (12) |
| C9—N5—C13   | 118.4 (2)   | H8—C8—C7    | 109.13 (13) |
| C20—N4—Fe1  | 106.07 (13) | H7—C8—N4    | 109.13 (12) |
| C14—N4—Fe1  | 106.38 (13) | H7—C8—C7    | 109.13 (13) |
| C14—N4—C20  | 111.74 (17) | H7—C8—H8    | 107.9       |
| C8—N4—Fe1   | 110.53 (14) | H6—C6—C7    | 120.54 (14) |
| C8—N4—C20   | 111.00 (18) | C5—C6—C7    | 118.9 (2)   |
| C8—N4—C14   | 110.91 (17) | C5—C6—H6    | 120.54 (14) |
| C7—N3—Fe1   | 114.84 (15) | N2—C2—S2    | 177.3 (2)   |
| C3—N3—Fe1   | 126.67 (16) | C16—C17—C18 | 119.0 (2)   |
| C3—N3—C7    | 118.4 (2)   | H17—C17—C18 | 120.52 (14) |
| C2—N2—Fe1   | 170.01 (18) | H17—C17—C16 | 120.52 (14) |
| C19—C18—H18 | 120.49 (14) | H3—C3—N3    | 118.71 (13) |
| C17—C18—H18 | 120.49 (14) | C4—C3—N3    | 122.6 (2)   |
| C17—C18—C19 | 119.0 (2)   | C4—C3—H3    | 118.71 (15) |
| C18—C19—N6  | 122.0 (2)   | H4—C4—C3    | 120.57 (15) |
| C20—C19—N6  | 114.67 (19) | C5—C4—C3    | 118.9 (2)   |
| C20—C19—C18 | 123.3 (2)   | C5—C4—H4    | 120.57 (14) |
| C13—C12—H12 | 120.38 (14) | C4—C5—C6    | 119.0 (2)   |
| C11—C12—H12 | 120.38 (14) | H5—C5—C6    | 120.48 (14) |
| C11—C12—C13 | 119.2 (2)   | H5—C5—C4    | 120.48 (14) |
| C12—C13—N5  | 122.3 (2)   | C24—C23—H23 | 120.77 (16) |
| C14—C13—N5  | 115.3 (2)   | C22—C23—H23 | 120.77 (16) |
| C14—C13—C12 | 122.4 (2)   | C22—C23—C24 | 118.5 (2)   |
| N1—C1—S1    | 178.4 (2)   | H24—C24—C23 | 120.87 (16) |
| C21—N7—C25  | 117.0 (2)   | C25—C24—C23 | 118.3 (3)   |
| H15—C15—N6  | 118.89 (13) | C25—C24—H24 | 120.87 (17) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C16—C15—N6      | 122.2 (2)    | C24—C25—N7      | 124.0 (3)    |
| C16—C15—H15     | 118.89 (14)  | H25—C25—N7      | 118.02 (16)  |
| H9—C9—N5        | 118.84 (13)  | H25—C25—C24     | 118.02 (17)  |
| C10—C9—N5       | 122.3 (2)    | H22—C22—C23     | 120.62 (16)  |
| C10—C9—H9       | 118.84 (14)  | C21—C22—C23     | 118.8 (3)    |
| H10—C10—C9      | 120.40 (14)  | C21—C22—H22     | 120.62 (17)  |
| C11—C10—C9      | 119.2 (2)    | C22—C21—N7      | 123.6 (3)    |
| C11—C10—H10     | 120.40 (14)  | H21—C21—N7      | 118.22 (16)  |
| C19—C20—N4      | 108.68 (18)  | H21—C21—C22     | 118.22 (17)  |
| H19—C20—N4      | 109.95 (11)  |                 |              |
| Fe1—N6—C19—C18  | −178.93 (14) | C19—C18—C17—C16 | 2.1 (3)      |
| Fe1—N6—C19—C20  | 2.49 (15)    | C19—C20—N4—C14  | 154.96 (17)  |
| Fe1—N6—C15—C16  | 179.97 (18)  | C19—C20—N4—C8   | −80.65 (19)  |
| Fe1—N5—C13—C12  | 179.57 (14)  | C12—C13—N5—C9   | 0.5 (3)      |
| Fe1—N5—C13—C14  | −2.92 (16)   | C12—C11—C10—C9  | 0.6 (3)      |
| Fe1—N5—C9—C10   | 179.87 (18)  | C13—N5—C9—C10   | −1.2 (2)     |
| Fe1—N4—C20—C19  | 39.45 (14)   | C13—C12—C11—C10 | −1.3 (2)     |
| Fe1—N4—C14—C13  | −36.92 (14)  | C13—C14—N4—C20  | −152.25 (18) |
| Fe1—N4—C8—C7    | −1.59 (15)   | C13—C14—N4—C8   | 83.3 (2)     |
| Fe1—N3—C7—C8    | −3.24 (18)   | N7—C25—C24—C23  | −1.1 (3)     |
| Fe1—N3—C7—C6    | 177.52 (16)  | N7—C21—C22—C23  | −0.9 (3)     |
| Fe1—N3—C3—C4    | −176.7 (2)   | C15—N6—C19—C20  | −179.0 (2)   |
| N6—C19—C18—C17  | −1.5 (2)     | C9—N5—C13—C14   | 178.0 (2)    |
| N6—C19—C20—N4   | −28.5 (2)    | C20—N4—C8—C7    | 115.82 (19)  |
| N6—C15—C16—C17  | −1.0 (3)     | C20—C19—C18—C17 | 176.9 (2)    |
| N5—C13—C12—C11  | 0.8 (3)      | C14—N4—C8—C7    | −119.33 (19) |
| N5—C13—C14—N4   | 27.2 (2)     | C14—C13—C12—C11 | −176.6 (2)   |
| N5—C9—C10—C11   | 0.7 (3)      | C7—N3—C3—C4     | 0.3 (2)      |
| N4—C20—C19—C18  | 152.91 (17)  | C7—C6—C5—C4     | 0.3 (3)      |
| N4—C14—C13—C12  | −155.25 (17) | C8—C7—N3—C3     | 179.5 (2)    |
| N4—C8—C7—N3     | 3.2 (2)      | C8—C7—C6—C5     | −179.7 (2)   |
| N4—C8—C7—C6     | −177.55 (19) | C6—C7—N3—C3     | 0.2 (3)      |
| N3—C7—C6—C5     | −0.5 (3)     | C6—C5—C4—C3     | 0.2 (3)      |
| N3—C3—C4—C5     | −0.5 (3)     | C24—C23—C22—C21 | 0.8 (3)      |
| C18—C19—N6—C15  | −0.4 (3)     | C24—C25—N7—C21  | 1.1 (3)      |
| C18—C17—C16—C15 | −0.9 (3)     | C25—N7—C21—C22  | −0.1 (3)     |
| C19—N6—C15—C16  | 1.7 (2)      | C25—C24—C23—C22 | 0.1 (3)      |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_115K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r$  = 541.49

Monoclinic,  $P2_1/n$

$a$  = 14.7135 (9) Å

$b$  = 12.0011 (5) Å

$c$  = 14.8597 (8) Å

$\beta$  = 110.561 (7)°

$V$  = 2456.8 (3) Å<sup>3</sup>

$Z$  = 4

$F(000)$  = 1123.013

$D_x$  = 1.464 Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 9693 reflections

$\theta$  = 2.1–30.9°

$\mu = 0.81 \text{ mm}^{-1}$   
 $T = 115 \text{ K}$

Block, red  
 $0.21 \times 0.08 \times 0.05 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.817$ ,  $T_{\max} = 1.000$   
 27020 measured reflections  
 5013 independent reflections  
 3867 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.038$   
 $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -18 \rightarrow 18$   
 $k = -15 \rightarrow 15$   
 $l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.084$   
 $S = 1.06$   
 5013 reflections  
 316 parameters  
 0 restraints

43 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 2.2132P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = -0.001$   
 $\Delta\rho_{\max} = 0.86 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.32940 (2)  | 0.35957 (3)  | 0.67833 (2)  | 0.01381 (9)                      |
| S2  | 0.51857 (5)  | 0.03747 (6)  | 0.80021 (5)  | 0.02486 (16)                     |
| S1  | 0.21794 (6)  | 0.36342 (6)  | 0.94332 (5)  | 0.03148 (17)                     |
| N6  | 0.42177 (14) | 0.48061 (16) | 0.73865 (13) | 0.0155 (4)                       |
| N5  | 0.21559 (14) | 0.26861 (16) | 0.60212 (13) | 0.0157 (4)                       |
| N4  | 0.24731 (14) | 0.48616 (16) | 0.60600 (13) | 0.0155 (4)                       |
| N3  | 0.38131 (14) | 0.35694 (16) | 0.57237 (13) | 0.0163 (4)                       |
| N1  | 0.27712 (14) | 0.37059 (16) | 0.78327 (14) | 0.0188 (4)                       |
| N2  | 0.40967 (15) | 0.23386 (17) | 0.74286 (14) | 0.0193 (4)                       |
| C18 | 0.43470 (18) | 0.6788 (2)   | 0.76140 (17) | 0.0196 (5)                       |
| C19 | 0.38090 (17) | 0.58270 (19) | 0.72544 (16) | 0.0168 (5)                       |
| C12 | 0.04722 (17) | 0.2824 (2)   | 0.50413 (16) | 0.0183 (5)                       |
| C13 | 0.13479 (17) | 0.32959 (19) | 0.56092 (16) | 0.0161 (5)                       |
| C14 | 0.14469 (17) | 0.4518 (2)   | 0.58345 (17) | 0.0191 (5)                       |
| C1  | 0.25296 (18) | 0.36914 (19) | 0.84960 (17) | 0.0193 (5)                       |
| C15 | 0.51749 (18) | 0.4716 (2)   | 0.78726 (16) | 0.0192 (5)                       |
| C20 | 0.27310 (17) | 0.58335 (19) | 0.67173 (16) | 0.0182 (5)                       |
| N7  | 0.30048 (19) | 0.1486 (2)   | 0.21514 (18) | 0.0386 (6)                       |
| C10 | 0.12547 (18) | 0.1050 (2)   | 0.53228 (16) | 0.0201 (5)                       |
| C9  | 0.21105 (18) | 0.15792 (19) | 0.58726 (16) | 0.0182 (5)                       |
| C7  | 0.34522 (17) | 0.4355 (2)   | 0.50479 (16) | 0.0179 (5)                       |
| C11 | 0.04220 (19) | 0.1677 (2)   | 0.49020 (17) | 0.0204 (5)                       |
| C2  | 0.45446 (17) | 0.1522 (2)   | 0.76891 (16) | 0.0182 (5)                       |
| C6  | 0.37694 (19) | 0.4478 (2)   | 0.42755 (17) | 0.0237 (6)                       |

|     |               |              |              |             |
|-----|---------------|--------------|--------------|-------------|
| C8  | 0.26762 (19)  | 0.5094 (2)   | 0.51561 (17) | 0.0226 (6)  |
| C16 | 0.57563 (18)  | 0.5637 (2)   | 0.82647 (17) | 0.0223 (5)  |
| C17 | 0.53285 (18)  | 0.6686 (2)   | 0.81428 (17) | 0.0218 (6)  |
| C4  | 0.48588 (19)  | 0.2959 (2)   | 0.48975 (18) | 0.0267 (6)  |
| C3  | 0.45048 (18)  | 0.2883 (2)   | 0.56423 (17) | 0.0226 (5)  |
| C5  | 0.44853 (19)  | 0.3770 (2)   | 0.41992 (18) | 0.0275 (6)  |
| C23 | 0.2158 (2)    | 0.2237 (2)   | 0.3452 (2)   | 0.0318 (6)  |
| C24 | 0.2804 (2)    | 0.1353 (2)   | 0.3683 (2)   | 0.0337 (7)  |
| C22 | 0.1948 (2)    | 0.2738 (2)   | 0.2563 (2)   | 0.0333 (7)  |
| C21 | 0.2388 (2)    | 0.2333 (3)   | 0.1942 (2)   | 0.0349 (7)  |
| C25 | 0.3212 (2)    | 0.1019 (3)   | 0.3011 (2)   | 0.0372 (7)  |
| H15 | 0.55133 (18)  | 0.3883 (2)   | 0.79650 (16) | 0.0230 (6)* |
| H16 | 0.65404 (18)  | 0.5536 (2)   | 0.86638 (17) | 0.0268 (7)* |
| H17 | 0.57648 (18)  | 0.7428 (2)   | 0.84629 (17) | 0.0262 (7)* |
| H18 | 0.39974 (18)  | 0.7614 (2)   | 0.74799 (17) | 0.0235 (6)* |
| H9  | 0.27723 (18)  | 0.10753 (19) | 0.61966 (16) | 0.0218 (6)* |
| H10 | 0.12387 (18)  | 0.0139 (2)   | 0.52225 (16) | 0.0241 (6)* |
| H11 | −0.02659 (19) | 0.1275 (2)   | 0.44658 (17) | 0.0244 (6)* |
| H12 | −0.01730 (17) | 0.3350 (2)   | 0.47063 (16) | 0.0219 (6)* |
| H3  | 0.48006 (18)  | 0.2233 (2)   | 0.61929 (17) | 0.0271 (7)* |
| H4  | 0.54291 (19)  | 0.2385 (2)   | 0.48586 (18) | 0.0320 (7)* |
| H5  | 0.47520 (19)  | 0.3851 (2)   | 0.35951 (18) | 0.0330 (7)* |
| H6  | 0.34565 (19)  | 0.5129 (2)   | 0.37306 (17) | 0.0284 (7)* |
| H21 | 0.2215 (2)    | 0.2738 (3)   | 0.1235 (2)   | 0.0419 (8)* |
| H25 | 0.3737 (2)    | 0.0326 (3)   | 0.3203 (2)   | 0.0447 (9)* |
| H24 | 0.2991 (2)    | 0.0925 (2)   | 0.4382 (2)   | 0.0404 (8)* |
| H23 | 0.1820 (2)    | 0.2534 (2)   | 0.3963 (2)   | 0.0382 (8)* |
| H22 | 0.1441 (2)    | 0.3447 (2)   | 0.2351 (2)   | 0.0399 (8)* |
| H14 | 0.12201 (17)  | 0.4695 (2)   | 0.64588 (17) | 0.0229 (6)* |
| H13 | 0.09693 (17)  | 0.5000 (2)   | 0.52084 (17) | 0.0229 (6)* |
| H19 | 0.25166 (17)  | 0.66175 (19) | 0.62983 (16) | 0.0219 (6)* |
| H20 | 0.23383 (17)  | 0.57877 (19) | 0.72328 (16) | 0.0219 (6)* |
| H8  | 0.19981 (19)  | 0.4975 (2)   | 0.45271 (17) | 0.0272 (7)* |
| H7  | 0.29029 (19)  | 0.5978 (2)   | 0.51599 (17) | 0.0272 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.01626 (17) | 0.01365 (17) | 0.01145 (16) | 0.00169 (15) | 0.00475 (13) | −0.00002 (14) |
| S2  | 0.0265 (4)   | 0.0219 (3)   | 0.0276 (3)   | 0.0063 (3)   | 0.0113 (3)   | 0.0052 (3)    |
| S1  | 0.0463 (4)   | 0.0318 (4)   | 0.0215 (3)   | −0.0146 (3)  | 0.0184 (3)   | −0.0074 (3)   |
| N6  | 0.0178 (11)  | 0.0145 (10)  | 0.0135 (9)   | −0.0002 (8)  | 0.0046 (8)   | 0.0008 (8)    |
| N5  | 0.0189 (11)  | 0.0144 (10)  | 0.0132 (9)   | 0.0024 (8)   | 0.0047 (8)   | 0.0001 (8)    |
| N4  | 0.0168 (10)  | 0.0134 (10)  | 0.0143 (9)   | 0.0020 (8)   | 0.0032 (8)   | 0.0001 (8)    |
| N3  | 0.0195 (10)  | 0.0184 (10)  | 0.0118 (9)   | 0.0018 (9)   | 0.0064 (8)   | −0.0006 (8)   |
| N1  | 0.0236 (11)  | 0.0180 (10)  | 0.0167 (10)  | −0.0008 (9)  | 0.0093 (9)   | −0.0018 (8)   |
| N2  | 0.0217 (11)  | 0.0181 (11)  | 0.0172 (10)  | 0.0040 (9)   | 0.0058 (8)   | 0.0019 (9)    |
| C18 | 0.0201 (13)  | 0.0188 (13)  | 0.0193 (12)  | −0.0014 (10) | 0.0060 (10)  | −0.0033 (10)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C19 | 0.0207 (13) | 0.0145 (12) | 0.0150 (11) | −0.0012 (10) | 0.0062 (10) | −0.0019 (9)  |
| C12 | 0.0168 (13) | 0.0192 (12) | 0.0172 (11) | 0.0003 (10)  | 0.0040 (10) | −0.0015 (10) |
| C13 | 0.0182 (13) | 0.0142 (12) | 0.0153 (11) | 0.0008 (10)  | 0.0053 (10) | −0.0018 (9)  |
| C14 | 0.0177 (12) | 0.0146 (12) | 0.0228 (13) | 0.0030 (10)  | 0.0046 (10) | −0.0016 (10) |
| C1  | 0.0258 (13) | 0.0154 (12) | 0.0165 (12) | −0.0035 (10) | 0.0072 (10) | −0.0020 (10) |
| C15 | 0.0184 (13) | 0.0211 (13) | 0.0161 (11) | 0.0038 (10)  | 0.0036 (10) | 0.0027 (10)  |
| C20 | 0.0222 (13) | 0.0144 (12) | 0.0175 (12) | 0.0017 (10)  | 0.0063 (10) | −0.0012 (9)  |
| N7  | 0.0436 (15) | 0.0419 (15) | 0.0383 (14) | −0.0029 (13) | 0.0242 (12) | −0.0086 (12) |
| C10 | 0.0254 (14) | 0.0158 (12) | 0.0176 (11) | −0.0013 (10) | 0.0058 (10) | −0.0009 (9)  |
| C9  | 0.0238 (13) | 0.0143 (12) | 0.0151 (11) | 0.0022 (10)  | 0.0049 (10) | 0.0004 (9)   |
| C7  | 0.0203 (13) | 0.0213 (12) | 0.0118 (11) | −0.0021 (10) | 0.0054 (10) | 0.0008 (9)   |
| C11 | 0.0231 (14) | 0.0180 (13) | 0.0191 (12) | −0.0032 (10) | 0.0063 (10) | −0.0019 (10) |
| C2  | 0.0178 (12) | 0.0200 (13) | 0.0157 (11) | −0.0005 (11) | 0.0046 (10) | 0.0001 (10)  |
| C6  | 0.0273 (14) | 0.0296 (14) | 0.0149 (12) | −0.0008 (12) | 0.0083 (10) | 0.0037 (11)  |
| C8  | 0.0294 (15) | 0.0228 (14) | 0.0161 (12) | 0.0054 (11)  | 0.0085 (11) | 0.0052 (10)  |
| C16 | 0.0170 (13) | 0.0295 (14) | 0.0174 (12) | −0.0006 (11) | 0.0023 (10) | 0.0018 (11)  |
| C17 | 0.0226 (14) | 0.0241 (14) | 0.0189 (12) | −0.0047 (11) | 0.0075 (11) | −0.0041 (10) |
| C4  | 0.0249 (14) | 0.0345 (16) | 0.0251 (13) | 0.0039 (12)  | 0.0144 (12) | −0.0017 (12) |
| C3  | 0.0252 (14) | 0.0270 (14) | 0.0188 (12) | 0.0045 (11)  | 0.0115 (11) | 0.0003 (10)  |
| C5  | 0.0279 (14) | 0.0404 (17) | 0.0187 (13) | −0.0004 (13) | 0.0139 (11) | 0.0004 (12)  |
| C23 | 0.0303 (15) | 0.0356 (16) | 0.0352 (15) | −0.0077 (13) | 0.0185 (13) | −0.0127 (13) |
| C24 | 0.0384 (16) | 0.0333 (16) | 0.0322 (15) | −0.0084 (14) | 0.0159 (13) | −0.0052 (13) |
| C22 | 0.0289 (15) | 0.0335 (16) | 0.0388 (16) | −0.0029 (13) | 0.0136 (13) | −0.0037 (13) |
| C21 | 0.0340 (16) | 0.0400 (17) | 0.0332 (16) | −0.0057 (14) | 0.0149 (13) | −0.0014 (14) |
| C25 | 0.0395 (18) | 0.0342 (16) | 0.0426 (18) | −0.0001 (14) | 0.0202 (15) | −0.0066 (14) |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N6  | 1.976 (2)   | C20—H20 | 1.1110    |
| Fe1—N5  | 1.986 (2)   | N7—C21  | 1.324 (4) |
| Fe1—N4  | 2.0025 (19) | N7—C25  | 1.328 (4) |
| Fe1—N3  | 1.9769 (19) | C10—C9  | 1.390 (3) |
| Fe1—N1  | 1.971 (2)   | C10—C11 | 1.386 (4) |
| Fe1—N2  | 1.948 (2)   | C10—H10 | 1.1030    |
| S2—C2   | 1.642 (3)   | C9—H9   | 1.1030    |
| S1—C1   | 1.647 (2)   | C7—C6   | 1.390 (3) |
| N6—C19  | 1.349 (3)   | C7—C8   | 1.498 (3) |
| N6—C15  | 1.343 (3)   | C11—H11 | 1.1030    |
| N5—C13  | 1.346 (3)   | C6—C5   | 1.388 (4) |
| N5—C9   | 1.344 (3)   | C6—H6   | 1.1030    |
| N4—C14  | 1.485 (3)   | C8—H8   | 1.1110    |
| N4—C20  | 1.483 (3)   | C8—H7   | 1.1110    |
| N4—C8   | 1.501 (3)   | C16—C17 | 1.390 (4) |
| N3—C7   | 1.343 (3)   | C16—H16 | 1.1030    |
| N3—C3   | 1.348 (3)   | C17—H17 | 1.1030    |
| N1—C1   | 1.159 (3)   | C4—C3   | 1.382 (3) |
| N2—C2   | 1.168 (3)   | C4—C5   | 1.388 (4) |
| C18—C19 | 1.394 (3)   | C4—H4   | 1.1030    |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C18—C17     | 1.386 (3)   | C3—H3       | 1.1030      |
| C18—H18     | 1.1030      | C5—H5       | 1.1030      |
| C19—C20     | 1.505 (3)   | C23—C24     | 1.384 (4)   |
| C12—C13     | 1.389 (3)   | C23—C22     | 1.385 (4)   |
| C12—C11     | 1.389 (3)   | C23—H23     | 1.1030      |
| C12—H12     | 1.1030      | C24—C25     | 1.393 (4)   |
| C13—C14     | 1.500 (3)   | C24—H24     | 1.1030      |
| C14—H14     | 1.1110      | C22—C21     | 1.387 (4)   |
| C14—H13     | 1.1110      | C22—H22     | 1.1030      |
| C15—C16     | 1.394 (3)   | C21—H21     | 1.1030      |
| C15—H15     | 1.1030      | C25—H25     | 1.1030      |
| C20—H19     | 1.1110      |             |             |
| N5—Fe1—N6   | 166.01 (8)  | H20—C20—N4  | 109.91 (12) |
| N4—Fe1—N6   | 82.82 (8)   | H20—C20—C19 | 109.91 (12) |
| N4—Fe1—N5   | 83.23 (8)   | H20—C20—H19 | 108.3       |
| N3—Fe1—N6   | 88.65 (8)   | C25—N7—C21  | 117.1 (3)   |
| N3—Fe1—N5   | 91.22 (8)   | C11—C10—C9  | 119.5 (2)   |
| N3—Fe1—N4   | 85.04 (8)   | H10—C10—C9  | 120.27 (14) |
| N1—Fe1—N6   | 89.38 (8)   | H10—C10—C11 | 120.27 (15) |
| N1—Fe1—N5   | 90.16 (8)   | C10—C9—N5   | 122.0 (2)   |
| N1—Fe1—N4   | 92.54 (8)   | H9—C9—N5    | 118.99 (14) |
| N1—Fe1—N3   | 177.05 (8)  | H9—C9—C10   | 118.99 (14) |
| N2—Fe1—N6   | 98.31 (8)   | C6—C7—N3    | 122.2 (2)   |
| N2—Fe1—N5   | 95.68 (8)   | C8—C7—N3    | 116.8 (2)   |
| N2—Fe1—N4   | 177.23 (8)  | C8—C7—C6    | 121.0 (2)   |
| N2—Fe1—N3   | 92.45 (8)   | C10—C11—C12 | 118.6 (2)   |
| N2—Fe1—N1   | 90.01 (8)   | H11—C11—C12 | 120.72 (15) |
| C19—N6—Fe1  | 113.66 (15) | H11—C11—C10 | 120.72 (15) |
| C15—N6—Fe1  | 127.53 (16) | N2—C2—S2    | 177.0 (2)   |
| C15—N6—C19  | 118.8 (2)   | C5—C6—C7    | 119.1 (2)   |
| C13—N5—Fe1  | 113.17 (15) | H6—C6—C7    | 120.44 (15) |
| C9—N5—Fe1   | 128.31 (17) | H6—C6—C5    | 120.44 (15) |
| C9—N5—C13   | 118.5 (2)   | C7—C8—N4    | 112.63 (19) |
| C14—N4—Fe1  | 106.52 (14) | H8—C8—N4    | 109.07 (12) |
| C20—N4—Fe1  | 106.06 (14) | H8—C8—C7    | 109.07 (13) |
| C20—N4—C14  | 111.81 (18) | H7—C8—N4    | 109.07 (13) |
| C8—N4—Fe1   | 110.43 (14) | H7—C8—C7    | 109.07 (14) |
| C8—N4—C14   | 110.84 (18) | H7—C8—H8    | 107.8       |
| C8—N4—C20   | 110.97 (18) | C17—C16—C15 | 118.7 (2)   |
| C7—N3—Fe1   | 114.98 (16) | H16—C16—C15 | 120.65 (15) |
| C3—N3—Fe1   | 126.61 (16) | H16—C16—C17 | 120.65 (15) |
| C3—N3—C7    | 118.3 (2)   | C16—C17—C18 | 119.2 (2)   |
| C1—N1—Fe1   | 173.3 (2)   | H17—C17—C18 | 120.42 (15) |
| C2—N2—Fe1   | 170.58 (19) | H17—C17—C16 | 120.42 (15) |
| C17—C18—C19 | 118.9 (2)   | C5—C4—C3    | 118.9 (2)   |
| H18—C18—C19 | 120.56 (14) | H4—C4—C3    | 120.53 (16) |
| H18—C18—C17 | 120.56 (15) | H4—C4—C5    | 120.53 (15) |



|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C18—C19—N6     | 122.1 (2)    | C4—C3—N3        | 122.7 (2)    |
| C20—C19—N6     | 114.5 (2)    | H3—C3—N3        | 118.65 (13)  |
| C20—C19—C18    | 123.4 (2)    | H3—C3—C4        | 118.65 (16)  |
| C11—C12—C13    | 119.0 (2)    | C4—C5—C6        | 118.7 (2)    |
| H12—C12—C13    | 120.52 (14)  | H5—C5—C6        | 120.64 (15)  |
| H12—C12—C11    | 120.52 (15)  | H5—C5—C4        | 120.64 (15)  |
| C12—C13—N5     | 122.5 (2)    | C22—C23—C24     | 118.2 (3)    |
| C14—C13—N5     | 115.4 (2)    | H23—C23—C24     | 120.88 (17)  |
| C14—C13—C12    | 122.1 (2)    | H23—C23—C22     | 120.88 (17)  |
| C13—C14—N4     | 109.45 (19)  | C25—C24—C23     | 118.3 (3)    |
| H14—C14—N4     | 109.78 (12)  | H24—C24—C23     | 120.87 (17)  |
| H14—C14—C13    | 109.78 (13)  | H24—C24—C25     | 120.87 (19)  |
| H13—C14—N4     | 109.78 (12)  | C21—C22—C23     | 118.8 (3)    |
| H13—C14—C13    | 109.78 (13)  | H22—C22—C23     | 120.58 (17)  |
| H13—C14—H14    | 108.2        | H22—C22—C21     | 120.58 (18)  |
| N1—C1—S1       | 178.4 (2)    | C22—C21—N7      | 123.6 (3)    |
| C16—C15—N6     | 122.3 (2)    | H21—C21—N7      | 118.18 (16)  |
| H15—C15—N6     | 118.84 (13)  | H21—C21—C22     | 118.18 (18)  |
| H15—C15—C16    | 118.84 (15)  | C24—C25—N7      | 123.9 (3)    |
| C19—C20—N4     | 108.88 (19)  | H25—C25—N7      | 118.06 (17)  |
| H19—C20—N4     | 109.91 (12)  | H25—C25—C24     | 118.06 (19)  |
| H19—C20—C19    | 109.91 (13)  |                 |              |
| Fe1—N6—C19—C18 | −179.02 (15) | C19—C18—C17—C16 | 2.7 (3)      |
| Fe1—N6—C19—C20 | 2.66 (16)    | C19—C20—N4—C14  | 155.08 (18)  |
| Fe1—N6—C15—C16 | 179.71 (18)  | C19—C20—N4—C8   | −80.6 (2)    |
| Fe1—N5—C13—C12 | 179.65 (15)  | C12—C13—N5—C9   | 0.2 (3)      |
| Fe1—N5—C13—C14 | −2.50 (16)   | C12—C11—C10—C9  | 0.3 (3)      |
| Fe1—N5—C9—C10  | 179.57 (19)  | C13—N5—C9—C10   | −1.1 (2)     |
| Fe1—N4—C14—C13 | −36.82 (14)  | C13—C12—C11—C10 | −1.1 (3)     |
| Fe1—N4—C20—C19 | 39.36 (14)   | C13—C14—N4—C20  | −152.27 (18) |
| Fe1—N4—C8—C7   | −1.88 (15)   | C13—C14—N4—C8   | 83.3 (2)     |
| Fe1—N3—C7—C6   | 177.40 (17)  | C14—N4—C8—C7    | −119.69 (19) |
| Fe1—N3—C7—C8   | −3.27 (18)   | C14—C13—N5—C9   | 178.1 (2)    |
| Fe1—N3—C3—C4   | −176.7 (2)   | C14—C13—C12—C11 | −176.8 (2)   |
| N6—C19—C18—C17 | −1.5 (3)     | C15—N6—C19—C20  | −178.9 (2)   |
| N6—C19—C20—N4  | −28.7 (2)    | C20—N4—C8—C7    | 115.45 (19)  |
| N6—C15—C16—C17 | −0.3 (3)     | C20—C19—C18—C17 | 176.6 (2)    |
| N5—C13—C12—C11 | 0.9 (3)      | N7—C21—C22—C23  | −0.1 (3)     |
| N5—C13—C14—N4  | 26.9 (2)     | N7—C25—C24—C23  | −1.2 (4)     |
| N5—C9—C10—C11  | 0.8 (3)      | C7—N3—C3—C4     | 0.4 (3)      |
| N4—C14—C13—C12 | −155.29 (18) | C7—C6—C5—C4     | 0.2 (3)      |
| N4—C20—C19—C18 | 153.05 (18)  | C6—C7—N3—C3     | −0.0 (3)     |
| N4—C8—C7—N3    | 3.4 (2)      | C6—C5—C4—C3     | 0.1 (3)      |
| N4—C8—C7—C6    | −177.3 (2)   | C8—C7—N3—C3     | 179.3 (2)    |
| N3—C7—C6—C5    | −0.3 (3)     | C8—C7—C6—C5     | −179.6 (2)   |
| N3—C3—C4—C5    | −0.4 (3)     | C24—C23—C22—C21 | 0.3 (3)      |
| C18—C19—N6—C15 | −0.6 (3)     | C24—C25—N7—C21  | 1.4 (4)      |

|                 |          |                 |          |
|-----------------|----------|-----------------|----------|
| C18—C17—C16—C15 | −1.8 (3) | C22—C23—C24—C25 | 0.3 (3)  |
| C19—N6—C15—C16  | 1.5 (2)  | C22—C21—N7—C25  | −0.8 (3) |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_135K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.7531$  (9) Å

$b = 12.0107$  (6) Å

$c = 14.8834$  (8) Å

$\beta = 110.619$  (7)°

$V = 2468.3$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.457$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9620 reflections

$\theta = 2.1$ – $30.5^\circ$

$\mu = 0.81$  mm<sup>−1</sup>

$T = 135$  K

Block, red

$0.21 \times 0.08 \times 0.05$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.822$ ,  $T_{\max} = 1.000$

27436 measured reflections

5036 independent reflections

3854 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -18 \rightarrow 18$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 18$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.085$

$S = 1.05$

5036 reflections

316 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 2.2495P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.0004$

$\Delta\rho_{\max} = 0.63$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.51$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.32965 (2)  | 0.35881 (3)  | 0.67890 (2)  | 0.01679 (9)                      |
| S2  | 0.51837 (5)  | 0.03672 (6)  | 0.80032 (5)  | 0.02951 (16)                     |
| S1  | 0.21862 (6)  | 0.36317 (6)  | 0.94389 (5)  | 0.03712 (19)                     |
| N6  | 0.42190 (14) | 0.48026 (16) | 0.73894 (13) | 0.0179 (4)                       |
| N4  | 0.24767 (14) | 0.48583 (16) | 0.60614 (13) | 0.0170 (4)                       |
| N3  | 0.38129 (14) | 0.35656 (17) | 0.57252 (13) | 0.0188 (4)                       |
| N5  | 0.21533 (14) | 0.26822 (16) | 0.60251 (13) | 0.0176 (4)                       |
| N1  | 0.27733 (14) | 0.37047 (16) | 0.78386 (14) | 0.0221 (4)                       |
| N2  | 0.40945 (15) | 0.23289 (18) | 0.74314 (14) | 0.0230 (5)                       |
| C18 | 0.43456 (18) | 0.6787 (2)   | 0.76076 (17) | 0.0223 (5)                       |
| C19 | 0.38103 (17) | 0.5825 (2)   | 0.72507 (16) | 0.0195 (5)                       |
| C13 | 0.13513 (17) | 0.32978 (19) | 0.56146 (16) | 0.0189 (5)                       |

|     |               |              |              |             |
|-----|---------------|--------------|--------------|-------------|
| C12 | 0.04743 (18)  | 0.2832 (2)   | 0.50466 (17) | 0.0215 (5)  |
| C1  | 0.25358 (18)  | 0.3690 (2)   | 0.85029 (17) | 0.0220 (5)  |
| C14 | 0.14547 (17)  | 0.4518 (2)   | 0.58409 (17) | 0.0216 (5)  |
| C15 | 0.51749 (18)  | 0.4718 (2)   | 0.78755 (17) | 0.0229 (5)  |
| C7  | 0.34523 (18)  | 0.4350 (2)   | 0.50511 (16) | 0.0205 (5)  |
| C9  | 0.21014 (18)  | 0.15766 (19) | 0.58731 (16) | 0.0209 (5)  |
| C10 | 0.12499 (19)  | 0.1050 (2)   | 0.53224 (17) | 0.0235 (5)  |
| C20 | 0.27384 (17)  | 0.58317 (19) | 0.67180 (17) | 0.0209 (5)  |
| C11 | 0.04237 (19)  | 0.1687 (2)   | 0.49080 (17) | 0.0231 (5)  |
| N7  | 0.29991 (19)  | 0.1494 (2)   | 0.21504 (18) | 0.0442 (6)  |
| C2  | 0.45421 (18)  | 0.1517 (2)   | 0.76876 (16) | 0.0228 (5)  |
| C6  | 0.37655 (19)  | 0.4477 (2)   | 0.42781 (17) | 0.0274 (6)  |
| C8  | 0.26765 (19)  | 0.5084 (2)   | 0.51587 (17) | 0.0248 (6)  |
| C16 | 0.57491 (18)  | 0.5636 (2)   | 0.82613 (17) | 0.0255 (6)  |
| C17 | 0.53256 (18)  | 0.6686 (2)   | 0.81322 (17) | 0.0249 (6)  |
| C3  | 0.45040 (19)  | 0.2883 (2)   | 0.56433 (17) | 0.0257 (6)  |
| C4  | 0.4855 (2)    | 0.2961 (2)   | 0.48986 (18) | 0.0311 (6)  |
| C5  | 0.44811 (19)  | 0.3772 (2)   | 0.42049 (18) | 0.0318 (6)  |
| C23 | 0.2159 (2)    | 0.2236 (3)   | 0.3450 (2)   | 0.0372 (7)  |
| C24 | 0.2799 (2)    | 0.1353 (3)   | 0.3680 (2)   | 0.0381 (7)  |
| C22 | 0.1946 (2)    | 0.2740 (3)   | 0.2566 (2)   | 0.0391 (7)  |
| C25 | 0.3204 (2)    | 0.1022 (3)   | 0.3006 (2)   | 0.0421 (7)  |
| C21 | 0.2381 (2)    | 0.2339 (3)   | 0.1945 (2)   | 0.0402 (7)  |
| H15 | 0.55153 (18)  | 0.3888 (2)   | 0.79712 (17) | 0.0275 (6)* |
| H16 | 0.65305 (18)  | 0.5536 (2)   | 0.86649 (17) | 0.0306 (7)* |
| H17 | 0.57639 (18)  | 0.7429 (2)   | 0.84431 (17) | 0.0299 (7)* |
| H18 | 0.39954 (18)  | 0.7612 (2)   | 0.74746 (17) | 0.0268 (6)* |
| H3  | 0.48022 (19)  | 0.2235 (2)   | 0.61937 (17) | 0.0308 (7)* |
| H4  | 0.5423 (2)    | 0.2387 (2)   | 0.48575 (18) | 0.0373 (8)* |
| H5  | 0.47482 (19)  | 0.3856 (2)   | 0.36030 (18) | 0.0382 (8)* |
| H6  | 0.34505 (19)  | 0.5126 (2)   | 0.37332 (17) | 0.0329 (7)* |
| H9  | 0.27597 (18)  | 0.10699 (19) | 0.61980 (16) | 0.0251 (6)* |
| H10 | 0.12308 (19)  | 0.0141 (2)   | 0.52161 (17) | 0.0282 (7)* |
| H11 | −0.02653 (19) | 0.1289 (2)   | 0.44738 (17) | 0.0277 (6)* |
| H12 | −0.01681 (18) | 0.3361 (2)   | 0.47128 (17) | 0.0258 (6)* |
| H25 | 0.3726 (2)    | 0.0326 (3)   | 0.3195 (2)   | 0.0505 (9)* |
| H21 | 0.2203 (2)    | 0.2744 (3)   | 0.1239 (2)   | 0.0482 (9)* |
| H22 | 0.1439 (2)    | 0.3448 (3)   | 0.2358 (2)   | 0.0469 (9)* |
| H23 | 0.1825 (2)    | 0.2532 (3)   | 0.3963 (2)   | 0.0447 (8)* |
| H24 | 0.2984 (2)    | 0.0923 (3)   | 0.4376 (2)   | 0.0457 (8)* |
| H14 | 0.12331 (17)  | 0.4695 (2)   | 0.64669 (17) | 0.0259 (6)* |
| H13 | 0.09763 (17)  | 0.5001 (2)   | 0.52174 (17) | 0.0259 (6)* |
| H19 | 0.25242 (17)  | 0.66146 (19) | 0.62989 (17) | 0.0250 (6)* |
| H20 | 0.23489 (17)  | 0.57882 (19) | 0.72343 (17) | 0.0250 (6)* |
| H8  | 0.19993 (19)  | 0.4959 (2)   | 0.45319 (17) | 0.0298 (7)* |
| H7  | 0.28982 (19)  | 0.5968 (2)   | 0.51571 (17) | 0.0298 (7)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.01915 (18) | 0.01674 (17) | 0.01499 (16) | 0.00135 (15) | 0.00665 (13) | −0.00122 (14) |
| S2  | 0.0309 (4)   | 0.0280 (4)   | 0.0318 (4)   | 0.0056 (3)   | 0.0138 (3)   | 0.0054 (3)    |
| S1  | 0.0554 (5)   | 0.0367 (4)   | 0.0250 (3)   | −0.0180 (4)  | 0.0213 (3)   | −0.0084 (3)   |
| N6  | 0.0202 (11)  | 0.0183 (11)  | 0.0150 (9)   | 0.0015 (9)   | 0.0057 (8)   | 0.0011 (8)    |
| N4  | 0.0197 (10)  | 0.0140 (10)  | 0.0164 (9)   | 0.0028 (8)   | 0.0052 (8)   | −0.0003 (8)   |
| N3  | 0.0229 (10)  | 0.0214 (10)  | 0.0134 (9)   | 0.0024 (9)   | 0.0082 (8)   | 0.0007 (8)    |
| N5  | 0.0217 (11)  | 0.0161 (10)  | 0.0140 (9)   | 0.0020 (9)   | 0.0051 (8)   | −0.0005 (8)   |
| N1  | 0.0280 (11)  | 0.0202 (11)  | 0.0209 (10)  | −0.0005 (9)  | 0.0121 (9)   | −0.0009 (8)   |
| N2  | 0.0254 (11)  | 0.0213 (12)  | 0.0215 (10)  | 0.0066 (10)  | 0.0072 (9)   | 0.0016 (9)    |
| C18 | 0.0243 (14)  | 0.0211 (13)  | 0.0218 (12)  | −0.0002 (11) | 0.0083 (11)  | −0.0026 (10)  |
| C19 | 0.0234 (13)  | 0.0186 (13)  | 0.0159 (11)  | −0.0011 (10) | 0.0062 (10)  | −0.0020 (9)   |
| C13 | 0.0209 (13)  | 0.0166 (12)  | 0.0186 (11)  | 0.0013 (10)  | 0.0064 (10)  | −0.0006 (9)   |
| C12 | 0.0207 (13)  | 0.0206 (13)  | 0.0220 (12)  | 0.0001 (10)  | 0.0061 (10)  | −0.0001 (10)  |
| C1  | 0.0278 (13)  | 0.0192 (12)  | 0.0180 (12)  | −0.0027 (11) | 0.0069 (10)  | −0.0017 (10)  |
| C14 | 0.0207 (13)  | 0.0163 (12)  | 0.0252 (13)  | 0.0022 (10)  | 0.0048 (10)  | −0.0024 (10)  |
| C15 | 0.0221 (13)  | 0.0257 (14)  | 0.0196 (11)  | 0.0011 (11)  | 0.0058 (10)  | 0.0032 (10)   |
| C7  | 0.0247 (13)  | 0.0228 (13)  | 0.0143 (11)  | −0.0011 (10) | 0.0072 (10)  | 0.0009 (10)   |
| C9  | 0.0279 (14)  | 0.0168 (13)  | 0.0172 (11)  | 0.0028 (10)  | 0.0068 (10)  | −0.0002 (9)   |
| C10 | 0.0320 (15)  | 0.0163 (12)  | 0.0230 (12)  | −0.0009 (11) | 0.0106 (11)  | −0.0003 (10)  |
| C20 | 0.0228 (13)  | 0.0159 (12)  | 0.0232 (12)  | 0.0018 (10)  | 0.0073 (10)  | −0.0023 (10)  |
| C11 | 0.0252 (14)  | 0.0208 (13)  | 0.0217 (12)  | −0.0024 (10) | 0.0064 (11)  | −0.0018 (10)  |
| N7  | 0.0491 (16)  | 0.0491 (17)  | 0.0432 (15)  | −0.0036 (14) | 0.0273 (13)  | −0.0103 (13)  |
| C2  | 0.0224 (13)  | 0.0279 (15)  | 0.0177 (11)  | −0.0016 (12) | 0.0067 (10)  | 0.0013 (11)   |
| C6  | 0.0318 (15)  | 0.0337 (15)  | 0.0183 (12)  | −0.0008 (12) | 0.0107 (11)  | 0.0053 (11)   |
| C8  | 0.0314 (15)  | 0.0240 (14)  | 0.0180 (12)  | 0.0081 (11)  | 0.0075 (11)  | 0.0072 (10)   |
| C16 | 0.0194 (13)  | 0.0344 (15)  | 0.0188 (12)  | −0.0005 (11) | 0.0019 (10)  | 0.0025 (11)   |
| C17 | 0.0260 (14)  | 0.0283 (15)  | 0.0200 (12)  | −0.0070 (11) | 0.0076 (11)  | −0.0060 (10)  |
| C3  | 0.0286 (14)  | 0.0299 (14)  | 0.0211 (13)  | 0.0054 (11)  | 0.0119 (11)  | −0.0007 (11)  |
| C4  | 0.0313 (15)  | 0.0413 (17)  | 0.0259 (14)  | 0.0042 (13)  | 0.0168 (12)  | −0.0036 (12)  |
| C5  | 0.0312 (15)  | 0.0478 (18)  | 0.0210 (13)  | 0.0004 (13)  | 0.0148 (11)  | 0.0005 (12)   |
| C23 | 0.0379 (17)  | 0.0396 (17)  | 0.0409 (17)  | −0.0082 (14) | 0.0222 (14)  | −0.0121 (14)  |
| C24 | 0.0437 (17)  | 0.0385 (17)  | 0.0359 (16)  | −0.0085 (15) | 0.0186 (14)  | −0.0044 (14)  |
| C22 | 0.0311 (16)  | 0.0416 (18)  | 0.0466 (18)  | −0.0033 (14) | 0.0161 (14)  | −0.0058 (15)  |
| C25 | 0.0453 (19)  | 0.0378 (17)  | 0.0459 (18)  | 0.0012 (15)  | 0.0194 (15)  | −0.0064 (15)  |
| C21 | 0.0391 (17)  | 0.0463 (19)  | 0.0368 (16)  | −0.0070 (15) | 0.0154 (14)  | −0.0024 (14)  |

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

|        |             |         |           |
|--------|-------------|---------|-----------|
| Fe1—N6 | 1.980 (2)   | C7—C8   | 1.498 (3) |
| Fe1—N4 | 2.0102 (19) | C9—C10  | 1.387 (4) |
| Fe1—N3 | 1.9844 (19) | C9—H9   | 1.1030    |
| Fe1—N5 | 1.993 (2)   | C10—C11 | 1.386 (4) |
| Fe1—N1 | 1.976 (2)   | C10—H10 | 1.1030    |
| Fe1—N2 | 1.948 (2)   | C20—H19 | 1.1110    |
| S2—C2  | 1.646 (3)   | C20—H20 | 1.1110    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| S1—C1      | 1.648 (2)   | C11—H11     | 1.1030      |
| N6—C19     | 1.351 (3)   | N7—C25      | 1.329 (4)   |
| N6—C15     | 1.343 (3)   | N7—C21      | 1.325 (4)   |
| N4—C14     | 1.483 (3)   | C6—C5       | 1.387 (4)   |
| N4—C20     | 1.485 (3)   | C6—H6       | 1.1030      |
| N4—C8      | 1.498 (3)   | C8—H8       | 1.1110      |
| N3—C7      | 1.342 (3)   | C8—H7       | 1.1110      |
| N3—C3      | 1.347 (3)   | C16—C17     | 1.390 (4)   |
| N5—C13     | 1.346 (3)   | C16—H16     | 1.1030      |
| N5—C9      | 1.345 (3)   | C17—H17     | 1.1030      |
| N1—C1      | 1.160 (3)   | C3—C4       | 1.382 (3)   |
| N2—C2      | 1.164 (3)   | C3—H3       | 1.1030      |
| C18—C19    | 1.395 (3)   | C4—C5       | 1.385 (4)   |
| C18—C17    | 1.385 (3)   | C4—H4       | 1.1030      |
| C18—H18    | 1.1030      | C5—H5       | 1.1030      |
| C19—C20    | 1.499 (3)   | C23—C24     | 1.379 (4)   |
| C13—C12    | 1.390 (3)   | C23—C22     | 1.381 (4)   |
| C13—C14    | 1.500 (3)   | C23—H23     | 1.1030      |
| C12—C11    | 1.390 (3)   | C24—C25     | 1.394 (4)   |
| C12—H12    | 1.1030      | C24—H24     | 1.1030      |
| C14—H14    | 1.1110      | C22—C21     | 1.384 (4)   |
| C14—H13    | 1.1110      | C22—H22     | 1.1030      |
| C15—C16    | 1.386 (4)   | C25—H25     | 1.1030      |
| C15—H15    | 1.1030      | C21—H21     | 1.1030      |
| C7—C6      | 1.391 (3)   |             |             |
| N4—Fe1—N6  | 82.62 (8)   | C10—C9—N5   | 122.4 (2)   |
| N3—Fe1—N6  | 88.51 (8)   | H9—C9—N5    | 118.81 (14) |
| N3—Fe1—N4  | 84.70 (8)   | H9—C9—C10   | 118.81 (15) |
| N5—Fe1—N6  | 165.64 (8)  | C11—C10—C9  | 118.9 (2)   |
| N5—Fe1—N4  | 83.06 (8)   | H10—C10—C9  | 120.55 (15) |
| N5—Fe1—N3  | 91.31 (8)   | H10—C10—C11 | 120.55 (15) |
| N1—Fe1—N6  | 89.36 (8)   | C19—C20—N4  | 108.97 (19) |
| N1—Fe1—N4  | 92.51 (8)   | H19—C20—N4  | 109.89 (12) |
| N1—Fe1—N3  | 176.69 (8)  | H19—C20—C19 | 109.89 (13) |
| N1—Fe1—N5  | 90.12 (8)   | H20—C20—N4  | 109.89 (12) |
| N2—Fe1—N6  | 98.62 (8)   | H20—C20—C19 | 109.89 (12) |
| N2—Fe1—N4  | 177.02 (8)  | H20—C20—H19 | 108.3       |
| N2—Fe1—N3  | 92.61 (8)   | C10—C11—C12 | 119.2 (2)   |
| N2—Fe1—N5  | 95.74 (8)   | H11—C11—C12 | 120.42 (15) |
| N2—Fe1—N1  | 90.22 (8)   | H11—C11—C10 | 120.42 (15) |
| C19—N6—Fe1 | 113.68 (15) | C21—N7—C25  | 117.0 (3)   |
| C15—N6—Fe1 | 127.73 (16) | N2—C2—S2    | 177.4 (2)   |
| C15—N6—C19 | 118.6 (2)   | C5—C6—C7    | 118.8 (2)   |
| C14—N4—Fe1 | 106.38 (14) | H6—C6—C7    | 120.59 (15) |
| C20—N4—Fe1 | 105.98 (14) | H6—C6—C5    | 120.59 (15) |
| C20—N4—C14 | 111.77 (18) | C7—C8—N4    | 112.86 (19) |
| C8—N4—Fe1  | 110.46 (14) | H8—C8—N4    | 109.02 (13) |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C8—N4—C14      | 110.93 (18)  | H8—C8—C7        | 109.02 (13)  |
| C8—N4—C20      | 111.10 (19)  | H7—C8—N4        | 109.02 (13)  |
| C7—N3—Fe1      | 115.12 (16)  | H7—C8—C7        | 109.02 (14)  |
| C3—N3—Fe1      | 126.60 (16)  | H7—C8—H8        | 107.8        |
| C3—N3—C7       | 118.2 (2)    | C17—C16—C15     | 119.0 (2)    |
| C13—N5—Fe1     | 113.03 (15)  | H16—C16—C15     | 120.50 (15)  |
| C9—N5—Fe1      | 128.45 (17)  | H16—C16—C17     | 120.50 (15)  |
| C9—N5—C13      | 118.5 (2)    | C16—C17—C18     | 119.1 (2)    |
| C1—N1—Fe1      | 172.9 (2)    | H17—C17—C18     | 120.45 (15)  |
| C2—N2—Fe1      | 170.48 (19)  | H17—C17—C16     | 120.45 (15)  |
| C17—C18—C19    | 118.7 (2)    | C4—C3—N3        | 122.8 (2)    |
| H18—C18—C19    | 120.64 (14)  | H3—C3—N3        | 118.61 (14)  |
| H18—C18—C17    | 120.64 (15)  | H3—C3—C4        | 118.61 (16)  |
| C18—C19—N6     | 122.2 (2)    | C5—C4—C3        | 118.8 (3)    |
| C20—C19—N6     | 114.6 (2)    | H4—C4—C3        | 120.62 (16)  |
| C20—C19—C18    | 123.2 (2)    | H4—C4—C5        | 120.62 (15)  |
| C12—C13—N5     | 122.5 (2)    | C4—C5—C6        | 119.0 (2)    |
| C14—C13—N5     | 115.6 (2)    | H5—C5—C6        | 120.48 (15)  |
| C14—C13—C12    | 121.9 (2)    | H5—C5—C4        | 120.48 (15)  |
| C11—C12—C13    | 118.6 (2)    | C22—C23—C24     | 118.6 (3)    |
| H12—C12—C13    | 120.70 (14)  | H23—C23—C24     | 120.69 (17)  |
| H12—C12—C11    | 120.70 (15)  | H23—C23—C22     | 120.69 (18)  |
| N1—C1—S1       | 178.3 (2)    | C25—C24—C23     | 118.1 (3)    |
| C13—C14—N4     | 109.51 (19)  | H24—C24—C23     | 120.95 (17)  |
| H14—C14—N4     | 109.77 (12)  | H24—C24—C25     | 120.95 (19)  |
| H14—C14—C13    | 109.77 (13)  | C21—C22—C23     | 118.7 (3)    |
| H13—C14—N4     | 109.77 (12)  | H22—C22—C23     | 120.64 (18)  |
| H13—C14—C13    | 109.77 (13)  | H22—C22—C21     | 120.64 (19)  |
| H13—C14—H14    | 108.2        | C24—C25—N7      | 123.8 (3)    |
| C16—C15—N6     | 122.4 (2)    | H25—C25—N7      | 118.10 (18)  |
| H15—C15—N6     | 118.80 (14)  | H25—C25—C24     | 118.10 (19)  |
| H15—C15—C16    | 118.80 (15)  | C22—C21—N7      | 123.7 (3)    |
| C6—C7—N3       | 122.4 (2)    | H21—C21—N7      | 118.14 (17)  |
| C8—C7—N3       | 116.7 (2)    | H21—C21—C22     | 118.14 (19)  |
| C8—C7—C6       | 120.9 (2)    |                 |              |
| Fe1—N6—C19—C18 | −179.12 (15) | C19—C18—C17—C16 | 1.9 (3)      |
| Fe1—N6—C19—C20 | 3.00 (16)    | C19—C20—N4—C14  | 155.01 (18)  |
| Fe1—N6—C15—C16 | 179.69 (19)  | C19—C20—N4—C8   | −80.5 (2)    |
| Fe1—N4—C14—C13 | −37.16 (14)  | C13—N5—C9—C10   | −1.0 (2)     |
| Fe1—N4—C20—C19 | 39.52 (14)   | C13—C12—C11—C10 | −1.3 (3)     |
| Fe1—N4—C8—C7   | −2.35 (15)   | C13—C14—N4—C20  | −152.40 (18) |
| Fe1—N3—C7—C6   | 177.39 (17)  | C13—C14—N4—C8   | 83.0 (2)     |
| Fe1—N3—C7—C8   | −3.46 (18)   | C12—C13—N5—C9   | 0.4 (3)      |
| Fe1—N3—C3—C4   | −176.8 (2)   | C12—C11—C10—C9  | 0.7 (3)      |
| Fe1—N5—C13—C12 | 179.66 (15)  | C14—N4—C8—C7    | −120.06 (19) |
| Fe1—N5—C13—C14 | −2.61 (16)   | C14—C13—N5—C9   | 178.1 (2)    |
| Fe1—N5—C9—C10  | 179.86 (19)  | C14—C13—C12—C11 | −176.8 (2)   |

|                 |              |                 |            |
|-----------------|--------------|-----------------|------------|
| N6—C19—C18—C17  | −0.9 (3)     | C15—N6—C19—C20  | −178.9 (2) |
| N6—C19—C20—N4   | −29.0 (2)    | C7—N3—C3—C4     | 0.2 (3)    |
| N6—C15—C16—C17  | −0.8 (3)     | C7—C6—C5—C4     | 0.5 (3)    |
| N4—C14—C13—N5   | 27.2 (2)     | C7—C8—N4—C20    | 115.0 (2)  |
| N4—C14—C13—C12  | −155.02 (18) | C20—C19—C18—C17 | 176.8 (2)  |
| N4—C20—C19—C18  | 153.12 (18)  | N7—C25—C24—C23  | −1.1 (4)   |
| N4—C8—C7—N3     | 3.9 (2)      | N7—C21—C22—C23  | −0.3 (4)   |
| N4—C8—C7—C6     | −177.0 (2)   | C6—C7—N3—C3     | 0.1 (3)    |
| N3—C7—C6—C5     | −0.4 (3)     | C6—C5—C4—C3     | −0.2 (3)   |
| N3—C3—C4—C5     | −0.1 (3)     | C8—C7—N3—C3     | 179.3 (2)  |
| N5—C13—C12—C11  | 0.8 (3)      | C8—C7—C6—C5     | −179.5 (3) |
| N5—C9—C10—C11   | 0.4 (3)      | C24—C23—C22—C21 | 0.1 (3)    |
| C18—C19—N6—C15  | −1.0 (3)     | C24—C25—N7—C21  | 1.0 (4)    |
| C18—C17—C16—C15 | −1.1 (3)     | C22—C23—C24—C25 | 0.5 (3)    |
| C19—N6—C15—C16  | 1.8 (2)      | C22—C21—N7—C25  | −0.2 (4)   |

### Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_145K)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.8047$  (9) Å

$b = 12.0082$  (6) Å

$c = 14.9005$  (8) Å

$\beta = 110.601$  (7)°

$V = 2479.6$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.450$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9612 reflections

$\theta = 2.1$ – $30.5^\circ$

$\mu = 0.81$  mm<sup>−1</sup>

$T = 145$  K

Block, red

$0.21 \times 0.08 \times 0.05$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.827$ ,  $T_{\max} = 1.000$

27601 measured reflections

5060 independent reflections

3856 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -18 \rightarrow 18$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.089$

$S = 1.06$

5060 reflections

316 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 2.8669P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.02$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.67$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.33021 (3)  | 0.35779 (3)  | 0.67996 (2)  | 0.01988 (10)                     |
| S2  | 0.51848 (6)  | 0.03519 (6)  | 0.80055 (5)  | 0.03395 (18)                     |
| S1  | 0.21866 (6)  | 0.36386 (7)  | 0.94458 (5)  | 0.0423 (2)                       |
| N3  | 0.38161 (14) | 0.35570 (18) | 0.57253 (13) | 0.0206 (4)                       |
| N6  | 0.42241 (15) | 0.48062 (17) | 0.73925 (14) | 0.0205 (5)                       |
| N5  | 0.21507 (15) | 0.26788 (17) | 0.60300 (13) | 0.0193 (4)                       |
| N4  | 0.24830 (14) | 0.48568 (16) | 0.60643 (14) | 0.0187 (4)                       |
| N1  | 0.27786 (16) | 0.37046 (18) | 0.78499 (15) | 0.0257 (5)                       |
| N2  | 0.40956 (16) | 0.2315 (2)   | 0.74371 (15) | 0.0275 (5)                       |
| C19 | 0.38150 (18) | 0.5829 (2)   | 0.72494 (16) | 0.0207 (5)                       |
| C18 | 0.43474 (19) | 0.6787 (2)   | 0.75992 (18) | 0.0247 (6)                       |
| C12 | 0.04807 (19) | 0.2841 (2)   | 0.50526 (17) | 0.0236 (6)                       |
| C13 | 0.13570 (18) | 0.3301 (2)   | 0.56212 (17) | 0.0209 (5)                       |
| C14 | 0.14656 (18) | 0.4521 (2)   | 0.58502 (18) | 0.0232 (5)                       |
| C1  | 0.25405 (19) | 0.3693 (2)   | 0.85136 (18) | 0.0250 (6)                       |
| C9  | 0.2095 (2)   | 0.1575 (2)   | 0.58744 (17) | 0.0244 (6)                       |
| C7  | 0.34538 (18) | 0.4345 (2)   | 0.50544 (17) | 0.0226 (5)                       |
| C20 | 0.27463 (18) | 0.5833 (2)   | 0.67191 (18) | 0.0229 (6)                       |
| C15 | 0.51732 (19) | 0.4726 (2)   | 0.78777 (18) | 0.0261 (6)                       |
| N7  | 0.2989 (2)   | 0.1501 (2)   | 0.2151 (2)   | 0.0475 (7)                       |
| C10 | 0.1241 (2)   | 0.1056 (2)   | 0.53230 (18) | 0.0274 (6)                       |
| C11 | 0.0417 (2)   | 0.1694 (2)   | 0.49072 (18) | 0.0264 (6)                       |
| C8  | 0.2678 (2)   | 0.5073 (2)   | 0.51616 (18) | 0.0284 (6)                       |
| C17 | 0.5325 (2)   | 0.6695 (2)   | 0.81263 (18) | 0.0281 (6)                       |
| C3  | 0.4505 (2)   | 0.2878 (2)   | 0.56429 (18) | 0.0278 (6)                       |
| C16 | 0.5745 (2)   | 0.5643 (2)   | 0.82598 (18) | 0.0295 (6)                       |
| C6  | 0.3769 (2)   | 0.4471 (2)   | 0.42846 (18) | 0.0303 (6)                       |
| C2  | 0.45425 (19) | 0.1499 (2)   | 0.76913 (18) | 0.0268 (6)                       |
| C4  | 0.4856 (2)   | 0.2957 (3)   | 0.4897 (2)   | 0.0338 (7)                       |
| C5  | 0.4480 (2)   | 0.3775 (3)   | 0.42051 (19) | 0.0348 (7)                       |
| C23 | 0.2155 (2)   | 0.2236 (3)   | 0.3453 (2)   | 0.0407 (8)                       |
| C24 | 0.2792 (2)   | 0.1352 (3)   | 0.3679 (2)   | 0.0418 (8)                       |
| C22 | 0.1940 (2)   | 0.2747 (3)   | 0.2571 (2)   | 0.0421 (8)                       |
| C25 | 0.3192 (2)   | 0.1029 (3)   | 0.3003 (2)   | 0.0453 (8)                       |
| C21 | 0.2373 (2)   | 0.2347 (3)   | 0.1949 (2)   | 0.0432 (8)                       |
| H21 | 0.2195 (2)   | 0.2756 (3)   | 0.1245 (2)   | 0.0518 (9)*                      |
| H22 | 0.1436 (2)   | 0.3457 (3)   | 0.2367 (2)   | 0.0505 (9)*                      |
| H23 | 0.1824 (2)   | 0.2529 (3)   | 0.3968 (2)   | 0.0488 (9)*                      |
| H24 | 0.2979 (2)   | 0.0917 (3)   | 0.4373 (2)   | 0.0502 (9)*                      |
| H25 | 0.3712 (2)   | 0.0333 (3)   | 0.3189 (2)   | 0.0543 (10)*                     |
| H3  | 0.4803 (2)   | 0.2231 (2)   | 0.61927 (18) | 0.0334 (7)*                      |
| H4  | 0.5421 (2)   | 0.2384 (3)   | 0.4853 (2)   | 0.0406 (8)*                      |
| H5  | 0.4744 (2)   | 0.3866 (3)   | 0.36033 (19) | 0.0418 (8)*                      |
| H6  | 0.3454 (2)   | 0.5122 (2)   | 0.37418 (18) | 0.0364 (8)*                      |
| H15 | 0.55130 (19) | 0.3896 (2)   | 0.79769 (18) | 0.0314 (7)*                      |



|     |               |            |              |             |
|-----|---------------|------------|--------------|-------------|
| H16 | 0.6523 (2)    | 0.5542 (2) | 0.86645 (18) | 0.0354 (7)* |
| H17 | 0.5759 (2)    | 0.7440 (2) | 0.84332 (18) | 0.0338 (7)* |
| H18 | 0.39971 (19)  | 0.7611 (2) | 0.74593 (18) | 0.0296 (7)* |
| H9  | 0.2748 (2)    | 0.1063 (2) | 0.61950 (17) | 0.0293 (7)* |
| H10 | 0.1218 (2)    | 0.0146 (2) | 0.52169 (18) | 0.0329 (7)* |
| H11 | −0.0272 (2)   | 0.1302 (2) | 0.44717 (18) | 0.0317 (7)* |
| H12 | −0.01551 (19) | 0.3377 (2) | 0.47216 (17) | 0.0283 (7)* |
| H14 | 0.12503 (18)  | 0.4697 (2) | 0.64789 (18) | 0.0278 (7)* |
| H13 | 0.09874 (18)  | 0.5007 (2) | 0.52306 (18) | 0.0278 (7)* |
| H19 | 0.25314 (18)  | 0.6615 (2) | 0.62991 (18) | 0.0275 (7)* |
| H20 | 0.23600 (18)  | 0.5791 (2) | 0.72365 (18) | 0.0275 (7)* |
| H8  | 0.2002 (2)    | 0.4939 (2) | 0.45387 (18) | 0.0341 (7)* |
| H7  | 0.2892 (2)    | 0.5959 (2) | 0.51516 (18) | 0.0341 (7)* |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.02183 (19) | 0.01972 (19) | 0.01972 (18) | 0.00066 (16) | 0.00933 (14) | −0.00348 (16) |
| S2  | 0.0340 (4)   | 0.0347 (4)   | 0.0359 (4)   | 0.0043 (3)   | 0.0157 (3)   | 0.0053 (3)    |
| S1  | 0.0627 (6)   | 0.0399 (4)   | 0.0292 (4)   | −0.0196 (4)  | 0.0224 (4)   | −0.0085 (3)   |
| N3  | 0.0243 (11)  | 0.0238 (11)  | 0.0157 (10)  | 0.0023 (9)   | 0.0095 (9)   | −0.0010 (9)   |
| N6  | 0.0221 (11)  | 0.0221 (12)  | 0.0164 (10)  | 0.0022 (9)   | 0.0055 (9)   | 0.0012 (9)    |
| N5  | 0.0236 (11)  | 0.0180 (11)  | 0.0150 (10)  | 0.0031 (9)   | 0.0053 (9)   | −0.0007 (8)   |
| N4  | 0.0212 (11)  | 0.0156 (11)  | 0.0186 (10)  | 0.0020 (9)   | 0.0060 (9)   | −0.0002 (8)   |
| N1  | 0.0314 (12)  | 0.0226 (12)  | 0.0248 (11)  | −0.0008 (10) | 0.0121 (10)  | −0.0004 (9)   |
| N2  | 0.0283 (12)  | 0.0277 (13)  | 0.0255 (12)  | 0.0039 (11)  | 0.0084 (10)  | −0.0008 (10)  |
| C19 | 0.0255 (14)  | 0.0186 (13)  | 0.0180 (12)  | 0.0012 (11)  | 0.0075 (11)  | 0.0004 (10)   |
| C18 | 0.0258 (15)  | 0.0236 (14)  | 0.0250 (13)  | −0.0008 (11) | 0.0094 (12)  | −0.0031 (11)  |
| C12 | 0.0242 (14)  | 0.0215 (13)  | 0.0226 (12)  | −0.0004 (11) | 0.0051 (11)  | −0.0022 (11)  |
| C13 | 0.0230 (13)  | 0.0198 (13)  | 0.0196 (12)  | 0.0011 (11)  | 0.0069 (11)  | −0.0032 (10)  |
| C14 | 0.0224 (13)  | 0.0178 (13)  | 0.0280 (14)  | 0.0044 (11)  | 0.0071 (11)  | −0.0017 (11)  |
| C1  | 0.0317 (14)  | 0.0180 (13)  | 0.0238 (13)  | −0.0032 (11) | 0.0079 (11)  | −0.0007 (11)  |
| C9  | 0.0351 (16)  | 0.0188 (13)  | 0.0188 (12)  | 0.0038 (12)  | 0.0089 (11)  | −0.0003 (10)  |
| C7  | 0.0271 (14)  | 0.0254 (14)  | 0.0156 (12)  | −0.0027 (11) | 0.0081 (11)  | 0.0000 (10)   |
| C20 | 0.0249 (14)  | 0.0152 (12)  | 0.0262 (13)  | 0.0029 (11)  | 0.0059 (11)  | −0.0034 (10)  |
| C15 | 0.0238 (14)  | 0.0304 (15)  | 0.0231 (13)  | 0.0030 (12)  | 0.0067 (11)  | 0.0049 (11)   |
| N7  | 0.0530 (17)  | 0.0535 (18)  | 0.0447 (16)  | −0.0046 (15) | 0.0281 (14)  | −0.0112 (14)  |
| C10 | 0.0394 (17)  | 0.0188 (13)  | 0.0239 (13)  | −0.0017 (12) | 0.0110 (12)  | −0.0017 (11)  |
| C11 | 0.0312 (15)  | 0.0236 (14)  | 0.0241 (13)  | −0.0051 (12) | 0.0093 (12)  | −0.0023 (11)  |
| C8  | 0.0322 (15)  | 0.0312 (15)  | 0.0212 (13)  | 0.0071 (12)  | 0.0085 (12)  | 0.0066 (11)   |
| C17 | 0.0296 (16)  | 0.0318 (16)  | 0.0223 (13)  | −0.0063 (12) | 0.0083 (12)  | −0.0044 (11)  |
| C3  | 0.0322 (15)  | 0.0317 (15)  | 0.0228 (13)  | 0.0052 (12)  | 0.0138 (12)  | 0.0003 (11)   |
| C16 | 0.0210 (14)  | 0.0408 (17)  | 0.0235 (13)  | −0.0007 (13) | 0.0037 (11)  | 0.0016 (12)   |
| C6  | 0.0365 (16)  | 0.0385 (16)  | 0.0184 (13)  | −0.0027 (13) | 0.0126 (12)  | 0.0030 (12)   |
| C2  | 0.0256 (14)  | 0.0346 (16)  | 0.0201 (12)  | −0.0051 (13) | 0.0080 (11)  | −0.0010 (12)  |
| C4  | 0.0320 (16)  | 0.0467 (18)  | 0.0300 (15)  | 0.0062 (14)  | 0.0200 (13)  | −0.0026 (13)  |
| C5  | 0.0372 (16)  | 0.052 (2)    | 0.0217 (14)  | −0.0002 (14) | 0.0179 (12)  | 0.0000 (13)   |
| C23 | 0.0402 (18)  | 0.0454 (19)  | 0.0445 (18)  | −0.0098 (15) | 0.0250 (15)  | −0.0136 (16)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C24 | 0.0498 (19) | 0.0384 (18) | 0.0407 (18) | −0.0108 (16) | 0.0201 (15) | −0.0046 (15) |
| C22 | 0.0357 (17) | 0.0401 (18) | 0.052 (2)   | −0.0010 (15) | 0.0168 (15) | −0.0044 (16) |
| C25 | 0.050 (2)   | 0.0369 (18) | 0.053 (2)   | 0.0024 (16)  | 0.0239 (17) | −0.0052 (16) |
| C21 | 0.0421 (18) | 0.051 (2)   | 0.0404 (18) | −0.0072 (16) | 0.0189 (15) | −0.0016 (16) |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N3    | 1.9998 (19) | C7—C6       | 1.390 (3)   |
| Fe1—N6    | 1.993 (2)   | C20—H19     | 1.1110      |
| Fe1—N5    | 2.003 (2)   | C20—H20     | 1.1110      |
| Fe1—N4    | 2.023 (2)   | C15—C16     | 1.384 (4)   |
| Fe1—N1    | 1.982 (2)   | C15—H15     | 1.1030      |
| Fe1—N2    | 1.949 (2)   | N7—C25      | 1.326 (4)   |
| S2—C2     | 1.645 (3)   | N7—C21      | 1.327 (4)   |
| S1—C1     | 1.649 (3)   | C10—C11     | 1.388 (4)   |
| N3—C7     | 1.344 (3)   | C10—H10     | 1.1030      |
| N3—C3     | 1.344 (3)   | C11—H11     | 1.1030      |
| N6—C19    | 1.352 (3)   | C8—H8       | 1.1110      |
| N6—C15    | 1.338 (3)   | C8—H7       | 1.1110      |
| N5—C13    | 1.344 (3)   | C17—C16     | 1.391 (4)   |
| N5—C9     | 1.343 (3)   | C17—H17     | 1.1030      |
| N4—C14    | 1.482 (3)   | C3—C4       | 1.386 (4)   |
| N4—C20    | 1.487 (3)   | C3—H3       | 1.1030      |
| N4—C8     | 1.494 (3)   | C16—H16     | 1.1030      |
| N1—C1     | 1.161 (3)   | C6—C5       | 1.382 (4)   |
| N2—C2     | 1.169 (3)   | C6—H6       | 1.1030      |
| C19—C18   | 1.388 (3)   | C4—C5       | 1.390 (4)   |
| C19—C20   | 1.499 (3)   | C4—H4       | 1.1030      |
| C18—C17   | 1.386 (4)   | C5—H5       | 1.1030      |
| C18—H18   | 1.1030      | C23—C24     | 1.380 (5)   |
| C12—C13   | 1.390 (3)   | C23—C22     | 1.383 (4)   |
| C12—C11   | 1.392 (4)   | C23—H23     | 1.1030      |
| C12—H12   | 1.1030      | C24—C25     | 1.391 (4)   |
| C13—C14   | 1.500 (3)   | C24—H24     | 1.1030      |
| C14—H14   | 1.1110      | C22—C21     | 1.384 (4)   |
| C14—H13   | 1.1110      | C22—H22     | 1.1030      |
| C9—C10    | 1.390 (4)   | C25—H25     | 1.1030      |
| C9—H9     | 1.1030      | C21—H21     | 1.1030      |
| C7—C8     | 1.497 (4)   |             |             |
| N6—Fe1—N3 | 88.28 (8)   | C19—C20—N4  | 109.1 (2)   |
| N5—Fe1—N3 | 91.25 (8)   | H19—C20—N4  | 109.85 (12) |
| N5—Fe1—N6 | 164.84 (8)  | H19—C20—C19 | 109.85 (13) |
| N4—Fe1—N3 | 84.29 (8)   | H20—C20—N4  | 109.85 (13) |
| N4—Fe1—N6 | 82.23 (8)   | H20—C20—C19 | 109.85 (13) |
| N4—Fe1—N5 | 82.64 (8)   | H20—C20—H19 | 108.3       |
| N1—Fe1—N3 | 176.26 (9)  | C16—C15—N6  | 122.6 (3)   |
| N1—Fe1—N6 | 89.45 (8)   | H15—C15—N6  | 118.70 (15) |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N1—Fe1—N5   | 90.17 (8)   | H15—C15—C16 | 118.70 (16) |
| N1—Fe1—N4   | 92.46 (8)   | C21—N7—C25  | 116.8 (3)   |
| N2—Fe1—N3   | 92.66 (9)   | C11—C10—C9  | 119.3 (2)   |
| N2—Fe1—N6   | 99.12 (9)   | H10—C10—C9  | 120.33 (16) |
| N2—Fe1—N5   | 96.04 (9)   | H10—C10—C11 | 120.33 (16) |
| N2—Fe1—N4   | 176.64 (8)  | C10—C11—C12 | 118.4 (3)   |
| N2—Fe1—N1   | 90.63 (9)   | H11—C11—C12 | 120.79 (16) |
| C7—N3—Fe1   | 114.96 (16) | H11—C11—C10 | 120.79 (16) |
| C3—N3—Fe1   | 126.52 (17) | C7—C8—N4    | 113.1 (2)   |
| C3—N3—C7    | 118.4 (2)   | H8—C8—N4    | 108.95 (13) |
| C19—N6—Fe1  | 113.78 (16) | H8—C8—C7    | 108.95 (14) |
| C15—N6—Fe1  | 127.68 (18) | H7—C8—N4    | 108.95 (14) |
| C15—N6—C19  | 118.5 (2)   | H7—C8—C7    | 108.95 (15) |
| C13—N5—Fe1  | 113.09 (16) | H7—C8—H8    | 107.8       |
| C9—N5—Fe1   | 128.27 (18) | C16—C17—C18 | 118.6 (3)   |
| C9—N5—C13   | 118.6 (2)   | H17—C17—C18 | 120.72 (16) |
| C14—N4—Fe1  | 106.26 (15) | H17—C17—C16 | 120.72 (16) |
| C20—N4—Fe1  | 105.91 (14) | C4—C3—N3    | 122.8 (3)   |
| C20—N4—C14  | 111.60 (19) | H3—C3—N3    | 118.59 (14) |
| C8—N4—Fe1   | 110.53 (15) | H3—C3—C4    | 118.59 (17) |
| C8—N4—C14   | 110.92 (19) | C17—C16—C15 | 119.1 (2)   |
| C8—N4—C20   | 111.4 (2)   | H16—C16—C15 | 120.47 (16) |
| C1—N1—Fe1   | 172.8 (2)   | H16—C16—C17 | 120.47 (16) |
| C2—N2—Fe1   | 170.5 (2)   | C5—C6—C7    | 119.4 (3)   |
| C18—C19—N6  | 122.0 (2)   | H6—C6—C7    | 120.29 (16) |
| C20—C19—N6  | 114.6 (2)   | H6—C6—C5    | 120.29 (16) |
| C20—C19—C18 | 123.3 (2)   | N2—C2—S2    | 177.4 (2)   |
| C17—C18—C19 | 119.2 (2)   | C5—C4—C3    | 118.5 (3)   |
| H18—C18—C19 | 120.38 (15) | H4—C4—C3    | 120.73 (17) |
| H18—C18—C17 | 120.38 (16) | H4—C4—C5    | 120.73 (16) |
| C11—C12—C13 | 119.0 (2)   | C4—C5—C6    | 118.9 (2)   |
| H12—C12—C13 | 120.48 (15) | H5—C5—C6    | 120.57 (16) |
| H12—C12—C11 | 120.48 (16) | H5—C5—C4    | 120.57 (16) |
| C12—C13—N5  | 122.3 (2)   | C22—C23—C24 | 118.8 (3)   |
| C14—C13—N5  | 115.7 (2)   | H23—C23—C24 | 120.60 (19) |
| C14—C13—C12 | 122.0 (2)   | H23—C23—C22 | 120.60 (19) |
| C13—C14—N4  | 109.6 (2)   | C25—C24—C23 | 117.8 (3)   |
| H14—C14—N4  | 109.76 (12) | H24—C24—C23 | 121.11 (19) |
| H14—C14—C13 | 109.76 (14) | H24—C24—C25 | 121.1 (2)   |
| H13—C14—N4  | 109.76 (12) | C21—C22—C23 | 118.5 (3)   |
| H13—C14—C13 | 109.76 (14) | H22—C22—C23 | 120.77 (19) |
| H13—C14—H14 | 108.2       | H22—C22—C21 | 120.8 (2)   |
| N1—C1—S1    | 178.2 (2)   | C24—C25—N7  | 124.3 (3)   |
| C10—C9—N5   | 122.2 (3)   | H25—C25—N7  | 117.83 (19) |
| H9—C9—N5    | 118.90 (15) | H25—C25—C24 | 117.8 (2)   |
| H9—C9—C10   | 118.90 (16) | C22—C21—N7  | 123.8 (3)   |
| C8—C7—N3    | 116.9 (2)   | H21—C21—N7  | 118.08 (18) |
| C6—C7—N3    | 121.9 (2)   | H21—C21—C22 | 118.1 (2)   |

|                 |              |                 |            |
|-----------------|--------------|-----------------|------------|
| C6—C7—C8        | 121.2 (2)    |                 |            |
| Fe1—N3—C7—C8    | −4.0 (2)     | C19—C20—N4—C8   | −80.4 (2)  |
| Fe1—N3—C7—C6    | 177.15 (18)  | C18—C19—N6—C15  | −0.7 (3)   |
| Fe1—N3—C3—C4    | −176.6 (2)   | C18—C17—C16—C15 | −1.1 (3)   |
| Fe1—N6—C19—C18  | −179.18 (16) | C12—C13—N5—C9   | 0.7 (3)    |
| Fe1—N6—C19—C20  | 2.91 (17)    | C12—C11—C10—C9  | 0.6 (3)    |
| Fe1—N6—C15—C16  | 180.0 (2)    | C13—N5—C9—C10   | −1.4 (2)   |
| Fe1—N5—C13—C12  | 179.67 (16)  | C13—C12—C11—C10 | −1.2 (3)   |
| Fe1—N5—C13—C14  | −2.74 (17)   | C13—C14—N4—C20  | −152.7 (2) |
| Fe1—N5—C9—C10   | 179.8 (2)    | C13—C14—N4—C8   | 82.5 (2)   |
| Fe1—N4—C14—C13  | −37.69 (15)  | C14—N4—C8—C7    | −120.7 (2) |
| Fe1—N4—C20—C19  | 39.81 (15)   | C14—C13—N5—C9   | 178.2 (2)  |
| Fe1—N4—C8—C7    | −3.06 (16)   | C14—C13—C12—C11 | −176.8 (2) |
| N3—C7—C8—N4     | 4.7 (2)      | C7—N3—C3—C4     | 0.0 (3)    |
| N3—C7—C6—C5     | −0.5 (3)     | C7—C8—N4—C20    | 114.4 (2)  |
| N3—C3—C4—C5     | 0.1 (3)      | C7—C6—C5—C4     | 0.5 (3)    |
| N6—C19—C18—C17  | −1.2 (3)     | C20—C19—N6—C15  | −178.7 (2) |
| N6—C19—C20—N4   | −29.2 (2)    | C20—C19—C18—C17 | 176.5 (2)  |
| N6—C15—C16—C17  | −0.9 (3)     | N7—C25—C24—C23  | −1.2 (4)   |
| N5—C13—C12—C11  | 0.6 (3)      | N7—C21—C22—C23  | −0.4 (4)   |
| N5—C13—C14—N4   | 27.8 (2)     | C8—C7—N3—C3     | 179.1 (2)  |
| N5—C9—C10—C11   | 0.8 (3)      | C8—C7—C6—C5     | −179.3 (3) |
| N4—C14—C13—C12  | −154.63 (19) | C3—N3—C7—C6     | 0.2 (3)    |
| N4—C20—C19—C18  | 152.92 (19)  | C3—C4—C5—C6     | −0.3 (3)   |
| N4—C8—C7—C6     | −176.4 (2)   | C24—C23—C22—C21 | 0.2 (3)    |
| C19—N6—C15—C16  | 1.8 (3)      | C24—C25—N7—C21  | 1.0 (4)    |
| C19—C18—C17—C16 | 2.1 (3)      | C22—C23—C24—C25 | 0.5 (3)    |
| C19—C20—N4—C14  | 155.0 (2)    | C22—C21—N7—C25  | −0.2 (4)   |

# Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_150K)

## Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.8800$  (13) Å

$b = 11.9936$  (8) Å

$c = 14.9016$  (11) Å

$\beta = 110.451$  (9)°

$V = 2491.8$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.443$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9582 reflections

$\theta = 2.1$ – $29.3^\circ$

$\mu = 0.80$  mm<sup>−1</sup>

$T = 150$  K

Block, red

$0.21 \times 0.08 \times 0.05$  mm

## Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.826$ ,  $T_{\max} = 1.000$

27854 measured reflections

5097 independent reflections

3866 reflections with  $I \geq 2\sigma(I)$  $R_{\text{int}} = 0.035$  $\theta_{\text{max}} = 26.4^\circ$ ,  $\theta_{\text{min}} = 2.2^\circ$  $h = -18 \rightarrow 18$  $k = -14 \rightarrow 14$  $l = -18 \rightarrow 18$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.045$  $wR(F^2) = 0.092$  $S = 1.06$ 

5097 reflections

316 parameters

0 restraints

43 constraints

Primary atom site location: dual

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 3.5547P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.0002$  $\Delta\rho_{\text{max}} = 1.00 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.93 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Fe1 | 0.66902 (3)  | 0.35640 (3)  | 0.31857 (3)  | 0.02270 (11)                     |
| S2  | 0.48116 (6)  | 0.03263 (7)  | 0.19895 (6)  | 0.0380 (2)                       |
| S1  | 0.78179 (7)  | 0.36497 (8)  | 0.05452 (6)  | 0.0462 (2)                       |
| N4  | 0.75100 (15) | 0.48580 (17) | 0.39314 (14) | 0.0198 (5)                       |
| N3  | 0.61792 (15) | 0.35460 (19) | 0.42740 (14) | 0.0223 (5)                       |
| N6  | 0.57677 (16) | 0.48163 (18) | 0.26020 (14) | 0.0220 (5)                       |
| N5  | 0.78557 (16) | 0.26743 (18) | 0.39688 (14) | 0.0211 (5)                       |
| N1  | 0.72160 (17) | 0.37062 (19) | 0.21329 (17) | 0.0290 (5)                       |
| N2  | 0.58990 (18) | 0.2289 (2)   | 0.25541 (17) | 0.0326 (6)                       |
| C19 | 0.61795 (19) | 0.5833 (2)   | 0.27534 (17) | 0.0210 (6)                       |
| C18 | 0.5653 (2)   | 0.6798 (2)   | 0.24107 (19) | 0.0263 (6)                       |
| C13 | 0.86410 (19) | 0.3304 (2)   | 0.43732 (18) | 0.0212 (6)                       |
| C7  | 0.65421 (19) | 0.4336 (2)   | 0.49420 (17) | 0.0224 (6)                       |
| C9  | 0.7920 (2)   | 0.1573 (2)   | 0.41262 (18) | 0.0259 (6)                       |
| C20 | 0.72438 (19) | 0.5836 (2)   | 0.32838 (19) | 0.0237 (6)                       |
| C14 | 0.85224 (18) | 0.4528 (2)   | 0.41418 (19) | 0.0237 (6)                       |
| C12 | 0.9515 (2)   | 0.2854 (2)   | 0.49419 (18) | 0.0248 (6)                       |
| C1  | 0.7457 (2)   | 0.3697 (2)   | 0.1472 (2)   | 0.0283 (6)                       |
| C11 | 0.9586 (2)   | 0.1707 (2)   | 0.50906 (19) | 0.0275 (6)                       |
| C15 | 0.4823 (2)   | 0.4733 (3)   | 0.21151 (19) | 0.0296 (6)                       |
| C8  | 0.7318 (2)   | 0.5064 (2)   | 0.48384 (19) | 0.0298 (7)                       |
| N7  | 0.7027 (2)   | 0.1509 (3)   | 0.7849 (2)   | 0.0510 (8)                       |
| C10 | 0.8772 (2)   | 0.1061 (2)   | 0.46757 (19) | 0.0298 (7)                       |
| C3  | 0.5494 (2)   | 0.2868 (3)   | 0.4357 (2)   | 0.0299 (6)                       |
| C17 | 0.4679 (2)   | 0.6709 (3)   | 0.18842 (19) | 0.0306 (7)                       |
| C16 | 0.4257 (2)   | 0.5657 (3)   | 0.17396 (19) | 0.0325 (7)                       |
| C6  | 0.6230 (2)   | 0.4471 (3)   | 0.57117 (19) | 0.0310 (7)                       |
| C5  | 0.5519 (2)   | 0.3771 (3)   | 0.5792 (2)   | 0.0365 (7)                       |
| C2  | 0.5452 (2)   | 0.1472 (3)   | 0.23019 (19) | 0.0307 (6)                       |
| C4  | 0.5144 (2)   | 0.2952 (3)   | 0.5104 (2)   | 0.0362 (7)                       |
| C23 | 0.7853 (2)   | 0.2239 (3)   | 0.6542 (2)   | 0.0419 (8)                       |
| C24 | 0.7220 (2)   | 0.1359 (3)   | 0.6322 (2)   | 0.0446 (8)                       |
| C25 | 0.6818 (3)   | 0.1031 (3)   | 0.6995 (3)   | 0.0472 (9)                       |

|     |              |            |              |              |
|-----|--------------|------------|--------------|--------------|
| C22 | 0.8067 (2)   | 0.2749 (3) | 0.7422 (3)   | 0.0444 (8)   |
| C21 | 0.7638 (2)   | 0.2351 (3) | 0.8047 (2)   | 0.0468 (9)   |
| H3  | 0.5198 (2)   | 0.2219 (3) | 0.3809 (2)   | 0.0358 (8)*  |
| H4  | 0.4581 (2)   | 0.2381 (3) | 0.5151 (2)   | 0.0434 (9)*  |
| H5  | 0.5256 (2)   | 0.3864 (3) | 0.6394 (2)   | 0.0438 (9)*  |
| H6  | 0.6542 (2)   | 0.5125 (3) | 0.62512 (19) | 0.0372 (8)*  |
| H15 | 0.4485 (2)   | 0.3902 (3) | 0.20100 (19) | 0.0355 (8)*  |
| H16 | 0.3484 (2)   | 0.5560 (3) | 0.13324 (19) | 0.0390 (8)*  |
| H17 | 0.4248 (2)   | 0.7458 (3) | 0.15865 (19) | 0.0367 (8)*  |
| H18 | 0.6003 (2)   | 0.7622 (2) | 0.25547 (19) | 0.0316 (7)*  |
| H9  | 0.7274 (2)   | 0.1054 (2) | 0.38084 (18) | 0.0311 (7)*  |
| H10 | 0.8801 (2)   | 0.0149 (2) | 0.47814 (19) | 0.0357 (8)*  |
| H11 | 1.0272 (2)   | 0.1321 (2) | 0.55277 (19) | 0.0330 (8)*  |
| H12 | 1.0142 (2)   | 0.3397 (2) | 0.52702 (18) | 0.0298 (7)*  |
| H25 | 0.6298 (3)   | 0.0337 (3) | 0.6810 (3)   | 0.0567 (10)* |
| H21 | 0.7817 (2)   | 0.2761 (3) | 0.8751 (2)   | 0.0561 (10)* |
| H22 | 0.8569 (2)   | 0.3459 (3) | 0.7623 (3)   | 0.0533 (10)* |
| H23 | 0.8182 (2)   | 0.2530 (3) | 0.6026 (2)   | 0.0503 (10)* |
| H24 | 0.7036 (2)   | 0.0924 (3) | 0.5629 (2)   | 0.0535 (10)* |
| H19 | 0.74556 (19) | 0.6616 (2) | 0.37074 (19) | 0.0284 (7)*  |
| H20 | 0.76284 (19) | 0.5802 (2) | 0.27655 (19) | 0.0284 (7)*  |
| H14 | 0.87342 (18) | 0.4707 (2) | 0.35115 (19) | 0.0285 (7)*  |
| H13 | 0.89956 (18) | 0.5018 (2) | 0.47587 (19) | 0.0285 (7)*  |
| H8  | 0.7989 (2)   | 0.4919 (2) | 0.54575 (19) | 0.0358 (8)*  |
| H7  | 0.7111 (2)   | 0.5951 (2) | 0.48583 (19) | 0.0358 (8)*  |

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Fe1 | 0.0240 (2)  | 0.0227 (2)  | 0.0242 (2)  | 0.00124 (18) | 0.01200 (16) | 0.00655 (17) |
| S2  | 0.0371 (4)  | 0.0416 (5)  | 0.0391 (4)  | −0.0013 (4)  | 0.0180 (4)   | −0.0042 (4)  |
| S1  | 0.0693 (6)  | 0.0416 (5)  | 0.0318 (4)  | 0.0211 (5)   | 0.0227 (4)   | 0.0080 (4)   |
| N4  | 0.0228 (12) | 0.0165 (11) | 0.0204 (11) | −0.0037 (9)  | 0.0079 (9)   | −0.0007 (9)  |
| N3  | 0.0263 (12) | 0.0274 (12) | 0.0152 (10) | −0.0001 (10) | 0.0096 (9)   | −0.0000 (9)  |
| N6  | 0.0242 (12) | 0.0223 (12) | 0.0200 (11) | −0.0031 (10) | 0.0083 (9)   | −0.0033 (9)  |
| N5  | 0.0264 (12) | 0.0192 (11) | 0.0170 (10) | −0.0051 (10) | 0.0069 (9)   | −0.0002 (9)  |
| N1  | 0.0338 (13) | 0.0226 (12) | 0.0328 (13) | 0.0002 (10)  | 0.0141 (11)  | −0.0001 (10) |
| N2  | 0.0314 (14) | 0.0386 (16) | 0.0288 (13) | −0.0010 (12) | 0.0118 (11)  | 0.0056 (12)  |
| C19 | 0.0237 (14) | 0.0201 (14) | 0.0185 (12) | 0.0007 (11)  | 0.0063 (11)  | 0.0020 (10)  |
| C18 | 0.0283 (16) | 0.0232 (15) | 0.0269 (14) | 0.0026 (12)  | 0.0089 (12)  | 0.0050 (11)  |
| C13 | 0.0239 (14) | 0.0167 (13) | 0.0221 (13) | −0.0023 (11) | 0.0069 (11)  | 0.0008 (10)  |
| C7  | 0.0265 (14) | 0.0260 (14) | 0.0157 (12) | 0.0024 (11)  | 0.0085 (11)  | 0.0003 (11)  |
| C9  | 0.0392 (17) | 0.0185 (14) | 0.0194 (12) | −0.0058 (12) | 0.0094 (12)  | 0.0005 (11)  |
| C20 | 0.0266 (15) | 0.0168 (13) | 0.0265 (14) | −0.0013 (11) | 0.0077 (12)  | 0.0022 (11)  |
| C14 | 0.0224 (14) | 0.0167 (13) | 0.0300 (15) | −0.0040 (11) | 0.0065 (12)  | 0.0014 (11)  |
| C12 | 0.0256 (15) | 0.0237 (14) | 0.0236 (13) | 0.0008 (12)  | 0.0068 (12)  | 0.0009 (11)  |
| C1  | 0.0340 (16) | 0.0193 (14) | 0.0278 (15) | 0.0050 (12)  | 0.0060 (13)  | 0.0007 (12)  |
| C11 | 0.0307 (16) | 0.0254 (15) | 0.0252 (14) | 0.0062 (12)  | 0.0080 (12)  | 0.0030 (11)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C15 | 0.0273 (15) | 0.0365 (17) | 0.0248 (14) | −0.0048 (13) | 0.0089 (12) | −0.0076 (13) |
| C8  | 0.0365 (17) | 0.0308 (16) | 0.0226 (14) | −0.0082 (13) | 0.0108 (13) | −0.0084 (12) |
| N7  | 0.0568 (19) | 0.0567 (19) | 0.0493 (18) | 0.0036 (17)  | 0.0307 (15) | 0.0110 (16)  |
| C10 | 0.0450 (19) | 0.0183 (14) | 0.0260 (14) | 0.0024 (13)  | 0.0124 (14) | 0.0021 (11)  |
| C3  | 0.0323 (16) | 0.0365 (17) | 0.0237 (14) | −0.0068 (13) | 0.0134 (12) | 0.0005 (12)  |
| C17 | 0.0295 (16) | 0.0378 (18) | 0.0240 (14) | 0.0085 (13)  | 0.0089 (13) | 0.0061 (12)  |
| C16 | 0.0253 (16) | 0.0440 (19) | 0.0249 (14) | −0.0008 (14) | 0.0045 (12) | −0.0055 (13) |
| C6  | 0.0362 (17) | 0.0395 (17) | 0.0193 (13) | 0.0022 (14)  | 0.0124 (12) | −0.0047 (12) |
| C5  | 0.0400 (17) | 0.051 (2)   | 0.0251 (15) | −0.0008 (15) | 0.0199 (13) | −0.0017 (14) |
| C2  | 0.0279 (15) | 0.0408 (18) | 0.0252 (14) | 0.0057 (15)  | 0.0116 (12) | 0.0019 (14)  |
| C4  | 0.0350 (17) | 0.048 (2)   | 0.0319 (16) | −0.0068 (15) | 0.0192 (14) | 0.0040 (14)  |
| C23 | 0.0420 (19) | 0.046 (2)   | 0.0458 (19) | 0.0121 (16)  | 0.0258 (16) | 0.0148 (16)  |
| C24 | 0.052 (2)   | 0.043 (2)   | 0.0412 (18) | 0.0119 (17)  | 0.0198 (16) | 0.0052 (16)  |
| C25 | 0.051 (2)   | 0.0398 (19) | 0.056 (2)   | −0.0041 (17) | 0.0256 (18) | 0.0068 (17)  |
| C22 | 0.0345 (18) | 0.044 (2)   | 0.056 (2)   | −0.0002 (15) | 0.0179 (16) | 0.0021 (17)  |
| C21 | 0.044 (2)   | 0.059 (2)   | 0.0420 (19) | 0.0068 (18)  | 0.0199 (16) | −0.0011 (17) |

*Geometric parameters (Å, °)*

|         |           |         |           |
|---------|-----------|---------|-----------|
| Fe1—N4  | 2.046 (2) | C14—H13 | 1.1110    |
| Fe1—N3  | 2.018 (2) | C12—C11 | 1.391 (4) |
| Fe1—N6  | 2.016 (2) | C12—H12 | 1.1030    |
| Fe1—N5  | 2.023 (2) | C11—C10 | 1.388 (4) |
| Fe1—N1  | 1.990 (2) | C11—H11 | 1.1030    |
| Fe1—N2  | 1.959 (3) | C15—C16 | 1.386 (4) |
| S2—C2   | 1.644 (3) | C15—H15 | 1.1030    |
| S1—C1   | 1.648 (3) | C8—H8   | 1.1110    |
| N4—C20  | 1.482 (3) | C8—H7   | 1.1110    |
| N4—C14  | 1.481 (3) | N7—C25  | 1.330 (5) |
| N4—C8   | 1.497 (3) | N7—C21  | 1.322 (5) |
| N3—C7   | 1.343 (3) | C10—H10 | 1.1030    |
| N3—C3   | 1.342 (3) | C3—C4   | 1.389 (4) |
| N6—C19  | 1.348 (3) | C3—H3   | 1.1030    |
| N6—C15  | 1.342 (3) | C17—C16 | 1.392 (4) |
| N5—C13  | 1.344 (3) | C17—H17 | 1.1030    |
| N5—C9   | 1.340 (3) | C16—H16 | 1.1030    |
| N1—C1   | 1.160 (3) | C6—C5   | 1.387 (4) |
| N2—C2   | 1.170 (4) | C6—H6   | 1.1030    |
| C19—C18 | 1.391 (4) | C5—C4   | 1.388 (4) |
| C19—C20 | 1.503 (4) | C5—H5   | 1.1030    |
| C18—C17 | 1.390 (4) | C4—H4   | 1.1030    |
| C18—H18 | 1.1030    | C23—C24 | 1.375 (5) |
| C13—C14 | 1.504 (3) | C23—C22 | 1.379 (5) |
| C13—C12 | 1.389 (4) | C23—H23 | 1.1030    |
| C7—C8   | 1.497 (4) | C24—C25 | 1.391 (5) |
| C7—C6   | 1.390 (3) | C24—H24 | 1.1030    |
| C9—C10  | 1.389 (4) | C25—H25 | 1.1030    |
| C9—H9   | 1.1030    | C22—C21 | 1.385 (5) |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C20—H19     | 1.1110      | C22—H22     | 1.1030      |
| C20—H20     | 1.1110      | C21—H21     | 1.1030      |
| C14—H14     | 1.1110      |             |             |
| N3—Fe1—N4   | 83.81 (9)   | H13—C14—C13 | 109.71 (14) |
| N6—Fe1—N4   | 81.72 (8)   | H13—C14—H14 | 108.2       |
| N6—Fe1—N3   | 87.92 (9)   | C11—C12—C13 | 118.9 (3)   |
| N5—Fe1—N4   | 81.87 (9)   | H12—C12—C13 | 120.54 (16) |
| N5—Fe1—N3   | 91.06 (8)   | H12—C12—C11 | 120.54 (17) |
| N5—Fe1—N6   | 163.57 (9)  | N1—C1—S1    | 178.3 (3)   |
| N1—Fe1—N4   | 92.25 (9)   | C10—C11—C12 | 118.4 (3)   |
| N1—Fe1—N3   | 175.58 (9)  | H11—C11—C12 | 120.80 (17) |
| N1—Fe1—N6   | 89.54 (9)   | H11—C11—C10 | 120.80 (17) |
| N1—Fe1—N5   | 90.35 (9)   | C16—C15—N6  | 122.2 (3)   |
| N2—Fe1—N4   | 176.08 (9)  | H15—C15—N6  | 118.90 (16) |
| N2—Fe1—N3   | 92.62 (9)   | H15—C15—C16 | 118.90 (17) |
| N2—Fe1—N6   | 99.80 (9)   | C7—C8—N4    | 113.2 (2)   |
| N2—Fe1—N5   | 96.62 (9)   | H8—C8—N4    | 108.93 (14) |
| N2—Fe1—N1   | 91.38 (10)  | H8—C8—C7    | 108.93 (15) |
| C20—N4—Fe1  | 105.89 (15) | H7—C8—N4    | 108.93 (14) |
| C14—N4—Fe1  | 106.34 (15) | H7—C8—C7    | 108.93 (15) |
| C14—N4—C20  | 111.7 (2)   | H7—C8—H8    | 107.7       |
| C8—N4—Fe1   | 110.48 (15) | C21—N7—C25  | 117.2 (3)   |
| C8—N4—C20   | 111.4 (2)   | C11—C10—C9  | 119.3 (3)   |
| C8—N4—C14   | 110.7 (2)   | H10—C10—C9  | 120.33 (17) |
| C7—N3—Fe1   | 114.91 (17) | H10—C10—C11 | 120.33 (17) |
| C3—N3—Fe1   | 126.28 (18) | C4—C3—N3    | 122.7 (3)   |
| C3—N3—C7    | 118.7 (2)   | H3—C3—N3    | 118.67 (15) |
| C19—N6—Fe1  | 113.62 (17) | H3—C3—C4    | 118.67 (18) |
| C15—N6—Fe1  | 127.23 (19) | C16—C17—C18 | 118.8 (3)   |
| C15—N6—C19  | 119.1 (2)   | H17—C17—C18 | 120.61 (17) |
| C13—N5—Fe1  | 113.47 (17) | H17—C17—C16 | 120.61 (17) |
| C9—N5—Fe1   | 128.01 (19) | C17—C16—C15 | 119.0 (3)   |
| C9—N5—C13   | 118.5 (2)   | H16—C16—C15 | 120.50 (17) |
| C1—N1—Fe1   | 172.7 (2)   | H16—C16—C17 | 120.50 (17) |
| C2—N2—Fe1   | 170.8 (2)   | C5—C6—C7    | 119.0 (3)   |
| C18—C19—N6  | 121.8 (2)   | H6—C6—C7    | 120.49 (17) |
| C20—C19—N6  | 115.1 (2)   | H6—C6—C5    | 120.49 (17) |
| C20—C19—C18 | 123.1 (2)   | C4—C5—C6    | 119.2 (3)   |
| C17—C18—C19 | 119.1 (3)   | H5—C5—C6    | 120.41 (17) |
| H18—C18—C19 | 120.47 (16) | H5—C5—C4    | 120.41 (17) |
| H18—C18—C17 | 120.47 (17) | N2—C2—S2    | 177.5 (3)   |
| C14—C13—N5  | 115.5 (2)   | C5—C4—C3    | 118.4 (3)   |
| C12—C13—N5  | 122.5 (2)   | H4—C4—C3    | 120.79 (18) |
| C12—C13—C14 | 121.9 (2)   | H4—C4—C5    | 120.79 (17) |
| C8—C7—N3    | 117.4 (2)   | C22—C23—C24 | 118.6 (3)   |
| C6—C7—N3    | 122.0 (3)   | H23—C23—C24 | 120.7 (2)   |
| C6—C7—C8    | 120.6 (2)   | H23—C23—C22 | 120.7 (2)   |



|                 |              |                 |            |
|-----------------|--------------|-----------------|------------|
| C10—C9—N5       | 122.3 (3)    | C25—C24—C23     | 118.5 (3)  |
| H9—C9—N5        | 118.85 (16)  | H24—C24—C23     | 120.8 (2)  |
| H9—C9—C10       | 118.85 (17)  | H24—C24—C25     | 120.8 (2)  |
| C19—C20—N4      | 109.5 (2)    | C24—C25—N7      | 123.4 (3)  |
| H19—C20—N4      | 109.78 (13)  | H25—C25—N7      | 118.3 (2)  |
| H19—C20—C19     | 109.78 (14)  | H25—C25—C24     | 118.3 (2)  |
| H20—C20—N4      | 109.78 (13)  | C21—C22—C23     | 118.6 (3)  |
| H20—C20—C19     | 109.78 (14)  | H22—C22—C23     | 120.7 (2)  |
| H20—C20—H19     | 108.2        | H22—C22—C21     | 120.7 (2)  |
| C13—C14—N4      | 109.8 (2)    | C22—C21—N7      | 123.8 (3)  |
| H14—C14—N4      | 109.71 (13)  | H21—C21—N7      | 118.1 (2)  |
| H14—C14—C13     | 109.71 (14)  | H21—C21—C22     | 118.1 (2)  |
| H13—C14—N4      | 109.71 (13)  |                 |            |
| Fe1—N4—C20—C19  | 39.71 (16)   | C19—C20—N4—C8   | −80.5 (2)  |
| Fe1—N4—C14—C13  | −38.02 (15)  | C18—C19—N6—C15  | −0.6 (3)   |
| Fe1—N4—C8—C7    | −3.72 (17)   | C18—C17—C16—C15 | −0.7 (3)   |
| Fe1—N3—C7—C8    | −4.3 (2)     | C13—N5—C9—C10   | −1.2 (3)   |
| Fe1—N3—C7—C6    | 176.75 (19)  | C13—C14—N4—C20  | −153.1 (2) |
| Fe1—N3—C3—C4    | −176.3 (2)   | C13—C14—N4—C8   | 82.0 (2)   |
| Fe1—N6—C19—C18  | −179.44 (17) | C13—C12—C11—C10 | −1.1 (3)   |
| Fe1—N6—C19—C20  | 2.62 (18)    | C7—N3—C3—C4     | 0.1 (3)    |
| Fe1—N6—C15—C16  | −179.7 (2)   | C7—C8—N4—C20    | 113.7 (2)  |
| Fe1—N5—C13—C14  | −2.29 (18)   | C7—C8—N4—C14    | −121.3 (2) |
| Fe1—N5—C13—C12  | 179.77 (16)  | C7—C6—C5—C4     | 0.3 (3)    |
| Fe1—N5—C9—C10   | 179.4 (2)    | C9—N5—C13—C14   | 178.3 (2)  |
| N4—C20—C19—N6   | −29.1 (2)    | C9—N5—C13—C12   | 0.4 (3)    |
| N4—C20—C19—C18  | 153.0 (2)    | C9—C10—C11—C12  | 0.3 (3)    |
| N4—C14—C13—N5   | 27.7 (2)     | C20—C19—N6—C15  | −178.5 (2) |
| N4—C14—C13—C12  | −154.3 (2)   | C20—C19—C18—C17 | 176.7 (3)  |
| N4—C8—C7—N3     | 5.4 (3)      | C14—C13—C12—C11 | −177.0 (2) |
| N4—C8—C7—C6     | −175.7 (2)   | C8—C7—N3—C3     | 178.9 (2)  |
| N3—C7—C6—C5     | −0.2 (3)     | C8—C7—C6—C5     | −179.1 (3) |
| N3—C3—C4—C5     | 0.0 (3)      | N7—C25—C24—C23  | −1.4 (4)   |
| N6—C19—C18—C17  | −1.1 (3)     | N7—C21—C22—C23  | −0.4 (4)   |
| N6—C15—C16—C17  | −1.0 (3)     | C3—N3—C7—C6     | −0.0 (3)   |
| N5—C13—C12—C11  | 0.8 (3)      | C3—C4—C5—C6     | −0.3 (4)   |
| N5—C9—C10—C11   | 0.9 (3)      | C24—C23—C22—C21 | 0.2 (4)    |
| C19—N6—C15—C16  | 1.7 (3)      | C24—C25—N7—C21  | 1.2 (4)    |
| C19—C18—C17—C16 | 1.7 (3)      | C25—N7—C21—C22  | −0.3 (4)   |
| C19—C20—N4—C14  | 155.1 (2)    | C25—C24—C23—C22 | 0.6 (4)    |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_155K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.9190$  (6) Å

$b = 11.9563$  (4) Å

$c = 14.9841$  (7) Å

$\beta = 110.392 (5)^\circ$   
 $V = 2505.30 (19) \text{ \AA}^3$   
 $Z = 4$   
 $F(000) = 1123.013$   
 $D_x = 1.436 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 17021 reflections  
 $\theta = 2.1\text{--}29.7^\circ$   
 $\mu = 0.80 \text{ mm}^{-1}$   
 $T = 155 \text{ K}$   
 , red  
 $0.21 \times 0.08 \times 0.05 \text{ mm}$

#### Data collection

Rigaku XtaLAB Synergy-R  
 diffractometer with a HyPix-Arc 100 detector  
 Radiation source: micro-focus sealed X-ray  
 tube, Mova (Mo) X-ray Source  
 Mirror monochromator  
 Detector resolution:  $10.0000 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: gaussian  
 (CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.841$ ,  $T_{\max} = 1.000$   
 52494 measured reflections  
 6210 independent reflections  
 4672 reflections with  $I \geq 2\sigma(I)$   
 $R_{\text{int}} = 0.038$   
 $\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -19 \rightarrow 19$   
 $k = -15 \rightarrow 15$   
 $l = -19 \rightarrow 19$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.086$   
 $S = 1.07$   
 6210 reflections  
 339 parameters  
 0 restraints

20 constraints  
 Primary atom site location: dual  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0228P)^2 + 2.0442P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.65 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.72 \text{ e \AA}^{-3}$

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| Fe1 | 0.68531 (2)  | 0.64713 (3)  | 0.33312 (2)   | 0.02781 (8)                      |
| S2  | 0.80228 (5)  | 0.97394 (6)  | 0.52011 (5)   | 0.04482 (16)                     |
| S1  | 0.94786 (5)  | 0.63235 (6)  | 0.21655 (6)   | 0.05228 (19)                     |
| N3  | 0.57304 (11) | 0.64696 (15) | 0.38334 (12)  | 0.0255 (4)                       |
| N5  | 0.60408 (11) | 0.73356 (14) | 0.21321 (12)  | 0.0230 (3)                       |
| N4  | 0.60771 (11) | 0.51422 (13) | 0.25109 (11)  | 0.0216 (3)                       |
| N6  | 0.74112 (11) | 0.51665 (14) | 0.42490 (12)  | 0.0249 (4)                       |
| N1  | 0.79199 (15) | 0.62953 (15) | 0.28008 (13)  | 0.0349 (4)                       |
| N2  | 0.74716 (14) | 0.7779 (2)   | 0.41119 (14)  | 0.0428 (5)                       |
| C19 | 0.72453 (14) | 0.41516 (17) | 0.38397 (14)  | 0.0240 (4)                       |
| C12 | 0.50665 (14) | 0.71133 (17) | 0.04932 (14)  | 0.0265 (4)                       |
| H12 | 0.47414 (14) | 0.65516 (17) | −0.01185 (14) | 0.048 (7)*                       |
| C13 | 0.56395 (14) | 0.66832 (16) | 0.13676 (14)  | 0.0218 (4)                       |
| C14 | 0.58810 (15) | 0.54599 (16) | 0.15066 (14)  | 0.0252 (4)                       |
| H14 | 0.65196 (15) | 0.52817 (16) | 0.13105 (14)  | 0.045 (7)*                       |
| H13 | 0.52745 (15) | 0.49566 (16) | 0.10353 (14)  | 0.052 (7)*                       |
| C18 | 0.75711 (15) | 0.31744 (19) | 0.43528 (15)  | 0.0296 (5)                       |
| H18 | 0.74103 (15) | 0.23513 (19) | 0.40003 (15)  | 0.054 (8)*                       |
| C7  | 0.50689 (14) | 0.56780 (17) | 0.34730 (14)  | 0.0260 (4)                       |
| C9  | 0.58733 (14) | 0.84415 (17) | 0.20458 (16)  | 0.0291 (5)                       |
| H9  | 0.61873 (14) | 0.89798 (17) | 0.26757 (16)  | 0.044 (7)*                       |

|     |              |              |               |             |
|-----|--------------|--------------|---------------|-------------|
| N7  | 0.21547 (17) | 0.8473 (2)   | 0.29412 (17)  | 0.0551 (6)  |
| C20 | 0.67121 (15) | 0.41530 (17) | 0.27829 (14)  | 0.0272 (4)  |
| H19 | 0.62794 (15) | 0.33771 (17) | 0.25754 (14)  | 0.049 (7)*  |
| H20 | 0.72295 (15) | 0.41702 (17) | 0.23999 (14)  | 0.049 (7)*  |
| C10 | 0.53191 (15) | 0.89298 (18) | 0.11918 (17)  | 0.0328 (5)  |
| H10 | 0.52063 (15) | 0.98428 (18) | 0.11434 (17)  | 0.054 (8)*  |
| C15 | 0.79007 (15) | 0.5238 (2)   | 0.51828 (15)  | 0.0341 (5)  |
| H15 | 0.80181 (15) | 0.6070 (2)   | 0.55223 (15)  | 0.047 (7)*  |
| C11 | 0.49084 (15) | 0.82574 (18) | 0.03992 (16)  | 0.0317 (5)  |
| H11 | 0.44667 (15) | 0.86239 (18) | −0.02892 (16) | 0.056 (8)*  |
| C8  | 0.51702 (15) | 0.4956 (2)   | 0.26941 (16)  | 0.0324 (5)  |
| H8  | 0.45584 (15) | 0.5122 (2)   | 0.20276 (16)  | 0.075 (9)*  |
| H7  | 0.51323 (15) | 0.4064 (2)   | 0.28859 (16)  | 0.067 (9)*  |
| C1  | 0.85720 (17) | 0.62959 (17) | 0.25435 (16)  | 0.0327 (5)  |
| C6  | 0.42977 (15) | 0.5536 (2)   | 0.37787 (16)  | 0.0337 (5)  |
| H6  | 0.37610 (15) | 0.4880 (2)   | 0.34635 (16)  | 0.055 (8)*  |
| C17 | 0.81028 (15) | 0.3253 (2)   | 0.53172 (16)  | 0.0348 (5)  |
| H17 | 0.83902 (15) | 0.2496 (2)   | 0.57397 (16)  | 0.056 (8)*  |
| C3  | 0.56400 (16) | 0.7150 (2)   | 0.45128 (16)  | 0.0337 (5)  |
| H3  | 0.61816 (16) | 0.7807 (2)   | 0.48074 (16)  | 0.056 (8)*  |
| C16 | 0.82668 (16) | 0.4304 (2)   | 0.57415 (16)  | 0.0388 (6)  |
| H16 | 0.86801 (16) | 0.4393 (2)   | 0.65077 (16)  | 0.054 (8)*  |
| C2  | 0.77135 (16) | 0.8599 (2)   | 0.45609 (16)  | 0.0372 (5)  |
| C5  | 0.42121 (16) | 0.6230 (2)   | 0.44847 (17)  | 0.0406 (6)  |
| H5  | 0.36120 (16) | 0.6128 (2)   | 0.47472 (17)  | 0.062 (8)*  |
| C4  | 0.48939 (17) | 0.7056 (2)   | 0.48562 (17)  | 0.0413 (6)  |
| H4  | 0.48423 (17) | 0.7627 (2)   | 0.54145 (17)  | 0.059 (8)*  |
| C24 | 0.36798 (19) | 0.8636 (2)   | 0.27605 (19)  | 0.0475 (6)  |
| H24 | 0.43708 (19) | 0.9076 (2)   | 0.29487 (19)  | 0.065 (9)*  |
| C23 | 0.34690 (19) | 0.7750 (2)   | 0.21340 (18)  | 0.0449 (6)  |
| H23 | 0.39900 (19) | 0.7460 (2)   | 0.18129 (18)  | 0.065 (8)*  |
| C25 | 0.3002 (2)   | 0.8961 (2)   | 0.3149 (2)    | 0.0512 (7)  |
| H25 | 0.3176 (2)   | 0.9664 (2)   | 0.3658 (2)    | 0.098 (12)* |
| C22 | 0.2592 (2)   | 0.7233 (2)   | 0.19167 (18)  | 0.0471 (6)  |
| H22 | 0.2397 (2)   | 0.6521 (2)   | 0.14179 (18)  | 0.070 (9)*  |
| C21 | 0.19623 (19) | 0.7627 (3)   | 0.23381 (19)  | 0.0495 (7)  |
| H21 | 0.12616 (19) | 0.7212 (3)   | 0.21599 (19)  | 0.054 (7)*  |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Fe1 | 0.03237 (17) | 0.02767 (16) | 0.02766 (15) | 0.01185 (14) | 0.01583 (12) | 0.00394 (13) |
| S2  | 0.0442 (4)   | 0.0540 (4)   | 0.0407 (3)   | −0.0012 (3)  | 0.0203 (3)   | 0.0040 (3)   |
| S1  | 0.0359 (3)   | 0.0458 (4)   | 0.0761 (5)   | 0.0080 (3)   | 0.0208 (3)   | 0.0248 (4)   |
| N3  | 0.0192 (8)   | 0.0289 (9)   | 0.0301 (9)   | −0.0003 (7)  | 0.0109 (7)   | 0.0001 (8)   |
| N5  | 0.0194 (8)   | 0.0192 (8)   | 0.0293 (9)   | 0.0001 (6)   | 0.0072 (7)   | −0.0043 (7)  |
| N4  | 0.0215 (8)   | 0.0186 (8)   | 0.0243 (8)   | −0.0002 (6)  | 0.0074 (7)   | −0.0040 (7)  |
| N6  | 0.0223 (8)   | 0.0261 (9)   | 0.0273 (9)   | −0.0040 (7)  | 0.0099 (7)   | −0.0047 (7)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| N1  | 0.0468 (12) | 0.0242 (10) | 0.0348 (10) | 0.0024 (8)   | 0.0158 (9)  | 0.0018 (8)   |
| N2  | 0.0385 (11) | 0.0546 (14) | 0.0374 (11) | 0.0135 (10)  | 0.0161 (9)  | 0.0055 (11)  |
| C19 | 0.0241 (10) | 0.0214 (10) | 0.0261 (10) | 0.0011 (8)   | 0.0082 (8)  | −0.0013 (8)  |
| C12 | 0.0265 (10) | 0.0245 (11) | 0.0264 (10) | 0.0020 (8)   | 0.0066 (8)  | −0.0001 (8)  |
| C13 | 0.0234 (9)  | 0.0173 (10) | 0.0243 (10) | 0.0010 (7)   | 0.0077 (8)  | −0.0011 (8)  |
| C14 | 0.0310 (11) | 0.0181 (10) | 0.0245 (10) | 0.0012 (8)   | 0.0074 (8)  | −0.0039 (8)  |
| C18 | 0.0284 (11) | 0.0279 (11) | 0.0327 (12) | 0.0057 (9)   | 0.0108 (9)  | 0.0049 (9)   |
| C7  | 0.0190 (9)  | 0.0296 (11) | 0.0309 (11) | −0.0003 (8)  | 0.0107 (8)  | 0.0033 (9)   |
| C9  | 0.0215 (9)  | 0.0201 (10) | 0.0445 (13) | −0.0001 (8)  | 0.0100 (9)  | −0.0063 (9)  |
| N7  | 0.0524 (14) | 0.0606 (16) | 0.0628 (15) | 0.0117 (12)  | 0.0333 (12) | 0.0030 (13)  |
| C20 | 0.0327 (11) | 0.0194 (10) | 0.0275 (11) | 0.0028 (8)   | 0.0078 (9)  | −0.0027 (8)  |
| C10 | 0.0258 (11) | 0.0203 (10) | 0.0512 (14) | 0.0019 (8)   | 0.0121 (10) | 0.0033 (10)  |
| C15 | 0.0300 (11) | 0.0425 (14) | 0.0292 (11) | −0.0113 (10) | 0.0096 (9)  | −0.0077 (10) |
| C11 | 0.0289 (11) | 0.0287 (12) | 0.0374 (12) | 0.0040 (9)   | 0.0114 (9)  | 0.0078 (9)   |
| C8  | 0.0261 (11) | 0.0345 (13) | 0.0384 (12) | −0.0081 (9)  | 0.0136 (9)  | −0.0094 (10) |
| C1  | 0.0361 (12) | 0.0209 (11) | 0.0368 (12) | 0.0014 (9)   | 0.0072 (10) | 0.0038 (9)   |
| C6  | 0.0216 (10) | 0.0431 (13) | 0.0392 (12) | −0.0025 (9)  | 0.0139 (9)  | 0.0028 (10)  |
| C17 | 0.0252 (11) | 0.0475 (15) | 0.0306 (12) | 0.0067 (10)  | 0.0081 (9)  | 0.0087 (10)  |
| C3  | 0.0279 (11) | 0.0400 (13) | 0.0357 (12) | −0.0008 (10) | 0.0140 (9)  | −0.0069 (10) |
| C16 | 0.0281 (11) | 0.0585 (17) | 0.0258 (11) | −0.0069 (11) | 0.0042 (9)  | 0.0008 (11)  |
| C2  | 0.0302 (11) | 0.0531 (16) | 0.0314 (12) | 0.0081 (11)  | 0.0144 (9)  | 0.0098 (12)  |
| C5  | 0.0282 (12) | 0.0586 (17) | 0.0429 (13) | −0.0001 (11) | 0.0225 (10) | 0.0005 (12)  |
| C4  | 0.0354 (13) | 0.0537 (16) | 0.0409 (13) | 0.0032 (11)  | 0.0210 (11) | −0.0065 (12) |
| C24 | 0.0432 (14) | 0.0469 (16) | 0.0547 (16) | 0.0005 (12)  | 0.0200 (12) | 0.0098 (13)  |
| C23 | 0.0508 (16) | 0.0476 (15) | 0.0450 (14) | 0.0152 (13)  | 0.0276 (12) | 0.0129 (12)  |
| C25 | 0.0618 (18) | 0.0425 (15) | 0.0553 (16) | 0.0069 (13)  | 0.0278 (14) | −0.0029 (13) |
| C22 | 0.0598 (17) | 0.0463 (16) | 0.0379 (13) | 0.0027 (13)  | 0.0205 (12) | 0.0022 (12)  |
| C21 | 0.0435 (15) | 0.0603 (18) | 0.0471 (15) | −0.0020 (13) | 0.0189 (12) | 0.0072 (14)  |

*Geometric parameters (Å, °)*

|        |             |         |           |
|--------|-------------|---------|-----------|
| Fe1—N3 | 2.0615 (16) | C9—C10  | 1.387 (3) |
| Fe1—N5 | 2.0609 (17) | N7—C25  | 1.327 (4) |
| Fe1—N4 | 2.0940 (16) | N7—C21  | 1.319 (4) |
| Fe1—N6 | 2.0543 (18) | C20—H19 | 1.1110    |
| Fe1—N1 | 2.023 (2)   | C20—H20 | 1.1110    |
| Fe1—N2 | 1.978 (2)   | C10—H10 | 1.1030    |
| S2—C2  | 1.638 (3)   | C10—C11 | 1.388 (3) |
| S1—C1  | 1.639 (3)   | C15—H15 | 1.1030    |
| N3—C7  | 1.338 (3)   | C15—C16 | 1.388 (3) |
| N3—C3  | 1.346 (3)   | C11—H11 | 1.1030    |
| N5—C13 | 1.342 (2)   | C8—H8   | 1.1110    |
| N5—C9  | 1.343 (3)   | C8—H7   | 1.1110    |
| N4—C14 | 1.479 (2)   | C6—H6   | 1.1030    |
| N4—C20 | 1.481 (2)   | C6—C5   | 1.385 (3) |
| N4—C8  | 1.488 (2)   | C17—H17 | 1.1030    |
| N6—C19 | 1.343 (2)   | C17—C16 | 1.390 (3) |
| N6—C15 | 1.337 (3)   | C3—H3   | 1.1030    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| N1—C1      | 1.165 (3)   | C3—C4       | 1.384 (3)   |
| N2—C2      | 1.173 (3)   | C16—H16     | 1.1030      |
| C19—C18    | 1.390 (3)   | C5—H5       | 1.1030      |
| C19—C20    | 1.504 (3)   | C5—C4       | 1.388 (3)   |
| C12—H12    | 1.1030      | C4—H4       | 1.1030      |
| C12—C13    | 1.390 (3)   | C24—H24     | 1.1030      |
| C12—C11    | 1.387 (3)   | C24—C23     | 1.377 (4)   |
| C13—C14    | 1.503 (3)   | C24—C25     | 1.387 (4)   |
| C14—H14    | 1.1110      | C23—H23     | 1.1030      |
| C14—H13    | 1.1110      | C23—C22     | 1.379 (4)   |
| C18—H18    | 1.1030      | C25—H25     | 1.1030      |
| C18—C17    | 1.388 (3)   | C22—H22     | 1.1030      |
| C7—C8      | 1.501 (3)   | C22—C21     | 1.385 (4)   |
| C7—C6      | 1.389 (3)   | C21—H21     | 1.1030      |
| C9—H9      | 1.1030      |             |             |
| N5—Fe1—N3  | 90.74 (6)   | C19—C20—N4  | 110.09 (16) |
| N4—Fe1—N3  | 81.96 (7)   | H19—C20—N4  | 109.64 (10) |
| N4—Fe1—N5  | 80.32 (6)   | H19—C20—C19 | 109.64 (11) |
| N6—Fe1—N3  | 86.93 (6)   | H20—C20—N4  | 109.64 (10) |
| N6—Fe1—N5  | 160.47 (7)  | H20—C20—C19 | 109.64 (11) |
| N6—Fe1—N4  | 80.15 (6)   | H20—C20—H19 | 108.2       |
| N1—Fe1—N3  | 173.73 (7)  | H10—C10—C9  | 120.36 (13) |
| N1—Fe1—N5  | 90.78 (7)   | C11—C10—C9  | 119.3 (2)   |
| N1—Fe1—N4  | 92.31 (7)   | C11—C10—H10 | 120.36 (13) |
| N1—Fe1—N6  | 89.61 (7)   | H15—C15—N6  | 118.78 (13) |
| N2—Fe1—N3  | 93.21 (7)   | C16—C15—N6  | 122.4 (2)   |
| N2—Fe1—N5  | 97.51 (7)   | C16—C15—H15 | 118.78 (14) |
| N2—Fe1—N4  | 174.65 (7)  | C10—C11—C12 | 118.4 (2)   |
| N2—Fe1—N6  | 101.98 (7)  | H11—C11—C12 | 120.79 (13) |
| N2—Fe1—N1  | 92.62 (8)   | H11—C11—C10 | 120.79 (13) |
| C7—N3—Fe1  | 115.61 (14) | C7—C8—N4    | 113.70 (17) |
| C3—N3—Fe1  | 125.72 (15) | H8—C8—N4    | 108.82 (11) |
| C3—N3—C7   | 118.58 (18) | H8—C8—C7    | 108.82 (12) |
| C13—N5—Fe1 | 113.87 (13) | H7—C8—N4    | 108.82 (11) |
| C9—N5—Fe1  | 127.63 (14) | H7—C8—C7    | 108.82 (12) |
| C9—N5—C13  | 118.50 (18) | H7—C8—H8    | 107.7       |
| C14—N4—Fe1 | 105.97 (12) | N1—C1—S1    | 178.6 (2)   |
| C20—N4—Fe1 | 105.94 (11) | H6—C6—C7    | 120.46 (13) |
| C20—N4—C14 | 111.45 (15) | C5—C6—C7    | 119.1 (2)   |
| C8—N4—Fe1  | 110.94 (12) | C5—C6—H6    | 120.46 (13) |
| C8—N4—C14  | 110.85 (16) | H17—C17—C18 | 120.55 (14) |
| C8—N4—C20  | 111.45 (16) | C16—C17—C18 | 118.9 (2)   |
| C19—N6—Fe1 | 114.41 (13) | C16—C17—H17 | 120.55 (13) |
| C15—N6—Fe1 | 126.76 (15) | H3—C3—N3    | 118.72 (12) |
| C15—N6—C19 | 118.83 (19) | C4—C3—N3    | 122.6 (2)   |
| C1—N1—Fe1  | 172.97 (18) | C4—C3—H3    | 118.72 (15) |
| C2—N2—Fe1  | 170.80 (19) | C17—C16—C15 | 118.8 (2)   |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C18—C19—N6     | 122.24 (18)  | H16—C16—C15     | 120.61 (13)  |
| C20—C19—N6     | 115.18 (17)  | H16—C16—C17     | 120.61 (13)  |
| C20—C19—C18    | 122.55 (18)  | N2—C2—S2        | 178.1 (2)    |
| C13—C12—H12    | 120.40 (12)  | H5—C5—C6        | 120.56 (13)  |
| C11—C12—H12    | 120.40 (13)  | C4—C5—C6        | 118.9 (2)    |
| C11—C12—C13    | 119.2 (2)    | C4—C5—H5        | 120.56 (13)  |
| C12—C13—N5     | 122.31 (18)  | C5—C4—C3        | 118.7 (2)    |
| C14—C13—N5     | 115.66 (17)  | H4—C4—C3        | 120.64 (15)  |
| C14—C13—C12    | 121.98 (18)  | H4—C4—C5        | 120.64 (13)  |
| C13—C14—N4     | 110.10 (16)  | C23—C24—H24     | 120.81 (16)  |
| H14—C14—N4     | 109.64 (10)  | C25—C24—H24     | 120.81 (18)  |
| H14—C14—C13    | 109.64 (11)  | C25—C24—C23     | 118.4 (3)    |
| H13—C14—N4     | 109.64 (10)  | H23—C23—C24     | 120.72 (16)  |
| H13—C14—C13    | 109.64 (11)  | C22—C23—C24     | 118.6 (2)    |
| H13—C14—H14    | 108.2        | C22—C23—H23     | 120.72 (16)  |
| H18—C18—C19    | 120.61 (12)  | C24—C25—N7      | 123.5 (3)    |
| C17—C18—C19    | 118.8 (2)    | H25—C25—N7      | 118.24 (16)  |
| C17—C18—H18    | 120.61 (14)  | H25—C25—C24     | 118.24 (18)  |
| C8—C7—N3       | 117.42 (17)  | H22—C22—C23     | 120.71 (16)  |
| C6—C7—N3       | 122.2 (2)    | C21—C22—C23     | 118.6 (3)    |
| C6—C7—C8       | 120.37 (19)  | C21—C22—H22     | 120.71 (17)  |
| H9—C9—N5       | 118.86 (12)  | C22—C21—N7      | 123.6 (3)    |
| C10—C9—N5      | 122.3 (2)    | H21—C21—N7      | 118.21 (16)  |
| C10—C9—H9      | 118.86 (13)  | H21—C21—C22     | 118.21 (17)  |
| C21—N7—C25     | 117.4 (2)    |                 |              |
| Fe1—N3—C7—C8   | −5.18 (16)   | C19—C20—N4—C8   | −80.87 (18)  |
| Fe1—N3—C7—C6   | 176.61 (14)  | C12—C13—N5—C9   | 0.5 (2)      |
| Fe1—N3—C3—C4   | −175.97 (19) | C12—C11—C10—C9  | 0.2 (2)      |
| Fe1—N5—C13—C12 | 179.99 (13)  | C13—N5—C9—C10   | −1.4 (2)     |
| Fe1—N5—C13—C14 | −2.33 (14)   | C13—C12—C11—C10 | −1.0 (2)     |
| Fe1—N5—C9—C10  | 179.23 (17)  | C13—C14—N4—C20  | −154.48 (16) |
| Fe1—N4—C14—C13 | −39.69 (12)  | C13—C14—N4—C8   | 80.77 (18)   |
| Fe1—N4—C20—C19 | 39.90 (12)   | C14—N4—C8—C7    | −122.74 (16) |
| Fe1—N4—C8—C7   | −5.28 (13)   | C14—C13—N5—C9   | 178.22 (17)  |
| Fe1—N6—C19—C18 | 179.94 (13)  | C14—C13—C12—C11 | −176.86 (19) |
| Fe1—N6—C19—C20 | 1.95 (14)    | C18—C19—N6—C15  | −0.5 (2)     |
| Fe1—N6—C15—C16 | −178.68 (17) | C18—C17—C16—C15 | −0.6 (2)     |
| N3—C7—C8—N4    | 7.0 (2)      | C7—N3—C3—C4     | 0.4 (2)      |
| N3—C7—C6—C5    | −0.5 (2)     | C7—C8—N4—C20    | 112.51 (19)  |
| N3—C3—C4—C5    | −0.1 (3)     | C7—C6—C5—C4     | 0.8 (3)      |
| N5—C13—C12—C11 | 0.7 (2)      | N7—C25—C24—C23  | −0.8 (3)     |
| N5—C13—C14—N4  | 29.15 (18)   | N7—C21—C22—C23  | −0.2 (3)     |
| N5—C9—C10—C11  | 1.0 (2)      | C20—C19—N6—C15  | −178.47 (17) |
| N4—C14—C13—C12 | −153.16 (16) | C20—C19—C18—C17 | 176.5 (2)    |
| N4—C20—C19—N6  | −28.99 (19)  | C8—C7—N3—C3     | 178.06 (19)  |
| N4—C20—C19—C18 | 153.03 (16)  | C8—C7—C6—C5     | −178.6 (2)   |
| N4—C8—C7—C6    | −174.73 (18) | C6—C7—N3—C3     | −0.1 (3)     |

|                 |             |                 |          |
|-----------------|-------------|-----------------|----------|
| N6—C19—C18—C17  | −1.3 (2)    | C6—C5—C4—C3     | −0.6 (3) |
| N6—C15—C16—C17  | −1.2 (2)    | C24—C23—C22—C21 | −0.0 (3) |
| C19—N6—C15—C16  | 1.8 (2)     | C24—C25—N7—C21  | 0.6 (3)  |
| C19—C18—C17—C16 | 1.9 (2)     | C25—N7—C21—C22  | −0.2 (3) |
| C19—C20—N4—C14  | 154.71 (16) | C25—C24—C23—C22 | 0.5 (3)  |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_175K)**

*Crystal data*

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 14.9829$  (6) Å

$b = 11.9287$  (4) Å

$c = 15.1810$  (6) Å

$\beta = 110.220$  (4)°

$V = 2546.04$  (18) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.413$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 18200 reflections

$\theta = 2.1$ – $28.8^\circ$

$\mu = 0.78$  mm<sup>−1</sup>

$T = 175$  K

Block, yellow

$0.21 \times 0.08 \times 0.05$  mm

*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.838$ ,  $T_{\max} = 1.000$

53595 measured reflections

6311 independent reflections

4823 reflections with  $I \geq 2\sigma(I)$

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

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -19 \rightarrow 19$

$k = -15 \rightarrow 15$

$l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.080$

$S = 1.07$

6311 reflections

349 parameters

0 restraints

20 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 1.8717P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.19$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.95$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | $x$          | $y$          | $z$           | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| Fe1 | 0.30660 (2)  | 0.65514 (2)  | 0.662480 (19) | 0.02655 (8)                      |
| S2  | 0.19439 (5)  | 0.98667 (5)  | 0.47638 (4)   | 0.04517 (15)                     |
| S1  | 0.04710 (10) | 0.62913 (12) | 0.78525 (11)  | 0.05415 (19)                     |
| N5  | 0.39435 (10) | 0.73571 (13) | 0.78941 (11)  | 0.0242 (3)                       |
| N4  | 0.39043 (11) | 0.51471 (12) | 0.74582 (10)  | 0.0229 (3)                       |
| N3  | 0.42655 (11) | 0.65186 (14) | 0.61423 (11)  | 0.0276 (3)                       |
| N6  | 0.25619 (11) | 0.51338 (13) | 0.57179 (11)  | 0.0259 (3)                       |
| N1  | 0.19727 (13) | 0.62985 (14) | 0.71711 (12)  | 0.0362 (4)                       |
| C19 | 0.27604 (13) | 0.41290 (15) | 0.61346 (13)  | 0.0242 (4)                       |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| N2  | 0.24701 (13) | 0.79278 (17) | 0.58466 (13) | 0.0423 (5)  |
| C13 | 0.43355 (13) | 0.66644 (15) | 0.86215 (12) | 0.0238 (4)  |
| C18 | 0.24622 (14) | 0.31331 (17) | 0.56413 (14) | 0.0305 (4)  |
| H18 | 0.26466 (14) | 0.23167 (17) | 0.59993 (14) | 0.053 (7)*  |
| C12 | 0.49132 (14) | 0.70528 (17) | 0.94937 (13) | 0.0296 (4)  |
| H12 | 0.52314 (14) | 0.64601 (17) | 1.00756 (13) | 0.052 (7)*  |
| C7  | 0.49117 (13) | 0.57176 (17) | 0.65067 (13) | 0.0279 (4)  |
| C14 | 0.40789 (15) | 0.54421 (16) | 0.84471 (12) | 0.0278 (4)  |
| H14 | 0.34307 (15) | 0.52643 (16) | 0.86206 (12) | 0.050 (7)*  |
| H13 | 0.46677 (15) | 0.49187 (16) | 0.89109 (12) | 0.051 (7)*  |
| C15 | 0.20653 (14) | 0.51792 (19) | 0.47975 (14) | 0.0340 (5)  |
| H15 | 0.19177 (14) | 0.60058 (19) | 0.44516 (14) | 0.056 (7)*  |
| C9  | 0.41258 (13) | 0.84587 (16) | 0.80185 (14) | 0.0299 (4)  |
| H9  | 0.38182 (13) | 0.90284 (16) | 0.74172 (14) | 0.052 (7)*  |
| C10 | 0.46845 (14) | 0.89084 (17) | 0.88716 (15) | 0.0336 (5)  |
| H10 | 0.48080 (14) | 0.98204 (17) | 0.89498 (15) | 0.056 (7)*  |
| C20 | 0.32907 (14) | 0.41446 (15) | 0.71754 (13) | 0.0284 (4)  |
| H19 | 0.37360 (14) | 0.33782 (15) | 0.73796 (13) | 0.051 (7)*  |
| H20 | 0.27732 (14) | 0.41374 (15) | 0.75519 (13) | 0.050 (7)*  |
| C11 | 0.50869 (14) | 0.81945 (17) | 0.96271 (15) | 0.0329 (5)  |
| H11 | 0.55340 (14) | 0.85262 (17) | 1.03167 (15) | 0.058 (7)*  |
| C2  | 0.22495 (15) | 0.87443 (19) | 0.53963 (14) | 0.0342 (5)  |
| C8  | 0.48111 (15) | 0.50041 (18) | 0.72824 (15) | 0.0350 (5)  |
| H8  | 0.54083 (15) | 0.51996 (18) | 0.79401 (15) | 0.071 (8)*  |
| H7  | 0.48798 (15) | 0.41100 (18) | 0.71109 (15) | 0.068 (8)*  |
| C1  | 0.13419 (15) | 0.62847 (16) | 0.74517 (14) | 0.0316 (4)  |
| N7  | 0.78396 (17) | 0.8452 (2)   | 0.71122 (17) | 0.0627 (6)  |
| C17 | 0.19288 (15) | 0.31866 (19) | 0.46911 (14) | 0.0355 (5)  |
| H17 | 0.16684 (15) | 0.24134 (19) | 0.42849 (14) | 0.062 (8)*  |
| C3  | 0.43686 (16) | 0.7183 (2)   | 0.54709 (15) | 0.0381 (5)  |
| H3  | 0.38375 (16) | 0.7848 (2)   | 0.51746 (15) | 0.058 (7)*  |
| C16 | 0.17289 (15) | 0.4226 (2)   | 0.42612 (15) | 0.0390 (5)  |
| H16 | 0.13100 (15) | 0.4295 (2)   | 0.35064 (15) | 0.056 (7)*  |
| C6  | 0.56776 (15) | 0.5552 (2)   | 0.62042 (15) | 0.0389 (5)  |
| H6  | 0.61999 (15) | 0.4883 (2)   | 0.65158 (15) | 0.068 (8)*  |
| C23 | 0.65138 (19) | 0.7733 (2)   | 0.78877 (17) | 0.0493 (6)  |
| H23 | 0.59879 (19) | 0.7442 (2)   | 0.81966 (17) | 0.086 (10)* |
| C4  | 0.51115 (17) | 0.7072 (2)   | 0.51344 (17) | 0.0481 (6)  |
| H4  | 0.51736 (17) | 0.7634 (2)   | 0.45819 (17) | 0.077 (9)*  |
| C5  | 0.57772 (17) | 0.6233 (2)   | 0.55092 (17) | 0.0494 (6)  |
| H5  | 0.63772 (17) | 0.6114 (2)   | 0.52554 (17) | 0.067 (8)*  |
| C22 | 0.7381 (2)   | 0.7216 (2)   | 0.81046 (17) | 0.0534 (7)  |
| H22 | 0.7565 (2)   | 0.6497 (2)   | 0.85922 (17) | 0.099 (11)* |
| C24 | 0.63167 (19) | 0.8624 (2)   | 0.72750 (19) | 0.0538 (7)  |
| H24 | 0.56308 (19) | 0.9070 (2)   | 0.70840 (19) | 0.085 (10)* |
| C25 | 0.7001 (2)   | 0.8948 (2)   | 0.6904 (2)   | 0.0598 (7)  |
| H25 | 0.6839 (2)   | 0.9657 (2)   | 0.6408 (2)   | 0.111 (12)* |
| C21 | 0.80183 (19) | 0.7605 (3)   | 0.77055 (18) | 0.0565 (7)  |



|     |              |            |              |            |
|-----|--------------|------------|--------------|------------|
| H21 | 0.87153 (19) | 0.7185 (3) | 0.78917 (18) | 0.075 (9)* |
|-----|--------------|------------|--------------|------------|

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.03052 (15) | 0.02510 (14) | 0.02656 (14) | −0.00672 (12) | 0.01309 (11) | −0.00107 (12) |
| S2  | 0.0550 (4)   | 0.0427 (3)   | 0.0415 (3)   | 0.0040 (3)    | 0.0214 (3)   | 0.0038 (3)    |
| S1  | 0.0396 (3)   | 0.0548 (4)   | 0.0737 (5)   | −0.0138 (3)   | 0.0269 (3)   | −0.0285 (4)   |
| N5  | 0.0222 (8)   | 0.0206 (8)   | 0.0282 (8)   | −0.0004 (6)   | 0.0066 (6)   | 0.0050 (6)    |
| N4  | 0.0261 (8)   | 0.0189 (8)   | 0.0239 (8)   | −0.0001 (6)   | 0.0088 (6)   | 0.0034 (6)    |
| N3  | 0.0230 (8)   | 0.0321 (9)   | 0.0298 (8)   | −0.0002 (7)   | 0.0116 (6)   | 0.0021 (7)    |
| N6  | 0.0254 (8)   | 0.0252 (8)   | 0.0271 (8)   | 0.0044 (6)    | 0.0089 (7)   | 0.0032 (7)    |
| N1  | 0.0464 (11)  | 0.0257 (9)   | 0.0396 (10)  | −0.0046 (8)   | 0.0188 (9)   | −0.0045 (7)   |
| C19 | 0.0268 (9)   | 0.0203 (9)   | 0.0257 (9)   | −0.0002 (7)   | 0.0092 (8)   | 0.0017 (7)    |
| N2  | 0.0401 (11)  | 0.0468 (12)  | 0.0400 (11)  | −0.0031 (9)   | 0.0141 (9)   | 0.0014 (9)    |
| C13 | 0.0265 (9)   | 0.0198 (9)   | 0.0237 (9)   | −0.0008 (7)   | 0.0069 (7)   | 0.0012 (7)    |
| C18 | 0.0337 (11)  | 0.0257 (10)  | 0.0325 (11)  | −0.0036 (8)   | 0.0118 (9)   | −0.0039 (8)   |
| C12 | 0.0321 (10)  | 0.0278 (10)  | 0.0258 (10)  | −0.0010 (8)   | 0.0059 (8)   | 0.0000 (8)    |
| C7  | 0.0217 (9)   | 0.0337 (11)  | 0.0290 (10)  | −0.0011 (8)   | 0.0096 (8)   | −0.0036 (8)   |
| C14 | 0.0382 (11)  | 0.0207 (9)   | 0.0224 (9)   | −0.0022 (8)   | 0.0079 (8)   | 0.0049 (7)    |
| C15 | 0.0329 (11)  | 0.0384 (12)  | 0.0275 (10)  | 0.0085 (9)    | 0.0065 (8)   | 0.0063 (9)    |
| C9  | 0.0270 (9)   | 0.0197 (9)   | 0.0418 (11)  | −0.0003 (8)   | 0.0105 (8)   | 0.0046 (9)    |
| C10 | 0.0286 (10)  | 0.0221 (10)  | 0.0477 (13)  | −0.0024 (8)   | 0.0101 (9)   | −0.0041 (9)   |
| C20 | 0.0355 (11)  | 0.0202 (10)  | 0.0269 (10)  | −0.0039 (8)   | 0.0077 (8)   | 0.0027 (8)    |
| C11 | 0.0306 (10)  | 0.0298 (11)  | 0.0350 (11)  | −0.0026 (8)   | 0.0069 (9)   | −0.0075 (9)   |
| C2  | 0.0348 (11)  | 0.0381 (13)  | 0.0314 (11)  | 0.0008 (9)    | 0.0134 (9)   | −0.0007 (9)   |
| C8  | 0.0317 (11)  | 0.0345 (12)  | 0.0406 (12)  | 0.0080 (9)    | 0.0149 (9)   | 0.0085 (9)    |
| C1  | 0.0317 (11)  | 0.0245 (10)  | 0.0381 (11)  | −0.0024 (8)   | 0.0115 (9)   | −0.0057 (8)   |
| N7  | 0.0597 (15)  | 0.0721 (16)  | 0.0687 (15)  | −0.0117 (13)  | 0.0381 (12)  | −0.0025 (13)  |
| C17 | 0.0321 (11)  | 0.0409 (13)  | 0.0318 (11)  | −0.0053 (9)   | 0.0089 (9)   | −0.0081 (9)   |
| C3  | 0.0352 (11)  | 0.0461 (13)  | 0.0365 (11)  | −0.0005 (10)  | 0.0168 (9)   | 0.0116 (10)   |
| C16 | 0.0323 (11)  | 0.0527 (15)  | 0.0264 (11)  | 0.0047 (10)   | 0.0031 (9)   | −0.0025 (10)  |
| C6  | 0.0264 (11)  | 0.0529 (14)  | 0.0414 (12)  | 0.0040 (10)   | 0.0167 (9)   | −0.0040 (11)  |
| C23 | 0.0564 (16)  | 0.0519 (15)  | 0.0504 (14)  | −0.0152 (13)  | 0.0323 (12)  | −0.0132 (12)  |
| C4  | 0.0450 (14)  | 0.0651 (17)  | 0.0423 (13)  | −0.0072 (12)  | 0.0252 (11)  | 0.0083 (12)   |
| C5  | 0.0348 (12)  | 0.0744 (18)  | 0.0489 (14)  | −0.0023 (12)  | 0.0274 (11)  | 0.0009 (13)   |
| C22 | 0.0708 (18)  | 0.0532 (16)  | 0.0418 (13)  | −0.0008 (14)  | 0.0266 (13)  | 0.0019 (12)   |
| C24 | 0.0493 (15)  | 0.0513 (16)  | 0.0622 (16)  | 0.0003 (13)   | 0.0211 (13)  | −0.0122 (13)  |
| C25 | 0.076 (2)    | 0.0462 (16)  | 0.0649 (18)  | −0.0077 (14)  | 0.0337 (15)  | 0.0044 (14)   |
| C21 | 0.0493 (16)  | 0.0688 (19)  | 0.0518 (15)  | 0.0071 (14)   | 0.0183 (12)  | −0.0028 (14)  |

*Geometric parameters (Å, °)*

|        |             |         |           |
|--------|-------------|---------|-----------|
| Fe1—N5 | 2.1480 (15) | C15—C16 | 1.388 (3) |
| Fe1—N4 | 2.2103 (15) | C9—H9   | 1.1030    |
| Fe1—N3 | 2.1628 (15) | C9—C10  | 1.384 (3) |
| Fe1—N6 | 2.1464 (16) | C10—H10 | 1.1030    |
| Fe1—N1 | 2.0980 (19) | C10—C11 | 1.388 (3) |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| Fe1—N2     | 2.039 (2)   | C20—H19     | 1.1110      |
| S2—C2      | 1.619 (2)   | C20—H20     | 1.1110      |
| S1—C1      | 1.619 (2)   | C11—H11     | 1.1030      |
| N5—C13     | 1.341 (2)   | C8—H8       | 1.1110      |
| N5—C9      | 1.342 (2)   | C8—H7       | 1.1110      |
| N4—C14     | 1.475 (2)   | N7—C25      | 1.324 (4)   |
| N4—C20     | 1.479 (2)   | N7—C21      | 1.319 (4)   |
| N4—C8      | 1.482 (2)   | C17—H17     | 1.1030      |
| N3—C7      | 1.336 (2)   | C17—C16     | 1.385 (3)   |
| N3—C3      | 1.342 (2)   | C3—H3       | 1.1030      |
| N6—C19     | 1.340 (2)   | C3—C4       | 1.382 (3)   |
| N6—C15     | 1.338 (2)   | C16—H16     | 1.1030      |
| N1—C1      | 1.163 (3)   | C6—H6       | 1.1030      |
| C19—C18    | 1.393 (3)   | C6—C5       | 1.380 (3)   |
| C19—C20    | 1.503 (2)   | C23—H23     | 1.1030      |
| N2—C2      | 1.170 (3)   | C23—C22     | 1.372 (4)   |
| C13—C12    | 1.387 (3)   | C23—C24     | 1.376 (4)   |
| C13—C14    | 1.508 (2)   | C4—H4       | 1.1030      |
| C18—H18    | 1.1030      | C4—C5       | 1.388 (4)   |
| C18—C17    | 1.388 (3)   | C5—H5       | 1.1030      |
| C12—H12    | 1.1030      | C22—H22     | 1.1030      |
| C12—C11    | 1.388 (3)   | C22—C21     | 1.377 (4)   |
| C7—C8      | 1.502 (3)   | C24—H24     | 1.1030      |
| C7—C6      | 1.390 (3)   | C24—C25     | 1.384 (4)   |
| C14—H14    | 1.1110      | C25—H25     | 1.1030      |
| C14—H13    | 1.1110      | C21—H21     | 1.1030      |
| C15—H15    | 1.1030      |             |             |
|            |             |             |             |
| N4—Fe1—N5  | 77.00 (6)   | C10—C9—N5   | 122.61 (19) |
| N3—Fe1—N5  | 89.39 (6)   | C10—C9—H9   | 118.70 (12) |
| N3—Fe1—N4  | 78.80 (6)   | H10—C10—C9  | 120.50 (12) |
| N6—Fe1—N5  | 153.96 (6)  | C11—C10—C9  | 119.00 (19) |
| N6—Fe1—N4  | 76.96 (6)   | C11—C10—H10 | 120.50 (12) |
| N6—Fe1—N3  | 85.29 (6)   | C19—C20—N4  | 111.39 (15) |
| N1—Fe1—N5  | 91.67 (6)   | H19—C20—N4  | 109.35 (10) |
| N1—Fe1—N4  | 91.81 (6)   | H19—C20—C19 | 109.35 (10) |
| N1—Fe1—N3  | 170.07 (7)  | H20—C20—N4  | 109.35 (10) |
| N1—Fe1—N6  | 89.46 (6)   | H20—C20—C19 | 109.35 (10) |
| N2—Fe1—N5  | 99.74 (7)   | H20—C20—H19 | 108.0       |
| N2—Fe1—N4  | 171.91 (7)  | C10—C11—C12 | 118.39 (19) |
| N2—Fe1—N3  | 93.85 (7)   | H11—C11—C12 | 120.81 (12) |
| N2—Fe1—N6  | 106.03 (7)  | H11—C11—C10 | 120.81 (12) |
| N2—Fe1—N1  | 95.71 (7)   | N2—C2—S2    | 179.4 (2)   |
| C13—N5—Fe1 | 114.90 (12) | C7—C8—N4    | 114.99 (16) |
| C9—N5—Fe1  | 126.58 (13) | H8—C8—N4    | 108.52 (10) |
| C9—N5—C13  | 118.52 (16) | H8—C8—C7    | 108.52 (11) |
| C14—N4—Fe1 | 105.53 (11) | H7—C8—N4    | 108.52 (10) |
| C20—N4—Fe1 | 105.87 (11) | H7—C8—C7    | 108.52 (11) |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C20—N4—C14     | 111.24 (14)  | H7—C8—H8        | 107.5        |
| C8—N4—Fe1      | 111.04 (11)  | N1—C1—S1        | 178.78 (19)  |
| C8—N4—C14      | 111.07 (15)  | C21—N7—C25      | 117.2 (2)    |
| C8—N4—C20      | 111.79 (15)  | H17—C17—C18     | 120.53 (13)  |
| C7—N3—Fe1      | 116.15 (13)  | C16—C17—C18     | 118.9 (2)    |
| C3—N3—Fe1      | 125.02 (14)  | C16—C17—H17     | 120.53 (12)  |
| C3—N3—C7       | 118.67 (17)  | H3—C3—N3        | 118.57 (12)  |
| C19—N6—Fe1     | 115.50 (12)  | C4—C3—N3        | 122.9 (2)    |
| C15—N6—Fe1     | 125.69 (14)  | C4—C3—H3        | 118.57 (14)  |
| C15—N6—C19     | 118.81 (17)  | C17—C16—C15     | 118.79 (19)  |
| C1—N1—Fe1      | 172.30 (16)  | H16—C16—C15     | 120.61 (12)  |
| C18—C19—N6     | 122.10 (17)  | H16—C16—C17     | 120.61 (12)  |
| C20—C19—N6     | 115.82 (16)  | H6—C6—C7        | 120.38 (13)  |
| C20—C19—C18    | 122.03 (17)  | C5—C6—C7        | 119.2 (2)    |
| C2—N2—Fe1      | 170.85 (18)  | C5—C6—H6        | 120.38 (14)  |
| C12—C13—N5     | 122.09 (17)  | C22—C23—H23     | 120.84 (16)  |
| C14—C13—N5     | 116.02 (16)  | C24—C23—H23     | 120.84 (16)  |
| C14—C13—C12    | 121.85 (17)  | C24—C23—C22     | 118.3 (2)    |
| H18—C18—C19    | 120.59 (11)  | H4—C4—C3        | 120.84 (14)  |
| C17—C18—C19    | 118.82 (19)  | C5—C4—C3        | 118.3 (2)    |
| C17—C18—H18    | 120.59 (13)  | C5—C4—H4        | 120.84 (13)  |
| H12—C12—C13    | 120.31 (11)  | C4—C5—C6        | 119.1 (2)    |
| C11—C12—C13    | 119.38 (19)  | H5—C5—C6        | 120.47 (14)  |
| C11—C12—H12    | 120.31 (12)  | H5—C5—C4        | 120.47 (13)  |
| C8—C7—N3       | 118.36 (17)  | H22—C22—C23     | 120.46 (16)  |
| C6—C7—N3       | 121.87 (19)  | C21—C22—C23     | 119.1 (3)    |
| C6—C7—C8       | 119.76 (19)  | C21—C22—H22     | 120.46 (17)  |
| C13—C14—N4     | 110.75 (14)  | H24—C24—C23     | 120.85 (16)  |
| H14—C14—N4     | 109.49 (10)  | C25—C24—C23     | 118.3 (3)    |
| H14—C14—C13    | 109.49 (10)  | C25—C24—H24     | 120.85 (18)  |
| H13—C14—N4     | 109.49 (10)  | C24—C25—N7      | 123.7 (3)    |
| H13—C14—C13    | 109.49 (10)  | H25—C25—N7      | 118.17 (17)  |
| H13—C14—H14    | 108.1        | H25—C25—C24     | 118.17 (18)  |
| H15—C15—N6     | 118.75 (12)  | C22—C21—N7      | 123.4 (3)    |
| C16—C15—N6     | 122.5 (2)    | H21—C21—N7      | 118.28 (16)  |
| C16—C15—H15    | 118.75 (12)  | H21—C21—C22     | 118.28 (17)  |
| H9—C9—N5       | 118.70 (11)  |                 |              |
| Fe1—N5—C13—C12 | −179.34 (13) | C19—C20—N4—C8   | −80.57 (17)  |
| Fe1—N5—C13—C14 | −1.77 (14)   | C13—N5—C9—C10   | −1.4 (2)     |
| Fe1—N5—C9—C10  | 178.43 (15)  | C13—C12—C11—C10 | −1.0 (2)     |
| Fe1—N4—C14—C13 | −42.23 (11)  | C13—C14—N4—C20  | −156.59 (15) |
| Fe1—N4—C20—C19 | 40.48 (11)   | C13—C14—N4—C8   | 78.20 (17)   |
| Fe1—N4—C8—C7   | −7.51 (12)   | C18—C19—N6—C15  | −0.3 (2)     |
| Fe1—N3—C7—C8   | −6.58 (15)   | C18—C17—C16—C15 | −0.3 (2)     |
| Fe1—N3—C7—C6   | 175.07 (14)  | C12—C13—N5—C9   | 0.5 (2)      |
| Fe1—N3—C3—C4   | −174.74 (18) | C12—C11—C10—C9  | 0.2 (2)      |
| Fe1—N6—C19—C18 | 178.61 (13)  | C7—N3—C3—C4     | 0.4 (2)      |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| Fe1—N6—C19—C20  | 1.00 (14)    | C7—C8—N4—C14    | −124.61 (18) |
| Fe1—N6—C15—C16  | −177.33 (16) | C7—C8—N4—C20    | 110.49 (18)  |
| N5—C13—C12—C11  | 0.7 (2)      | C7—C6—C5—C4     | 0.4 (3)      |
| N5—C13—C14—N4   | 31.08 (17)   | C14—C13—N5—C9   | 178.10 (16)  |
| N5—C9—C10—C11   | 1.1 (2)      | C14—C13—C12—C11 | −176.75 (19) |
| N4—C14—C13—C12  | −151.34 (15) | C15—N6—C19—C20  | −177.95 (17) |
| N4—C20—C19—N6   | −29.29 (18)  | C20—C19—C18—C17 | 176.35 (19)  |
| N4—C20—C19—C18  | 153.10 (15)  | C8—C7—N3—C3     | 177.81 (19)  |
| N4—C8—C7—N3     | 9.6 (2)      | C8—C7—C6—C5     | −178.2 (2)   |
| N4—C8—C7—C6     | −172.01 (17) | N7—C25—C24—C23  | −0.6 (3)     |
| N3—C7—C6—C5     | 0.1 (2)      | N7—C21—C22—C23  | −0.6 (3)     |
| N3—C3—C4—C5     | 0.1 (3)      | C3—N3—C7—C6     | −0.5 (2)     |
| N6—C19—C18—C17  | −1.1 (2)     | C3—C4—C5—C6     | −0.4 (3)     |
| N6—C15—C16—C17  | −1.2 (2)     | C22—C23—C24—C25 | 0.4 (3)      |
| C19—N6—C15—C16  | 1.5 (2)      | C22—C21—N7—C25  | 0.5 (3)      |
| C19—C18—C17—C16 | 1.4 (2)      | C24—C23—C22—C21 | 0.1 (3)      |
| C19—C20—N4—C14  | 154.63 (16)  | C24—C25—N7—C21  | 0.1 (3)      |

### Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) pyridine monosolvate (py\_195K)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 15.0146$  (6) Å

$b = 11.9342$  (4) Å

$c = 15.2467$  (6) Å

$\beta = 110.162$  (4)°

$V = 2564.60$  (18) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.402$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 19099 reflections

$\theta = 2.1$ – $29.3^\circ$

$\mu = 0.78$  mm<sup>−1</sup>

$T = 195$  K

Block, yellow

$0.21 \times 0.08 \times 0.05$  mm

#### Data collection

Rigaku XtaLAB Synergy-R  
diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray  
tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian  
(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.826$ ,  $T_{\max} = 1.000$

54219 measured reflections

6356 independent reflections

4858 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -20 \rightarrow 20$

$k = -15 \rightarrow 15$

$l = -20 \rightarrow 20$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.072$

$S = 1.06$

6356 reflections

359 parameters

0 restraints

20 constraints

Primary atom site location: dual

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 1.0173P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.85$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.64$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.695102 (16) | 0.657500 (19) | 0.338293 (16) | 0.02627 (7)                      |
| S2  | 0.80620 (9)   | 0.98854 (10)  | 0.52349 (8)   | 0.04722 (13)                     |
| S1  | 0.95447 (8)   | 0.62955 (11)  | 0.21530 (9)   | 0.05746 (17)                     |
| N4  | 0.61009 (9)   | 0.51555 (11)  | 0.25491 (9)   | 0.0249 (3)                       |
| N5  | 0.60609 (9)   | 0.73659 (11)  | 0.20975 (9)   | 0.0263 (3)                       |
| N6  | 0.74425 (9)   | 0.51336 (11)  | 0.42883 (9)   | 0.0274 (3)                       |
| N3  | 0.57352 (9)   | 0.65374 (12)  | 0.38602 (9)   | 0.0304 (3)                       |
| N1  | 0.80508 (11)  | 0.63066 (12)  | 0.28376 (10)  | 0.0370 (3)                       |
| N2  | 0.75390 (11)  | 0.79603 (14)  | 0.41587 (11)  | 0.0423 (4)                       |
| C19 | 0.72358 (11)  | 0.41334 (13)  | 0.38700 (11)  | 0.0263 (3)                       |
| C18 | 0.75255 (12)  | 0.31332 (14)  | 0.43574 (12)  | 0.0326 (4)                       |
| H18 | 0.73345 (12)  | 0.23197 (14)  | 0.39981 (12)  | 0.054 (6)*                       |
| C13 | 0.56753 (11)  | 0.66641 (13)  | 0.13819 (11)  | 0.0263 (3)                       |
| C7  | 0.50969 (11)  | 0.57325 (15)  | 0.34989 (11)  | 0.0297 (4)                       |
| C12 | 0.50954 (12)  | 0.70394 (15)  | 0.05097 (11)  | 0.0319 (4)                       |
| H12 | 0.47791 (12)  | 0.64399 (15)  | −0.00643 (11) | 0.057 (6)*                       |
| C14 | 0.59327 (13)  | 0.54447 (13)  | 0.15659 (11)  | 0.0309 (4)                       |
| H14 | 0.65823 (13)  | 0.52649 (13)  | 0.13984 (11)  | 0.041 (5)*                       |
| H13 | 0.53483 (13)  | 0.49179 (13)  | 0.11046 (11)  | 0.056 (6)*                       |
| C15 | 0.79396 (12)  | 0.51725 (16)  | 0.52060 (12)  | 0.0355 (4)                       |
| H15 | 0.80951 (12)  | 0.59964 (16)  | 0.55535 (12)  | 0.057 (6)*                       |
| C9  | 0.58769 (11)  | 0.84663 (14)  | 0.19650 (12)  | 0.0311 (4)                       |
| H9  | 0.61837 (11)  | 0.90433 (14)  | 0.25580 (12)  | 0.045 (5)*                       |
| C20 | 0.67069 (12)  | 0.41517 (13)  | 0.28331 (11)  | 0.0313 (4)                       |
| H19 | 0.62586 (12)  | 0.33887 (13)  | 0.26302 (11)  | 0.060 (6)*                       |
| H20 | 0.72239 (12)  | 0.41379 (13)  | 0.24587 (11)  | 0.061 (6)*                       |
| C1  | 0.86760 (12)  | 0.62919 (14)  | 0.25547 (12)  | 0.0323 (4)                       |
| C11 | 0.49194 (12)  | 0.81794 (15)  | 0.03701 (12)  | 0.0358 (4)                       |
| H11 | 0.44718 (12)  | 0.85018 (15)  | −0.03189 (12) | 0.059 (6)*                       |
| C10 | 0.53160 (12)  | 0.89043 (15)  | 0.11114 (13)  | 0.0355 (4)                       |
| H10 | 0.51878 (12)  | 0.98144 (15)  | 0.10242 (13)  | 0.060 (6)*                       |
| C2  | 0.77603 (12)  | 0.87715 (16)  | 0.46087 (12)  | 0.0341 (4)                       |
| C17 | 0.80572 (12)  | 0.31796 (16)  | 0.53003 (12)  | 0.0371 (4)                       |
| H17 | 0.83117 (12)  | 0.24032 (16)  | 0.57008 (12)  | 0.064 (7)*                       |
| C8  | 0.51954 (12)  | 0.50245 (16)  | 0.27216 (13)  | 0.0372 (4)                       |
| H8  | 0.46036 (12)  | 0.52321 (16)  | 0.20676 (13)  | 0.079 (8)*                       |
| H7  | 0.51163 (12)  | 0.41305 (16)  | 0.28842 (13)  | 0.075 (7)*                       |
| C16 | 0.82651 (13)  | 0.42159 (17)  | 0.57338 (12)  | 0.0401 (4)                       |
| H16 | 0.86823 (13)  | 0.42776 (17)  | 0.64856 (12)  | 0.056 (6)*                       |
| C3  | 0.56315 (14)  | 0.71940 (17)  | 0.45320 (13)  | 0.0420 (4)                       |
| H3  | 0.61617 (14)  | 0.78562 (17)  | 0.48321 (13)  | 0.061 (6)*                       |
| C6  | 0.43335 (13)  | 0.55587 (18)  | 0.37998 (13)  | 0.0437 (5)                       |
| H6  | 0.38154 (13)  | 0.48881 (18)  | 0.34882 (13)  | 0.065 (7)*                       |
| N7  | 0.21659 (16)  | 0.8452 (2)    | 0.28725 (16)  | 0.0717 (6)                       |
| C23 | 0.34906 (17)  | 0.7733 (2)    | 0.21091 (15)  | 0.0551 (6)                       |

|     |              |            |              |             |
|-----|--------------|------------|--------------|-------------|
| H23 | 0.40186 (17) | 0.7443 (2) | 0.18053 (15) | 0.099 (9)*  |
| C4  | 0.48894 (15) | 0.7077 (2) | 0.48644 (15) | 0.0542 (6)  |
| H4  | 0.48225 (15) | 0.7640 (2) | 0.54120 (15) | 0.077 (8)*  |
| C5  | 0.42310 (15) | 0.6236 (2) | 0.44941 (15) | 0.0565 (6)  |
| H5  | 0.36345 (15) | 0.6109 (2) | 0.47492 (15) | 0.090 (8)*  |
| C22 | 0.26291 (18) | 0.7213 (2) | 0.18916 (15) | 0.0592 (6)  |
| H22 | 0.24507 (18) | 0.6492 (2) | 0.14095 (15) | 0.102 (10)* |
| C24 | 0.36819 (17) | 0.8624 (2) | 0.27134 (17) | 0.0605 (6)  |
| H24 | 0.43647 (17) | 0.9073 (2) | 0.29021 (17) | 0.087 (8)*  |
| C25 | 0.3001 (2)   | 0.8946 (2) | 0.30792 (18) | 0.0668 (7)  |
| H25 | 0.3161 (2)   | 0.9656 (2) | 0.35724 (18) | 0.119 (11)* |
| C21 | 0.19884 (17) | 0.7603 (2) | 0.22839 (16) | 0.0625 (6)  |
| H21 | 0.12927 (17) | 0.7184 (2) | 0.20966 (16) | 0.108 (10)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|-------------|---------------|
| Fe1 | 0.02813 (12) | 0.02423 (12) | 0.02773 (12) | 0.00307 (10) | 0.01128 (9) | −0.00122 (10) |
| S2  | 0.0630 (3)   | 0.0385 (3)   | 0.0440 (3)   | −0.0070 (2)  | 0.0234 (2)  | −0.0090 (2)   |
| S1  | 0.0416 (3)   | 0.0627 (4)   | 0.0777 (4)   | 0.0173 (3)   | 0.0328 (3)  | 0.0323 (3)    |
| N4  | 0.0277 (7)   | 0.0205 (7)   | 0.0257 (6)   | 0.0003 (5)   | 0.0084 (5)  | −0.0034 (5)   |
| N5  | 0.0257 (7)   | 0.0222 (7)   | 0.0295 (7)   | 0.0005 (5)   | 0.0077 (5)  | −0.0041 (6)   |
| N6  | 0.0279 (7)   | 0.0255 (7)   | 0.0286 (7)   | −0.0030 (6)  | 0.0096 (6)  | −0.0028 (6)   |
| N3  | 0.0271 (7)   | 0.0355 (8)   | 0.0316 (7)   | −0.0002 (6)  | 0.0137 (6)  | −0.0050 (6)   |
| N1  | 0.0421 (9)   | 0.0298 (8)   | 0.0441 (9)   | 0.0041 (6)   | 0.0211 (7)  | 0.0052 (6)    |
| N2  | 0.0425 (9)   | 0.0394 (9)   | 0.0435 (9)   | −0.0032 (7)  | 0.0128 (7)  | −0.0061 (8)   |
| C19 | 0.0281 (8)   | 0.0229 (8)   | 0.0282 (8)   | 0.0009 (6)   | 0.0101 (7)  | −0.0012 (6)   |
| C18 | 0.0352 (9)   | 0.0267 (9)   | 0.0354 (9)   | 0.0036 (7)   | 0.0116 (7)  | 0.0036 (7)    |
| C13 | 0.0300 (8)   | 0.0210 (8)   | 0.0265 (8)   | 0.0004 (6)   | 0.0079 (6)  | −0.0018 (6)   |
| C7  | 0.0244 (8)   | 0.0353 (9)   | 0.0309 (8)   | 0.0004 (7)   | 0.0112 (7)  | 0.0023 (7)    |
| C12 | 0.0357 (9)   | 0.0287 (9)   | 0.0273 (8)   | 0.0008 (7)   | 0.0060 (7)  | 0.0013 (7)    |
| C14 | 0.0436 (10)  | 0.0219 (8)   | 0.0260 (8)   | 0.0015 (7)   | 0.0103 (7)  | −0.0046 (6)   |
| C15 | 0.0342 (9)   | 0.0382 (10)  | 0.0301 (9)   | −0.0077 (8)  | 0.0061 (7)  | −0.0049 (8)   |
| C9  | 0.0284 (8)   | 0.0211 (8)   | 0.0413 (9)   | 0.0001 (7)   | 0.0085 (7)  | −0.0041 (7)   |
| C20 | 0.0417 (10)  | 0.0204 (8)   | 0.0282 (8)   | 0.0035 (7)   | 0.0076 (7)  | −0.0035 (7)   |
| C1  | 0.0303 (9)   | 0.0273 (9)   | 0.0394 (9)   | 0.0037 (7)   | 0.0121 (7)  | 0.0077 (7)    |
| C11 | 0.0359 (9)   | 0.0323 (10)  | 0.0351 (9)   | 0.0030 (7)   | 0.0068 (8)  | 0.0072 (7)    |
| C10 | 0.0330 (9)   | 0.0232 (9)   | 0.0470 (10)  | 0.0026 (7)   | 0.0094 (8)  | 0.0040 (8)    |
| C2  | 0.0366 (9)   | 0.0346 (10)  | 0.0326 (9)   | −0.0024 (8)  | 0.0137 (7)  | −0.0017 (8)   |
| C17 | 0.0354 (9)   | 0.0393 (11)  | 0.0340 (9)   | 0.0033 (8)   | 0.0087 (8)  | 0.0072 (8)    |
| C8  | 0.0322 (9)   | 0.0388 (10)  | 0.0417 (10)  | −0.0082 (8)  | 0.0139 (8)  | −0.0097 (8)   |
| C16 | 0.0361 (10)  | 0.0488 (12)  | 0.0290 (9)   | −0.0038 (8)  | 0.0029 (7)  | 0.0047 (8)    |
| C3  | 0.0398 (10)  | 0.0493 (12)  | 0.0411 (10)  | 0.0008 (9)   | 0.0192 (8)  | −0.0133 (9)   |
| C6  | 0.0310 (10)  | 0.0582 (13)  | 0.0468 (11)  | −0.0044 (9)  | 0.0198 (8)  | 0.0049 (10)   |
| N7  | 0.0714 (14)  | 0.0835 (16)  | 0.0733 (14)  | 0.0149 (13)  | 0.0418 (12) | 0.0005 (13)   |
| C23 | 0.0626 (15)  | 0.0571 (14)  | 0.0573 (13)  | 0.0172 (12)  | 0.0355 (12) | 0.0152 (11)   |
| C4  | 0.0498 (12)  | 0.0748 (16)  | 0.0474 (12)  | 0.0088 (11)  | 0.0287 (10) | −0.0115 (11)  |
| C5  | 0.0407 (11)  | 0.0867 (18)  | 0.0537 (13)  | 0.0028 (11)  | 0.0312 (10) | −0.0006 (12)  |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C22 | 0.0794 (17) | 0.0550 (14) | 0.0462 (12) | 0.0020 (12)  | 0.0255 (12) | −0.0007 (11) |
| C24 | 0.0562 (14) | 0.0576 (15) | 0.0680 (15) | −0.0040 (12) | 0.0221 (12) | 0.0136 (12)  |
| C25 | 0.087 (2)   | 0.0490 (14) | 0.0706 (16) | 0.0042 (13)  | 0.0353 (15) | −0.0098 (12) |
| C21 | 0.0522 (14) | 0.0796 (18) | 0.0582 (14) | −0.0074 (13) | 0.0222 (11) | 0.0056 (13)  |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N4    | 2.2353 (13) | C15—C16     | 1.385 (3)   |
| Fe1—N5    | 2.1708 (13) | C9—H9       | 1.1030      |
| Fe1—N6    | 2.1696 (14) | C9—C10      | 1.386 (2)   |
| Fe1—N3    | 2.1862 (13) | C20—H19     | 1.1110      |
| Fe1—N1    | 2.1135 (15) | C20—H20     | 1.1110      |
| Fe1—N2    | 2.0474 (17) | C11—H11     | 1.1030      |
| S2—C2     | 1.608 (2)   | C11—C10     | 1.384 (2)   |
| S1—C1     | 1.621 (2)   | C10—H10     | 1.1030      |
| N4—C14    | 1.473 (2)   | C17—H17     | 1.1030      |
| N4—C20    | 1.476 (2)   | C17—C16     | 1.386 (3)   |
| N4—C8     | 1.479 (2)   | C8—H8       | 1.1110      |
| N5—C13    | 1.3385 (19) | C8—H7       | 1.1110      |
| N5—C9     | 1.343 (2)   | C16—H16     | 1.1030      |
| N6—C19    | 1.338 (2)   | C3—H3       | 1.1030      |
| N6—C15    | 1.341 (2)   | C3—C4       | 1.381 (3)   |
| N3—C7     | 1.335 (2)   | C6—H6       | 1.1030      |
| N3—C3     | 1.341 (2)   | C6—C5       | 1.382 (3)   |
| N1—C1     | 1.160 (2)   | N7—C25      | 1.321 (3)   |
| N2—C2     | 1.167 (2)   | N7—C21      | 1.319 (3)   |
| C19—C18   | 1.394 (2)   | C23—H23     | 1.1030      |
| C19—C20   | 1.505 (2)   | C23—C22     | 1.369 (3)   |
| C18—H18   | 1.1030      | C23—C24     | 1.371 (3)   |
| C18—C17   | 1.384 (2)   | C4—H4       | 1.1030      |
| C13—C12   | 1.389 (2)   | C4—C5       | 1.385 (3)   |
| C13—C14   | 1.507 (2)   | C5—H5       | 1.1030      |
| C7—C8     | 1.504 (2)   | C22—H22     | 1.1030      |
| C7—C6     | 1.389 (2)   | C22—C21     | 1.377 (3)   |
| C12—H12   | 1.1030      | C24—H24     | 1.1030      |
| C12—C11   | 1.388 (2)   | C24—C25     | 1.378 (3)   |
| C14—H14   | 1.1110      | C25—H25     | 1.1030      |
| C14—H13   | 1.1110      | C21—H21     | 1.1030      |
| C15—H15   | 1.1030      |             |             |
| N5—Fe1—N4 | 76.28 (5)   | C10—C9—N5   | 122.48 (16) |
| N6—Fe1—N4 | 76.31 (5)   | C10—C9—H9   | 118.76 (10) |
| N6—Fe1—N5 | 152.59 (5)  | C19—C20—N4  | 111.63 (13) |
| N3—Fe1—N4 | 78.18 (5)   | H19—C20—N4  | 109.29 (8)  |
| N3—Fe1—N5 | 89.12 (5)   | H19—C20—C19 | 109.29 (9)  |
| N3—Fe1—N6 | 84.94 (5)   | H20—C20—N4  | 109.29 (9)  |
| N1—Fe1—N4 | 91.82 (5)   | H20—C20—C19 | 109.29 (9)  |
| N1—Fe1—N5 | 91.84 (5)   | H20—C20—H19 | 108.0       |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N1—Fe1—N6   | 89.40 (5)   | N1—C1—S1    | 178.93 (16) |
| N1—Fe1—N3   | 169.43 (6)  | H11—C11—C12 | 120.60 (10) |
| N2—Fe1—N4   | 171.31 (6)  | C10—C11—C12 | 118.80 (16) |
| N2—Fe1—N5   | 100.32 (6)  | C10—C11—H11 | 120.60 (10) |
| N2—Fe1—N6   | 106.76 (6)  | C11—C10—C9  | 118.82 (16) |
| N2—Fe1—N3   | 93.87 (6)   | H10—C10—C9  | 120.59 (10) |
| N2—Fe1—N1   | 96.31 (6)   | H10—C10—C11 | 120.59 (10) |
| C14—N4—Fe1  | 105.38 (9)  | N2—C2—S2    | 179.55 (17) |
| C20—N4—Fe1  | 105.95 (9)  | H17—C17—C18 | 120.48 (11) |
| C20—N4—C14  | 111.15 (12) | C16—C17—C18 | 119.04 (17) |
| C8—N4—Fe1   | 111.01 (9)  | C16—C17—H17 | 120.48 (10) |
| C8—N4—C14   | 111.10 (13) | C7—C8—N4    | 115.27 (14) |
| C8—N4—C20   | 111.93 (13) | H8—C8—N4    | 108.46 (9)  |
| C13—N5—Fe1  | 115.00 (10) | H8—C8—C7    | 108.46 (9)  |
| C9—N5—Fe1   | 126.28 (11) | H7—C8—N4    | 108.46 (9)  |
| C9—N5—C13   | 118.72 (14) | H7—C8—C7    | 108.46 (10) |
| C19—N6—Fe1  | 115.58 (10) | H7—C8—H8    | 107.5       |
| C15—N6—Fe1  | 125.56 (12) | C17—C16—C15 | 118.81 (16) |
| C15—N6—C19  | 118.85 (15) | H16—C16—C15 | 120.59 (11) |
| C7—N3—Fe1   | 116.15 (11) | H16—C16—C17 | 120.59 (10) |
| C3—N3—Fe1   | 124.93 (12) | H3—C3—N3    | 118.58 (11) |
| C3—N3—C7    | 118.69 (15) | C4—C3—N3    | 122.84 (19) |
| C1—N1—Fe1   | 172.00 (14) | C4—C3—H3    | 118.58 (13) |
| C2—N2—Fe1   | 171.29 (15) | H6—C6—C7    | 120.39 (11) |
| C18—C19—N6  | 122.08 (14) | C5—C6—C7    | 119.22 (19) |
| C20—C19—N6  | 116.06 (14) | C5—C6—H6    | 120.39 (13) |
| C20—C19—C18 | 121.81 (14) | C21—N7—C25  | 117.3 (2)   |
| H18—C18—C19 | 120.60 (10) | C22—C23—H23 | 120.77 (14) |
| C17—C18—C19 | 118.79 (16) | C24—C23—H23 | 120.77 (14) |
| C17—C18—H18 | 120.60 (11) | C24—C23—C22 | 118.5 (2)   |
| C12—C13—N5  | 122.07 (15) | H4—C4—C3    | 120.78 (13) |
| C14—C13—N5  | 116.19 (14) | C5—C4—C3    | 118.44 (19) |
| C14—C13—C12 | 121.71 (14) | C5—C4—H4    | 120.78 (12) |
| C8—C7—N3    | 118.56 (14) | C4—C5—C6    | 118.93 (18) |
| C6—C7—N3    | 121.88 (16) | H5—C5—C6    | 120.54 (13) |
| C6—C7—C8    | 119.53 (16) | H5—C5—C4    | 120.54 (12) |
| H12—C12—C13 | 120.46 (10) | H22—C22—C23 | 120.50 (14) |
| C11—C12—C13 | 119.09 (16) | C21—C22—C23 | 119.0 (2)   |
| C11—C12—H12 | 120.46 (10) | C21—C22—H22 | 120.50 (16) |
| C13—C14—N4  | 111.03 (13) | H24—C24—C23 | 120.81 (14) |
| H14—C14—N4  | 109.43 (9)  | C25—C24—C23 | 118.4 (2)   |
| H14—C14—C13 | 109.43 (9)  | C25—C24—H24 | 120.81 (16) |
| H13—C14—N4  | 109.43 (8)  | C24—C25—N7  | 123.7 (2)   |
| H13—C14—C13 | 109.43 (9)  | H25—C25—N7  | 118.16 (15) |
| H13—C14—H14 | 108.0       | H25—C25—C24 | 118.16 (16) |
| H15—C15—N6  | 118.80 (10) | C22—C21—N7  | 123.2 (2)   |
| C16—C15—N6  | 122.40 (17) | H21—C21—N7  | 118.40 (14) |
| C16—C15—H15 | 118.80 (11) | H21—C21—C22 | 118.40 (16) |



|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| H9—C9—N5        | 118.76 (9)   |                 |              |
| Fe1—N4—C14—C13  | −42.68 (10)  | C19—C20—N4—C8   | −80.56 (15)  |
| Fe1—N4—C20—C19  | 40.57 (10)   | C18—C19—N6—C15  | −0.36 (19)   |
| Fe1—N4—C8—C7    | −8.41 (10)   | C18—C17—C16—C15 | −0.5 (2)     |
| Fe1—N5—C13—C12  | −179.41 (11) | C13—N5—C9—C10   | −1.11 (17)   |
| Fe1—N5—C13—C14  | −1.42 (12)   | C13—C12—C11—C10 | −1.08 (19)   |
| Fe1—N5—C9—C10   | 178.44 (13)  | C13—C14—N4—C20  | −157.02 (14) |
| Fe1—N6—C19—C18  | 178.39 (11)  | C13—C14—N4—C8   | 77.64 (15)   |
| Fe1—N6—C19—C20  | 0.87 (12)    | C7—N3—C3—C4     | −0.3 (2)     |
| Fe1—N6—C15—C16  | −177.19 (14) | C7—C8—N4—C14    | −125.34 (15) |
| Fe1—N3—C7—C8    | −7.51 (13)   | C7—C8—N4—C20    | 109.75 (16)  |
| Fe1—N3—C7—C6    | 174.62 (12)  | C7—C6—C5—C4     | 0.6 (2)      |
| Fe1—N3—C3—C4    | −174.45 (16) | C12—C13—N5—C9   | 0.19 (19)    |
| N4—C14—C13—N5   | 31.30 (16)   | C12—C11—C10—C9  | 0.2 (2)      |
| N4—C14—C13—C12  | −150.70 (13) | C14—C13—N5—C9   | 178.18 (14)  |
| N4—C20—C19—N6   | −29.37 (16)  | C14—C13—C12—C11 | −176.98 (17) |
| N4—C20—C19—C18  | 153.10 (13)  | C15—N6—C19—C20  | −177.88 (15) |
| N4—C8—C7—N3     | 10.89 (18)   | C20—C19—C18—C17 | 176.27 (16)  |
| N4—C8—C7—C6     | −171.19 (15) | C8—C7—N3—C3     | 177.82 (16)  |
| N5—C13—C12—C11  | 0.90 (19)    | C8—C7—C6—C5     | −177.97 (18) |
| N5—C9—C10—C11   | 0.91 (19)    | C3—N3—C7—C6     | −0.04 (19)   |
| N6—C19—C18—C17  | −1.11 (18)   | C3—C4—C5—C6     | −0.9 (2)     |
| N6—C15—C16—C17  | −1.0 (2)     | N7—C25—C24—C23  | −0.8 (3)     |
| N3—C7—C6—C5     | −0.1 (2)     | N7—C21—C22—C23  | −0.8 (3)     |
| N3—C3—C4—C5     | 0.8 (2)      | C22—C23—C24—C25 | 0.6 (3)      |
| C19—N6—C15—C16  | 1.42 (17)    | C22—C21—N7—C25  | 0.6 (3)      |
| C19—C18—C17—C16 | 1.51 (19)    | C24—C23—C22—C21 | 0.2 (2)      |
| C19—C20—N4—C14  | 154.55 (14)  | C24—C25—N7—C21  | 0.2 (3)      |

### Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_215K)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 15.0532$  (5) Å

$b = 11.9435$  (4) Å

$c = 15.2993$  (6) Å

$\beta = 110.128$  (4)°

$V = 2582.64$  (17) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.393$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 18486 reflections

$\theta = 2.1$ – $29.1$ °

$\mu = 0.77$  mm<sup>−1</sup>

$T = 215$  K

, yellow

$0.21 \times 0.08 \times 0.06$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.801$ ,  $T_{\max} = 1.000$

54272 measured reflections

6407 independent reflections

4807 reflections with  $I \geq 2\sigma(I)$  $R_{\text{int}} = 0.030$  $\theta_{\text{max}} = 28.3^\circ$ ,  $\theta_{\text{min}} = 2.2^\circ$  $h = -20 \rightarrow 20$  $k = -15 \rightarrow 15$  $l = -20 \rightarrow 20$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.036$  $wR(F^2) = 0.073$  $S = 1.07$ 

6407 reflections

354 parameters

0 restraints

25 constraints

Primary atom site location: dual

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.027P)^2 + 0.809P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = -0.001$  $\Delta\rho_{\text{max}} = 0.70 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.40 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>     | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|---------------|----------------------------------|
| Fe1 | 0.695881 (16) | 0.65914 (2)  | 0.338512 (16) | 0.02765 (7)                      |
| S2  | 0.80691 (9)   | 0.98968 (10) | 0.52351 (8)   | 0.05126 (14)                     |
| S1  | 0.95535 (8)   | 0.63053 (11) | 0.21668 (9)   | 0.06301 (18)                     |
| N5  | 0.60640 (9)   | 0.73738 (11) | 0.20929 (9)   | 0.0288 (3)                       |
| N6  | 0.74436 (9)   | 0.51398 (12) | 0.42895 (9)   | 0.0298 (3)                       |
| N4  | 0.61062 (9)   | 0.51648 (11) | 0.25551 (9)   | 0.0281 (3)                       |
| N3  | 0.57390 (9)   | 0.65514 (13) | 0.38615 (9)   | 0.0340 (3)                       |
| N1  | 0.80632 (11)  | 0.63171 (13) | 0.28442 (10)  | 0.0396 (4)                       |
| N2  | 0.75414 (11)  | 0.79770 (14) | 0.41582 (11)  | 0.0453 (4)                       |
| C19 | 0.72332 (11)  | 0.41403 (14) | 0.38728 (11)  | 0.0287 (3)                       |
| C18 | 0.75163 (12)  | 0.31421 (15) | 0.43571 (12)  | 0.0359 (4)                       |
| H18 | 0.73233 (12)  | 0.23373 (15) | 0.40020 (12)  | 0.055 (6)*                       |
| C7  | 0.51045 (11)  | 0.57458 (15) | 0.35019 (11)  | 0.0329 (4)                       |
| C13 | 0.56815 (11)  | 0.66679 (14) | 0.13851 (11)  | 0.0288 (3)                       |
| C9  | 0.58784 (11)  | 0.84713 (14) | 0.19534 (12)  | 0.0332 (4)                       |
| H9  | 0.61817 (11)  | 0.90479 (14) | 0.25349 (12)  | 0.044 (5)*                       |
| C1  | 0.86875 (12)  | 0.63031 (14) | 0.25642 (12)  | 0.0343 (4)                       |
| C15 | 0.79387 (12)  | 0.51762 (16) | 0.52017 (12)  | 0.0380 (4)                       |
| H15 | 0.80962 (12)  | 0.59920 (16) | 0.55443 (12)  | 0.058 (6)*                       |
| C14 | 0.59412 (13)  | 0.54485 (14) | 0.15739 (11)  | 0.0347 (4)                       |
| H14 | 0.65850 (13)  | 0.52707 (14) | 0.14112 (11)  | 0.055 (6)*                       |
| H13 | 0.53659 (13)  | 0.49236 (14) | 0.11192 (11)  | 0.060 (6)*                       |
| C12 | 0.51027 (12)  | 0.70334 (15) | 0.05122 (12)  | 0.0356 (4)                       |
| H12 | 0.47936 (12)  | 0.64345 (15) | -0.00510 (12) | 0.059 (6)*                       |
| C2  | 0.77647 (12)  | 0.87841 (16) | 0.46094 (12)  | 0.0361 (4)                       |
| C11 | 0.49234 (12)  | 0.81721 (16) | 0.03670 (13)  | 0.0393 (4)                       |
| H11 | 0.44792 (12)  | 0.84854 (16) | -0.03146 (13) | 0.069 (7)*                       |
| C17 | 0.80474 (12)  | 0.31857 (17) | 0.52976 (12)  | 0.0400 (4)                       |
| H17 | 0.82950 (12)  | 0.24157 (17) | 0.56926 (12)  | 0.068 (7)*                       |
| C10 | 0.53175 (12)  | 0.89026 (16) | 0.11012 (13)  | 0.0390 (4)                       |
| H10 | 0.51885 (12)  | 0.98029 (16) | 0.10105 (13)  | 0.058 (6)*                       |
| C20 | 0.67085 (13)  | 0.41599 (14) | 0.28388 (11)  | 0.0350 (4)                       |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| H19 | 0.62642 (13) | 0.34055 (14) | 0.26368 (11) | 0.069 (7)*  |
| H20 | 0.72212 (13) | 0.41449 (14) | 0.24707 (11) | 0.062 (6)*  |
| C8  | 0.51999 (12) | 0.50427 (17) | 0.27232 (13) | 0.0406 (4)  |
| H8  | 0.46188 (12) | 0.52589 (17) | 0.20778 (13) | 0.079 (7)*  |
| H7  | 0.51127 (12) | 0.41571 (17) | 0.28767 (13) | 0.074 (7)*  |
| C16 | 0.82597 (13) | 0.42199 (18) | 0.57297 (13) | 0.0435 (5)  |
| H16 | 0.86721 (13) | 0.42805 (18) | 0.64718 (13) | 0.065 (6)*  |
| C3  | 0.56315 (14) | 0.72037 (19) | 0.45298 (13) | 0.0467 (5)  |
| H3  | 0.61523 (14) | 0.78625 (19) | 0.48260 (13) | 0.068 (7)*  |
| C6  | 0.43421 (13) | 0.5566 (2)   | 0.38029 (14) | 0.0494 (5)  |
| H6  | 0.38322 (13) | 0.4899 (2)   | 0.34968 (14) | 0.079 (7)*  |
| C23 | 0.34908 (18) | 0.7737 (2)   | 0.21060 (17) | 0.0634 (6)  |
| H23 | 0.40134 (18) | 0.7452 (2)   | 0.18056 (17) | 0.0761 (8)* |
| N7  | 0.21657 (18) | 0.8451 (2)   | 0.28583 (17) | 0.0826 (7)  |
| C4  | 0.48923 (16) | 0.7082 (2)   | 0.48614 (15) | 0.0607 (6)  |
| H4  | 0.48253 (16) | 0.7638 (2)   | 0.54028 (15) | 0.089 (8)*  |
| C22 | 0.2637 (2)   | 0.7216 (2)   | 0.18908 (15) | 0.0665 (7)  |
| H22 | 0.2464 (2)   | 0.6502 (2)   | 0.14153 (15) | 0.0798 (8)* |
| C5  | 0.42403 (15) | 0.6244 (2)   | 0.44954 (16) | 0.0635 (7)  |
| H5  | 0.36516 (15) | 0.6117 (2)   | 0.47486 (16) | 0.101 (9)*  |
| C24 | 0.36781 (18) | 0.8623 (2)   | 0.27050 (18) | 0.0701 (7)  |
| H24 | 0.43530 (18) | 0.9066 (2)   | 0.28929 (18) | 0.0841 (8)* |
| C21 | 0.19979 (18) | 0.7597 (3)   | 0.22770 (17) | 0.0707 (7)  |
| H21 | 0.13146 (18) | 0.7173 (3)   | 0.20948 (17) | 0.0848 (8)* |
| C25 | 0.3001 (2)   | 0.8944 (2)   | 0.30663 (19) | 0.0774 (8)  |
| H25 | 0.3159 (2)   | 0.9647 (2)   | 0.35534 (19) | 0.0928 (9)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$      |
|-----|--------------|--------------|--------------|--------------|-------------|---------------|
| Fe1 | 0.02910 (11) | 0.02450 (12) | 0.03023 (12) | 0.00096 (10) | 0.01132 (9) | −0.00271 (10) |
| S2  | 0.0720 (4)   | 0.0383 (3)   | 0.0474 (3)   | −0.0098 (3)  | 0.0256 (3)  | −0.0128 (2)   |
| S1  | 0.0454 (3)   | 0.0708 (4)   | 0.0851 (4)   | 0.0209 (3)   | 0.0382 (3)  | 0.0369 (3)    |
| N5  | 0.0294 (7)   | 0.0232 (7)   | 0.0318 (7)   | 0.0006 (5)   | 0.0080 (6)  | −0.0032 (6)   |
| N6  | 0.0315 (7)   | 0.0268 (8)   | 0.0301 (7)   | −0.0036 (6)  | 0.0093 (6)  | −0.0026 (6)   |
| N4  | 0.0326 (7)   | 0.0229 (7)   | 0.0276 (7)   | −0.0001 (6)  | 0.0091 (5)  | −0.0050 (6)   |
| N3  | 0.0313 (7)   | 0.0388 (8)   | 0.0352 (7)   | 0.0009 (6)   | 0.0157 (6)  | −0.0061 (7)   |
| N1  | 0.0421 (8)   | 0.0334 (9)   | 0.0500 (9)   | 0.0049 (7)   | 0.0243 (7)  | 0.0062 (7)    |
| N2  | 0.0463 (9)   | 0.0393 (10)  | 0.0481 (9)   | −0.0061 (7)  | 0.0136 (7)  | −0.0117 (8)   |
| C19 | 0.0319 (8)   | 0.0231 (9)   | 0.0313 (8)   | −0.0004 (6)  | 0.0110 (7)  | 0.0007 (7)    |
| C18 | 0.0400 (9)   | 0.0277 (10)  | 0.0388 (9)   | 0.0032 (7)   | 0.0122 (8)  | 0.0036 (7)    |
| C7  | 0.0268 (8)   | 0.0387 (10)  | 0.0343 (8)   | −0.0001 (7)  | 0.0119 (7)  | 0.0024 (7)    |
| C13 | 0.0322 (8)   | 0.0246 (9)   | 0.0283 (8)   | −0.0001 (7)  | 0.0087 (6)  | −0.0028 (7)   |
| C9  | 0.0331 (8)   | 0.0227 (9)   | 0.0405 (9)   | −0.0009 (7)  | 0.0084 (7)  | −0.0042 (8)   |
| C1  | 0.0316 (8)   | 0.0296 (9)   | 0.0429 (9)   | 0.0043 (7)   | 0.0140 (7)  | 0.0082 (7)    |
| C15 | 0.0386 (9)   | 0.0381 (11)  | 0.0324 (9)   | −0.0081 (8)  | 0.0060 (7)  | −0.0052 (8)   |
| C14 | 0.0483 (10)  | 0.0241 (9)   | 0.0294 (8)   | 0.0024 (8)   | 0.0106 (8)  | −0.0047 (7)   |
| C12 | 0.0406 (9)   | 0.0316 (10)  | 0.0303 (9)   | 0.0005 (8)   | 0.0067 (7)  | 0.0002 (7)    |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C2  | 0.0408 (9)  | 0.0341 (10) | 0.0343 (9)  | −0.0061 (8)  | 0.0142 (7)  | −0.0039 (8)  |
| C11 | 0.0387 (9)  | 0.0359 (11) | 0.0389 (10) | 0.0030 (8)   | 0.0078 (8)  | 0.0078 (8)   |
| C17 | 0.0406 (9)  | 0.0398 (12) | 0.0371 (10) | 0.0041 (8)   | 0.0101 (8)  | 0.0081 (8)   |
| C10 | 0.0382 (9)  | 0.0258 (9)  | 0.0496 (11) | 0.0028 (7)   | 0.0107 (8)  | 0.0056 (8)   |
| C20 | 0.0468 (10) | 0.0232 (9)  | 0.0319 (9)  | 0.0032 (8)   | 0.0094 (8)  | −0.0031 (7)  |
| C8  | 0.0363 (9)  | 0.0411 (11) | 0.0452 (10) | −0.0092 (8)  | 0.0152 (8)  | −0.0088 (9)  |
| C16 | 0.0413 (10) | 0.0502 (13) | 0.0325 (9)  | −0.0030 (9)  | 0.0045 (8)  | 0.0032 (9)   |
| C3  | 0.0440 (10) | 0.0578 (13) | 0.0433 (10) | 0.0035 (10)  | 0.0212 (9)  | −0.0149 (10) |
| C6  | 0.0345 (10) | 0.0682 (15) | 0.0504 (11) | −0.0052 (10) | 0.0209 (9)  | 0.0054 (10)  |
| C23 | 0.0737 (16) | 0.0656 (17) | 0.0662 (15) | 0.0210 (13)  | 0.0439 (13) | 0.0185 (13)  |
| N7  | 0.0821 (16) | 0.0956 (19) | 0.0846 (16) | 0.0211 (15)  | 0.0472 (13) | 0.0034 (14)  |
| C4  | 0.0561 (13) | 0.0852 (18) | 0.0519 (12) | 0.0110 (12)  | 0.0327 (11) | −0.0110 (12) |
| C22 | 0.0932 (19) | 0.0609 (16) | 0.0495 (13) | 0.0026 (14)  | 0.0300 (13) | −0.0020 (11) |
| C5  | 0.0445 (12) | 0.099 (2)   | 0.0590 (13) | 0.0022 (12)  | 0.0337 (11) | −0.0012 (13) |
| C24 | 0.0646 (15) | 0.0677 (18) | 0.0779 (17) | −0.0050 (13) | 0.0245 (13) | 0.0160 (14)  |
| C21 | 0.0597 (15) | 0.089 (2)   | 0.0647 (15) | −0.0096 (14) | 0.0236 (12) | 0.0054 (15)  |
| C25 | 0.104 (2)   | 0.0582 (17) | 0.0765 (17) | 0.0070 (16)  | 0.0393 (17) | −0.0109 (14) |

*Geometric parameters (Å, °)*

|         |             |         |           |
|---------|-------------|---------|-----------|
| Fe1—N5  | 2.1825 (13) | C14—H13 | 1.1010    |
| Fe1—N6  | 2.1812 (14) | C12—H12 | 1.0930    |
| Fe1—N4  | 2.2453 (13) | C12—C11 | 1.389 (2) |
| Fe1—N3  | 2.1969 (13) | C11—H11 | 1.0930    |
| Fe1—N1  | 2.1220 (15) | C11—C10 | 1.384 (3) |
| Fe1—N2  | 2.0476 (16) | C17—H17 | 1.0930    |
| S2—C2   | 1.609 (2)   | C17—C16 | 1.385 (3) |
| S1—C1   | 1.617 (2)   | C10—H10 | 1.0930    |
| N5—C13  | 1.336 (2)   | C20—H19 | 1.1010    |
| N5—C9   | 1.342 (2)   | C20—H20 | 1.1010    |
| N6—C19  | 1.339 (2)   | C8—H8   | 1.1010    |
| N6—C15  | 1.338 (2)   | C8—H7   | 1.1010    |
| N4—C14  | 1.474 (2)   | C16—H16 | 1.0930    |
| N4—C20  | 1.477 (2)   | C3—H3   | 1.0930    |
| N4—C8   | 1.480 (2)   | C3—C4   | 1.380 (3) |
| N3—C7   | 1.334 (2)   | C6—H6   | 1.0930    |
| N3—C3   | 1.339 (2)   | C6—C5   | 1.383 (3) |
| N1—C1   | 1.159 (2)   | C23—H23 | 1.0930    |
| N2—C2   | 1.166 (2)   | C23—C22 | 1.363 (3) |
| C19—C18 | 1.391 (2)   | C23—C24 | 1.364 (4) |
| C19—C20 | 1.506 (2)   | N7—C21  | 1.319 (3) |
| C18—H18 | 1.0930      | N7—C25  | 1.324 (4) |
| C18—C17 | 1.386 (2)   | C4—H4   | 1.0930    |
| C7—C8   | 1.504 (2)   | C4—C5   | 1.378 (3) |
| C7—C6   | 1.393 (2)   | C22—H22 | 1.0930    |
| C13—C14 | 1.510 (2)   | C22—C21 | 1.369 (3) |
| C13—C12 | 1.390 (2)   | C5—H5   | 1.0930    |
| C9—H9   | 1.0930      | C24—H24 | 1.0930    |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| C9—C10      | 1.386 (2)   | C24—C25     | 1.371 (4)   |
| C15—H15     | 1.0930      | C21—H21     | 1.0930      |
| C15—C16     | 1.386 (3)   | C25—H25     | 1.0930      |
| C14—H14     | 1.1010      |             |             |
| N6—Fe1—N5   | 151.93 (5)  | H12—C12—C13 | 120.52 (10) |
| N4—Fe1—N5   | 76.00 (5)   | C11—C12—C13 | 118.95 (17) |
| N4—Fe1—N6   | 75.93 (5)   | C11—C12—H12 | 120.52 (11) |
| N3—Fe1—N5   | 89.09 (5)   | N2—C2—S2    | 179.70 (17) |
| N3—Fe1—N6   | 84.70 (5)   | H11—C11—C12 | 120.60 (11) |
| N3—Fe1—N4   | 77.93 (5)   | C10—C11—C12 | 118.79 (17) |
| N1—Fe1—N5   | 92.00 (5)   | C10—C11—H11 | 120.60 (11) |
| N1—Fe1—N6   | 89.35 (5)   | H17—C17—C18 | 120.50 (11) |
| N1—Fe1—N4   | 91.96 (5)   | C16—C17—C18 | 119.00 (17) |
| N1—Fe1—N3   | 169.26 (6)  | C16—C17—H17 | 120.50 (11) |
| N2—Fe1—N5   | 100.68 (6)  | C11—C10—C9  | 118.78 (17) |
| N2—Fe1—N6   | 107.02 (6)  | H10—C10—C9  | 120.61 (11) |
| N2—Fe1—N4   | 171.06 (6)  | H10—C10—C11 | 120.61 (11) |
| N2—Fe1—N3   | 93.82 (6)   | C19—C20—N4  | 111.62 (13) |
| N2—Fe1—N1   | 96.47 (6)   | H19—C20—N4  | 109.30 (9)  |
| C13—N5—Fe1  | 115.05 (11) | H19—C20—C19 | 109.30 (9)  |
| C9—N5—Fe1   | 126.30 (11) | H20—C20—N4  | 109.30 (9)  |
| C9—N5—C13   | 118.64 (14) | H20—C20—C19 | 109.30 (9)  |
| C19—N6—Fe1  | 115.72 (10) | H20—C20—H19 | 108.0       |
| C15—N6—Fe1  | 125.49 (12) | C7—C8—N4    | 115.31 (14) |
| C15—N6—C19  | 118.77 (15) | H8—C8—N4    | 108.45 (9)  |
| C14—N4—Fe1  | 105.35 (10) | H8—C8—C7    | 108.45 (10) |
| C20—N4—Fe1  | 106.08 (9)  | H7—C8—N4    | 108.45 (9)  |
| C20—N4—C14  | 110.94 (12) | H7—C8—C7    | 108.45 (10) |
| C8—N4—Fe1   | 110.99 (10) | H7—C8—H8    | 107.5       |
| C8—N4—C14   | 110.93 (13) | C17—C16—C15 | 118.66 (16) |
| C8—N4—C20   | 112.23 (14) | H16—C16—C15 | 120.67 (11) |
| C7—N3—Fe1   | 116.13 (11) | H16—C16—C17 | 120.67 (11) |
| C3—N3—Fe1   | 125.11 (13) | H3—C3—N3    | 118.53 (11) |
| C3—N3—C7    | 118.51 (15) | C4—C3—N3    | 122.9 (2)   |
| C1—N1—Fe1   | 171.82 (14) | C4—C3—H3    | 118.53 (14) |
| C2—N2—Fe1   | 171.58 (15) | H6—C6—C7    | 120.53 (12) |
| C18—C19—N6  | 122.12 (15) | C5—C6—C7    | 118.9 (2)   |
| C20—C19—N6  | 116.04 (14) | C5—C6—H6    | 120.53 (13) |
| C20—C19—C18 | 121.78 (15) | C22—C23—H23 | 120.78 (15) |
| H18—C18—C19 | 120.58 (10) | C24—C23—H23 | 120.78 (15) |
| C17—C18—C19 | 118.84 (17) | C24—C23—C22 | 118.4 (2)   |
| C17—C18—H18 | 120.58 (11) | C25—N7—C21  | 116.8 (2)   |
| C8—C7—N3    | 118.65 (14) | H4—C4—C3    | 120.70 (14) |
| C6—C7—N3    | 121.99 (17) | C5—C4—C3    | 118.6 (2)   |
| C6—C7—C8    | 119.32 (17) | C5—C4—H4    | 120.70 (12) |
| C14—C13—N5  | 116.27 (14) | H22—C22—C23 | 120.38 (15) |
| C12—C13—N5  | 122.22 (16) | C21—C22—C23 | 119.2 (2)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C12—C13—C14     | 121.47 (15)  | C21—C22—H22     | 120.38 (17)  |
| H9—C9—N5        | 118.70 (9)   | C4—C5—C6        | 119.03 (19)  |
| C10—C9—N5       | 122.60 (16)  | H5—C5—C6        | 120.49 (13)  |
| C10—C9—H9       | 118.70 (11)  | H5—C5—C4        | 120.49 (12)  |
| N1—C1—S1        | 179.01 (16)  | H24—C24—C23     | 120.74 (15)  |
| H15—C15—N6      | 118.71 (10)  | C25—C24—C23     | 118.5 (3)    |
| C16—C15—N6      | 122.58 (17)  | C25—C24—H24     | 120.74 (18)  |
| C16—C15—H15     | 118.71 (11)  | C22—C21—N7      | 123.3 (2)    |
| C13—C14—N4      | 110.99 (13)  | H21—C21—N7      | 118.37 (16)  |
| H14—C14—N4      | 109.44 (9)   | H21—C21—C22     | 118.37 (17)  |
| H14—C14—C13     | 109.44 (9)   | C24—C25—N7      | 123.7 (3)    |
| H13—C14—N4      | 109.44 (9)   | H25—C25—N7      | 118.15 (16)  |
| H13—C14—C13     | 109.44 (9)   | H25—C25—C24     | 118.15 (18)  |
| H13—C14—H14     | 108.0        |                 |              |
| Fe1—N5—C13—C14  | −1.46 (12)   | C19—C20—N4—C8   | −80.51 (15)  |
| Fe1—N5—C13—C12  | −179.29 (11) | C18—C19—N6—C15  | −0.29 (19)   |
| Fe1—N5—C9—C10   | 178.48 (13)  | C18—C17—C16—C15 | −0.5 (2)     |
| Fe1—N6—C19—C18  | 178.43 (11)  | C7—N3—C3—C4     | −0.2 (2)     |
| Fe1—N6—C19—C20  | 1.06 (12)    | C7—C8—N4—C14    | −126.05 (16) |
| Fe1—N6—C15—C16  | −177.21 (14) | C7—C8—N4—C20    | 109.22 (16)  |
| Fe1—N4—C14—C13  | −42.97 (10)  | C7—C6—C5—C4     | 0.5 (2)      |
| Fe1—N4—C20—C19  | 40.86 (10)   | C13—N5—C9—C10   | −0.96 (17)   |
| Fe1—N4—C8—C7    | −9.28 (11)   | C13—C14—N4—C20  | −157.34 (14) |
| Fe1—N3—C7—C8    | −7.98 (14)   | C13—C14—N4—C8   | 77.21 (16)   |
| Fe1—N3—C7—C6    | 174.37 (13)  | C13—C12—C11—C10 | −0.87 (19)   |
| Fe1—N3—C3—C4    | −174.20 (17) | C9—N5—C13—C14   | 178.04 (15)  |
| N5—C13—C14—N4   | 31.56 (15)   | C9—N5—C13—C12   | 0.21 (17)    |
| N5—C13—C12—C11  | 0.70 (18)    | C9—C10—C11—C12  | 0.2 (2)      |
| N5—C9—C10—C11   | 0.77 (19)    | C15—N6—C19—C20  | −177.66 (15) |
| N6—C19—C18—C17  | −1.15 (18)   | C14—C13—C12—C11 | −177.02 (17) |
| N6—C19—C20—N4   | −29.66 (15)  | C17—C18—C19—C20 | 176.06 (15)  |
| N6—C15—C16—C17  | −1.0 (2)     | C8—C7—N3—C3     | 177.46 (17)  |
| N4—C14—C13—C12  | −150.59 (13) | C8—C7—C6—C5     | −177.60 (19) |
| N4—C20—C19—C18  | 152.96 (13)  | C3—N3—C7—C6     | −0.2 (2)     |
| N4—C8—C7—N3     | 11.83 (19)   | C3—C4—C5—C6     | −0.8 (3)     |
| N4—C8—C7—C6     | −170.45 (16) | C23—C22—C21—N7  | −0.3 (3)     |
| N3—C7—C6—C5     | 0.0 (2)      | C23—C24—C25—N7  | −0.8 (3)     |
| N3—C3—C4—C5     | 0.7 (2)      | C22—C23—C24—C25 | 0.3 (3)      |
| C19—N6—C15—C16  | 1.37 (17)    | C22—C21—N7—C25  | −0.2 (3)     |
| C19—C18—C17—C16 | 1.53 (19)    | C24—C23—C22—C21 | 0.2 (3)      |
| C19—C20—N4—C14  | 154.77 (14)  | C24—C25—N7—C21  | 0.8 (3)      |

**Bis(thiocyanato- $\kappa$ N)[tris(pyridin-2-ylmethyl)amine- $\kappa^4$ N,N',N'',N''']iron(II) pyridine monosolvate (py\_235K)***Crystal data*[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N $M_r = 541.49$ Monoclinic,  $P2_1/n$  $a = 15.0842$  (7) Å $b = 11.9641$  (4) Å $c = 15.3418$  (7) Å $\beta = 110.131$  (5)° $V = 2599.6$  (2) Å<sup>3</sup> $Z = 4$  $F(000) = 1123.013$  $D_x = 1.384$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 16386 reflections

 $\theta = 2.1$ – $26.9^\circ$  $\mu = 0.77$  mm<sup>-1</sup> $T = 235$  K

Block, yellow

 $0.21 \times 0.08 \times 0.06$  mm*Data collection*

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup> $\omega$  scans

Absorption correction: gaussian

(CrysAlis PRO; Rigaku OD, 2025)

 $T_{\min} = 0.809$ ,  $T_{\max} = 1.000$ 

56527 measured reflections

5300 independent reflections

4157 reflections with  $I \geq 2\sigma(I)$  $R_{\text{int}} = 0.044$  $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$  $h = -18 \rightarrow 18$  $k = -14 \rightarrow 14$  $l = -19 \rightarrow 19$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.027$  $wR(F^2) = 0.063$  $S = 1.05$ 

5300 reflections

354 parameters

0 restraints

25 constraints

Primary atom site location: dual

H atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.4614P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.0004$  $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Fe1 | 0.303705 (15) | 0.660572 (18) | 0.661506 (14) | 0.02956 (7)                      |
| S2  | 0.19293 (8)   | 0.99058 (9)   | 0.47672 (7)   | 0.05597 (14)                     |
| S1  | 0.04375 (8)   | 0.63213 (10)  | 0.78148 (9)   | 0.06938 (18)                     |
| N6  | 0.25561 (8)   | 0.51496 (11)  | 0.57102 (8)   | 0.0322 (3)                       |
| N5  | 0.39330 (8)   | 0.73823 (10)  | 0.79114 (8)   | 0.0315 (3)                       |
| N4  | 0.38898 (9)   | 0.51771 (10)  | 0.74436 (8)   | 0.0308 (3)                       |
| N3  | 0.42603 (9)   | 0.65654 (12)  | 0.61390 (9)   | 0.0368 (3)                       |
| N1  | 0.19326 (10)  | 0.63321 (12)  | 0.71525 (10)  | 0.0427 (3)                       |
| N2  | 0.24561 (10)  | 0.79922 (13)  | 0.58421 (10)  | 0.0488 (4)                       |
| C19 | 0.27700 (10)  | 0.41519 (13)  | 0.61266 (10)  | 0.0318 (3)                       |
| C18 | 0.24902 (11)  | 0.31522 (14)  | 0.56442 (11)  | 0.0396 (4)                       |
| H18 | 0.26840 (11)  | 0.23496 (14)  | 0.59993 (11)  | 0.070 (6)*                       |
| C13 | 0.43134 (10)  | 0.66704 (13)  | 0.86149 (10)  | 0.0323 (3)                       |
| C9  | 0.41215 (11)  | 0.84774 (13)  | 0.80532 (11)  | 0.0362 (3)                       |

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|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| H9  | 0.38203 (11) | 0.90546 (13) | 0.74742 (11) | 0.054 (5)*  |
| C7  | 0.48908 (10) | 0.57568 (14) | 0.64979 (10) | 0.0362 (4)  |
| C15 | 0.20626 (11) | 0.51804 (15) | 0.48004 (11) | 0.0408 (4)  |
| H15 | 0.19005 (11) | 0.59938 (15) | 0.44577 (11) | 0.069 (6)*  |
| C2  | 0.22340 (12) | 0.87954 (14) | 0.53920 (11) | 0.0390 (4)  |
| C14 | 0.40503 (12) | 0.54582 (13) | 0.84206 (10) | 0.0380 (4)  |
| H14 | 0.34048 (12) | 0.52834 (13) | 0.85782 (10) | 0.056 (5)*  |
| H13 | 0.46205 (12) | 0.49300 (13) | 0.88748 (10) | 0.055 (5)*  |
| C20 | 0.32920 (12) | 0.41714 (13) | 0.71586 (10) | 0.0382 (4)  |
| H19 | 0.37377 (12) | 0.34202 (13) | 0.73603 (10) | 0.071 (6)*  |
| H20 | 0.27792 (12) | 0.41525 (13) | 0.75243 (10) | 0.061 (5)*  |
| C11 | 0.50708 (12) | 0.81699 (14) | 0.96319 (12) | 0.0431 (4)  |
| H11 | 0.55133 (12) | 0.84786 (14) | 1.03134 (12) | 0.072 (6)*  |
| C1  | 0.13075 (11) | 0.63162 (13) | 0.74272 (11) | 0.0381 (4)  |
| C12 | 0.48910 (11) | 0.70367 (14) | 0.94855 (11) | 0.0394 (4)  |
| H12 | 0.51993 (11) | 0.64385 (14) | 1.00469 (11) | 0.065 (6)*  |
| C10 | 0.46821 (11) | 0.89062 (14) | 0.89033 (12) | 0.0419 (4)  |
| H10 | 0.48145 (11) | 0.98040 (14) | 0.89966 (12) | 0.064 (6)*  |
| C17 | 0.19622 (12) | 0.31943 (16) | 0.47069 (12) | 0.0445 (4)  |
| H17 | 0.17175 (12) | 0.24248 (16) | 0.43136 (12) | 0.072 (6)*  |
| C8  | 0.47977 (12) | 0.50592 (16) | 0.72760 (12) | 0.0447 (4)  |
| H8  | 0.53763 (12) | 0.52807 (16) | 0.79189 (12) | 0.088 (7)*  |
| H7  | 0.48890 (12) | 0.41750 (16) | 0.71269 (12) | 0.083 (7)*  |
| C16 | 0.17483 (12) | 0.42264 (16) | 0.42740 (12) | 0.0472 (4)  |
| H16 | 0.13402 (12) | 0.42854 (16) | 0.35330 (12) | 0.072 (6)*  |
| C3  | 0.43654 (13) | 0.72133 (18) | 0.54715 (12) | 0.0513 (5)  |
| H3  | 0.38457 (13) | 0.78709 (18) | 0.51757 (12) | 0.068 (6)*  |
| C6  | 0.56488 (13) | 0.55732 (19) | 0.61937 (13) | 0.0549 (5)  |
| H6  | 0.61558 (13) | 0.49051 (19) | 0.64962 (13) | 0.095 (8)*  |
| C23 | 0.65080 (18) | 0.7738 (2)   | 0.78953 (16) | 0.0705 (6)  |
| H23 | 0.59831 (18) | 0.7452 (2)   | 0.81900 (16) | 0.0846 (8)* |
| C4  | 0.51023 (15) | 0.7088 (2)   | 0.51393 (14) | 0.0679 (6)  |
| H4  | 0.51701 (15) | 0.7642 (2)   | 0.45994 (14) | 0.097 (8)*  |
| N7  | 0.78328 (19) | 0.8450 (2)   | 0.71526 (17) | 0.0950 (7)  |
| C22 | 0.7357 (2)   | 0.7219 (2)   | 0.81096 (15) | 0.0756 (7)  |
| H22 | 0.7527 (2)   | 0.6505 (2)   | 0.85830 (15) | 0.0907 (8)* |
| C5  | 0.57494 (15) | 0.6250 (2)   | 0.55030 (15) | 0.0717 (7)  |
| H5  | 0.63355 (15) | 0.6120 (2)   | 0.52489 (15) | 0.112 (9)*  |
| C24 | 0.63285 (18) | 0.8625 (2)   | 0.73016 (18) | 0.0794 (7)  |
| H24 | 0.56570 (18) | 0.9073 (2)   | 0.71146 (18) | 0.0953 (8)* |
| C21 | 0.79969 (18) | 0.7596 (3)   | 0.77297 (17) | 0.0803 (7)  |
| H21 | 0.86778 (18) | 0.7171 (3)   | 0.79127 (17) | 0.0964 (9)* |
| C25 | 0.7002 (2)   | 0.8944 (2)   | 0.69462 (18) | 0.0879 (8)  |
| H25 | 0.6846 (2)   | 0.9648 (2)   | 0.64628 (18) | 0.1055 (9)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Fe1 | 0.03039 (11) | 0.02603 (12) | 0.03262 (12) | 0.00045 (9)  | 0.01130 (8) | 0.00351 (9)  |
| S2  | 0.0802 (4)   | 0.0404 (3)   | 0.0514 (3)   | 0.0121 (2)   | 0.0280 (3)  | 0.0156 (2)   |
| S1  | 0.0494 (3)   | 0.0791 (4)   | 0.0942 (4)   | −0.0241 (3)  | 0.0434 (3)  | −0.0422 (3)  |
| N6  | 0.0346 (7)   | 0.0277 (7)   | 0.0336 (7)   | 0.0031 (5)   | 0.0108 (5)  | 0.0015 (5)   |
| N5  | 0.0325 (6)   | 0.0258 (7)   | 0.0343 (7)   | −0.0015 (5)  | 0.0092 (5)  | 0.0026 (5)   |
| N4  | 0.0364 (7)   | 0.0242 (7)   | 0.0307 (6)   | 0.0007 (5)   | 0.0103 (5)  | 0.0048 (5)   |
| N3  | 0.0332 (7)   | 0.0432 (8)   | 0.0374 (7)   | −0.0006 (6)  | 0.0168 (6)  | 0.0067 (6)   |
| N1  | 0.0426 (8)   | 0.0386 (8)   | 0.0544 (9)   | −0.0055 (6)  | 0.0263 (7)  | −0.0068 (6)  |
| N2  | 0.0518 (9)   | 0.0396 (9)   | 0.0518 (9)   | 0.0100 (7)   | 0.0139 (7)  | 0.0146 (7)   |
| C19 | 0.0340 (8)   | 0.0273 (8)   | 0.0335 (8)   | −0.0004 (6)  | 0.0110 (6)  | 0.0002 (6)   |
| C18 | 0.0444 (9)   | 0.0322 (9)   | 0.0412 (9)   | −0.0036 (7)  | 0.0134 (7)  | −0.0038 (7)  |
| C13 | 0.0361 (8)   | 0.0280 (8)   | 0.0305 (7)   | −0.0007 (7)  | 0.0086 (6)  | 0.0032 (7)   |
| C9  | 0.0351 (8)   | 0.0250 (8)   | 0.0455 (9)   | 0.0002 (7)   | 0.0100 (7)  | 0.0020 (7)   |
| C7  | 0.0275 (8)   | 0.0442 (10)  | 0.0377 (8)   | −0.0007 (7)  | 0.0121 (6)  | −0.0029 (7)  |
| C15 | 0.0407 (9)   | 0.0414 (10)  | 0.0345 (8)   | 0.0075 (7)   | 0.0056 (7)  | 0.0035 (7)   |
| C2  | 0.0459 (9)   | 0.0347 (9)   | 0.0377 (8)   | 0.0070 (7)   | 0.0161 (7)  | 0.0060 (7)   |
| C14 | 0.0523 (10)  | 0.0269 (8)   | 0.0319 (8)   | −0.0020 (7)  | 0.0107 (7)  | 0.0053 (6)   |
| C20 | 0.0509 (10)  | 0.0250 (8)   | 0.0354 (8)   | −0.0043 (7)  | 0.0106 (7)  | 0.0041 (7)   |
| C11 | 0.0448 (9)   | 0.0375 (10)  | 0.0411 (9)   | −0.0025 (7)  | 0.0072 (8)  | −0.0075 (8)  |
| C1  | 0.0347 (9)   | 0.0338 (9)   | 0.0472 (9)   | −0.0044 (7)  | 0.0159 (7)  | −0.0113 (7)  |
| C12 | 0.0455 (9)   | 0.0350 (9)   | 0.0327 (8)   | −0.0004 (7)  | 0.0069 (7)  | 0.0004 (7)   |
| C10 | 0.0415 (9)   | 0.0281 (9)   | 0.0521 (10)  | −0.0038 (7)  | 0.0109 (8)  | −0.0062 (8)  |
| C17 | 0.0452 (9)   | 0.0434 (11)  | 0.0419 (9)   | −0.0053 (8)  | 0.0111 (8)  | −0.0093 (8)  |
| C8  | 0.0387 (9)   | 0.0455 (11)  | 0.0503 (10)  | 0.0112 (8)   | 0.0156 (8)  | 0.0099 (8)   |
| C16 | 0.0454 (10)  | 0.0532 (12)  | 0.0351 (9)   | 0.0027 (8)   | 0.0038 (7)  | −0.0044 (8)  |
| C3  | 0.0496 (10)  | 0.0632 (13)  | 0.0469 (10)  | −0.0036 (9)  | 0.0242 (8)  | 0.0169 (9)   |
| C6  | 0.0379 (10)  | 0.0767 (15)  | 0.0558 (11)  | 0.0048 (10)  | 0.0232 (8)  | −0.0063 (10) |
| C23 | 0.0814 (16)  | 0.0719 (16)  | 0.0748 (15)  | −0.0250 (13) | 0.0480 (13) | −0.0224 (13) |
| C4  | 0.0624 (13)  | 0.0961 (18)  | 0.0581 (12)  | −0.0129 (13) | 0.0372 (11) | 0.0129 (12)  |
| N7  | 0.0969 (18)  | 0.109 (2)    | 0.0958 (16)  | −0.0247 (15) | 0.0543 (14) | −0.0036 (15) |
| C22 | 0.106 (2)    | 0.0683 (16)  | 0.0556 (13)  | −0.0034 (15) | 0.0322 (13) | 0.0031 (11)  |
| C5  | 0.0501 (12)  | 0.114 (2)    | 0.0655 (13)  | −0.0018 (13) | 0.0391 (11) | 0.0015 (13)  |
| C24 | 0.0750 (16)  | 0.0741 (18)  | 0.0874 (17)  | 0.0070 (13)  | 0.0258 (14) | −0.0199 (14) |
| C21 | 0.0653 (15)  | 0.102 (2)    | 0.0751 (15)  | 0.0097 (14)  | 0.0256 (13) | −0.0082 (15) |
| C25 | 0.119 (2)    | 0.0649 (17)  | 0.0858 (18)  | −0.0083 (17) | 0.0432 (18) | 0.0127 (14)  |

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

|        |             |         |           |
|--------|-------------|---------|-----------|
| Fe1—N6 | 2.1897 (13) | C14—H13 | 1.1010    |
| Fe1—N5 | 2.1897 (12) | C20—H19 | 1.1010    |
| Fe1—N4 | 2.2508 (12) | C20—H20 | 1.1010    |
| Fe1—N3 | 2.2064 (12) | C11—H11 | 1.0930    |
| Fe1—N1 | 2.1232 (14) | C11—C12 | 1.386 (2) |
| Fe1—N2 | 2.0520 (15) | C11—C10 | 1.385 (2) |
| S2—C2  | 1.6098 (19) | C12—H12 | 1.0930    |

|            |             |             |             |
|------------|-------------|-------------|-------------|
| S1—C1      | 1.6164 (19) | C10—H10     | 1.0930      |
| N6—C19     | 1.3399 (19) | C17—H17     | 1.0930      |
| N6—C15     | 1.3375 (19) | C17—C16     | 1.387 (2)   |
| N5—C13     | 1.3392 (18) | C8—H8       | 1.1010      |
| N5—C9      | 1.3425 (19) | C8—H7       | 1.1010      |
| N4—C14     | 1.4726 (19) | C16—H16     | 1.0930      |
| N4—C20     | 1.4768 (19) | C3—H3       | 1.0930      |
| N4—C8      | 1.485 (2)   | C3—C4       | 1.381 (3)   |
| N3—C7      | 1.335 (2)   | C6—H6       | 1.0930      |
| N3—C3      | 1.337 (2)   | C6—C5       | 1.383 (3)   |
| N1—C1      | 1.158 (2)   | C23—H23     | 1.0930      |
| N2—C2      | 1.163 (2)   | C23—C22     | 1.358 (3)   |
| C19—C18    | 1.393 (2)   | C23—C24     | 1.364 (3)   |
| C19—C20    | 1.506 (2)   | C4—H4       | 1.0930      |
| C18—H18    | 1.0930      | C4—C5       | 1.377 (3)   |
| C18—C17    | 1.384 (2)   | N7—C21      | 1.318 (3)   |
| C13—C14    | 1.506 (2)   | N7—C25      | 1.322 (4)   |
| C13—C12    | 1.391 (2)   | C22—H22     | 1.0930      |
| C9—H9      | 1.0930      | C22—C21     | 1.365 (3)   |
| C9—C10     | 1.386 (2)   | C5—H5       | 1.0930      |
| C7—C8      | 1.502 (2)   | C24—H24     | 1.0930      |
| C7—C6      | 1.393 (2)   | C24—C25     | 1.362 (4)   |
| C15—H15    | 1.0930      | C21—H21     | 1.0930      |
| C15—C16    | 1.384 (2)   | C25—H25     | 1.0930      |
| C14—H14    | 1.1010      |             |             |
| N5—Fe1—N6  | 151.59 (5)  | C19—C20—N4  | 111.64 (12) |
| N4—Fe1—N6  | 75.77 (4)   | H19—C20—N4  | 109.29 (8)  |
| N4—Fe1—N5  | 75.83 (4)   | H19—C20—C19 | 109.29 (9)  |
| N3—Fe1—N6  | 84.55 (5)   | H20—C20—N4  | 109.29 (8)  |
| N3—Fe1—N5  | 89.13 (5)   | H20—C20—C19 | 109.29 (8)  |
| N3—Fe1—N4  | 77.87 (5)   | H20—C20—H19 | 108.0       |
| N1—Fe1—N6  | 89.46 (5)   | C12—C11—H11 | 120.44 (10) |
| N1—Fe1—N5  | 91.96 (5)   | C10—C11—H11 | 120.44 (10) |
| N1—Fe1—N4  | 92.06 (5)   | C10—C11—C12 | 119.11 (15) |
| N1—Fe1—N3  | 169.28 (5)  | N1—C1—S1    | 178.82 (15) |
| N2—Fe1—N6  | 107.12 (5)  | C11—C12—C13 | 119.15 (15) |
| N2—Fe1—N5  | 100.91 (5)  | H12—C12—C13 | 120.43 (10) |
| N2—Fe1—N4  | 170.98 (5)  | H12—C12—C11 | 120.43 (10) |
| N2—Fe1—N3  | 93.78 (5)   | C11—C10—C9  | 118.42 (15) |
| N2—Fe1—N1  | 96.49 (6)   | H10—C10—C9  | 120.79 (10) |
| C19—N6—Fe1 | 115.70 (9)  | H10—C10—C11 | 120.79 (10) |
| C15—N6—Fe1 | 125.71 (11) | H17—C17—C18 | 120.46 (11) |
| C15—N6—C19 | 118.57 (14) | C16—C17—C18 | 119.07 (16) |
| C13—N5—Fe1 | 114.93 (10) | C16—C17—H17 | 120.46 (10) |
| C9—N5—Fe1  | 126.25 (10) | C7—C8—N4    | 115.40 (13) |
| C9—N5—C13  | 118.82 (13) | H8—C8—N4    | 108.43 (8)  |
| C14—N4—Fe1 | 105.26 (9)  | H8—C8—C7    | 108.43 (9)  |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| C20—N4—Fe1     | 106.23 (9)   | H7—C8—N4        | 108.43 (9)   |
| C20—N4—C14     | 110.92 (11)  | H7—C8—C7        | 108.43 (9)   |
| C8—N4—Fe1      | 110.88 (9)   | H7—C8—H8        | 107.5        |
| C8—N4—C14      | 111.08 (12)  | C17—C16—C15     | 118.58 (15)  |
| C8—N4—C20      | 112.15 (13)  | H16—C16—C15     | 120.71 (10)  |
| C7—N3—Fe1      | 115.94 (10)  | H16—C16—C17     | 120.71 (10)  |
| C3—N3—Fe1      | 125.04 (12)  | H3—C3—N3        | 118.58 (10)  |
| C3—N3—C7       | 118.73 (14)  | C4—C3—N3        | 122.85 (19)  |
| C1—N1—Fe1      | 171.90 (13)  | C4—C3—H3        | 118.58 (13)  |
| C2—N2—Fe1      | 171.60 (14)  | H6—C6—C7        | 120.54 (11)  |
| C18—C19—N6     | 122.16 (14)  | C5—C6—C7        | 118.92 (19)  |
| C20—C19—N6     | 116.12 (13)  | C5—C6—H6        | 120.54 (13)  |
| C20—C19—C18    | 121.65 (14)  | C22—C23—H23     | 120.95 (15)  |
| H18—C18—C19    | 120.62 (9)   | C24—C23—H23     | 120.95 (16)  |
| C17—C18—C19    | 118.77 (16)  | C24—C23—C22     | 118.1 (2)    |
| C17—C18—H18    | 120.62 (11)  | H4—C4—C3        | 120.72 (13)  |
| C14—C13—N5     | 116.25 (13)  | C5—C4—C3        | 118.56 (19)  |
| C12—C13—N5     | 121.78 (14)  | C5—C4—H4        | 120.72 (11)  |
| C12—C13—C14    | 121.94 (14)  | C25—N7—C21      | 116.8 (2)    |
| H9—C9—N5       | 118.64 (9)   | H22—C22—C23     | 120.24 (15)  |
| C10—C9—N5      | 122.71 (15)  | C21—C22—C23     | 119.5 (2)    |
| C10—C9—H9      | 118.64 (10)  | C21—C22—H22     | 120.24 (17)  |
| C8—C7—N3       | 118.82 (13)  | C4—C5—C6        | 119.18 (18)  |
| C6—C7—N3       | 121.76 (16)  | H5—C5—C6        | 120.41 (13)  |
| C6—C7—C8       | 119.37 (16)  | H5—C5—C4        | 120.41 (11)  |
| H15—C15—N6     | 118.59 (10)  | H24—C24—C23     | 120.62 (16)  |
| C16—C15—N6     | 122.82 (16)  | C25—C24—C23     | 118.8 (2)    |
| C16—C15—H15    | 118.59 (10)  | C25—C24—H24     | 120.62 (18)  |
| N2—C2—S2       | 179.80 (16)  | C22—C21—N7      | 123.1 (2)    |
| C13—C14—N4     | 111.18 (12)  | H21—C21—N7      | 118.45 (16)  |
| H14—C14—N4     | 109.40 (8)   | H21—C21—C22     | 118.45 (17)  |
| H14—C14—C13    | 109.40 (9)   | C24—C25—N7      | 123.7 (3)    |
| H13—C14—N4     | 109.40 (8)   | H25—C25—N7      | 118.13 (17)  |
| H13—C14—C13    | 109.40 (8)   | H25—C25—C24     | 118.13 (18)  |
| H13—C14—H14    | 108.0        |                 |              |
| Fe1—N6—C19—C18 | 178.39 (10)  | C19—C20—N4—C8   | −80.45 (14)  |
| Fe1—N6—C19—C20 | 1.21 (11)    | C18—C19—N6—C15  | −0.42 (18)   |
| Fe1—N6—C15—C16 | −177.43 (13) | C18—C17—C16—C15 | −0.76 (19)   |
| Fe1—N5—C13—C14 | −1.61 (11)   | C13—N5—C9—C10   | −1.09 (16)   |
| Fe1—N5—C13—C12 | −179.33 (10) | C13—C14—N4—C20  | −157.85 (13) |
| Fe1—N5—C9—C10  | 178.61 (12)  | C13—C14—N4—C8   | 76.70 (15)   |
| Fe1—N4—C14—C13 | −43.36 (9)   | C13—C12—C11—C10 | −0.99 (18)   |
| Fe1—N4—C20—C19 | 40.83 (9)    | C9—N5—C13—C14   | 178.12 (14)  |
| Fe1—N4—C8—C7   | −9.72 (10)   | C9—N5—C13—C12   | 0.40 (16)    |
| Fe1—N3—C7—C8   | −8.44 (13)   | C9—C10—C11—C12  | 0.35 (18)    |
| Fe1—N3—C7—C6   | 174.00 (12)  | C7—N3—C3—C4     | −0.40 (19)   |
| Fe1—N3—C3—C4   | −173.94 (16) | C7—C8—N4—C14    | −126.39 (15) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| N6—C19—C18—C17  | −0.96 (17)   | C7—C8—N4—C20    | 108.85 (15)  |
| N6—C19—C20—N4   | −29.71 (14)  | C7—C6—C5—C4     | 0.3 (2)      |
| N6—C15—C16—C17  | −0.65 (19)   | C15—N6—C19—C20  | −177.59 (14) |
| N5—C13—C14—N4   | 31.96 (14)   | C14—C13—C12—C11 | −176.96 (16) |
| N5—C13—C12—C11  | 0.63 (17)    | C20—C19—C18—C17 | 176.06 (15)  |
| N5—C9—C10—C11   | 0.72 (18)    | C8—C7—N3—C3     | 177.43 (16)  |
| N4—C14—C13—C12  | −150.33 (13) | C8—C7—C6—C5     | −177.40 (18) |
| N4—C20—C19—C18  | 153.10 (13)  | C3—N3—C7—C6     | −0.12 (19)   |
| N4—C8—C7—N3     | 12.47 (18)   | C3—C4—C5—C6     | −0.8 (2)     |
| N4—C8—C7—C6     | −169.92 (15) | C23—C22—C21—N7  | −0.2 (3)     |
| N3—C7—C6—C5     | 0.1 (2)      | C23—C24—C25—N7  | −0.9 (3)     |
| N3—C3—C4—C5     | 0.9 (2)      | C22—C23—C24—C25 | 0.6 (3)      |
| C19—N6—C15—C16  | 1.24 (16)    | C22—C21—N7—C25  | −0.1 (3)     |
| C19—C18—C17—C16 | 1.53 (18)    | C24—C23—C22—C21 | −0.0 (3)     |
| C19—C20—N4—C14  | 154.70 (13)  | C24—C25—N7—C21  | 0.7 (3)      |

### Bis(thiocyanato- $\kappa N$ )[tris(pyridin-2-ylmethyl)amine- $\kappa^4 N, N', N'', N'''$ ]iron(II) pyridine monosolvate (py\_255)

#### Crystal data

[Fe(NCS)<sub>2</sub>(C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>)]·C<sub>5</sub>H<sub>5</sub>N

$M_r = 541.49$

Monoclinic,  $P2_1/n$

$a = 15.1196$  (10) Å

$b = 11.9834$  (5) Å

$c = 15.3909$  (10) Å

$\beta = 110.127$  (7)°

$V = 2618.3$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 1123.013$

$D_x = 1.374$  Mg m<sup>−3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 12325 reflections

$\theta = 2.1$ – $26.4^\circ$

$\mu = 0.76$  mm<sup>−1</sup>

$T = 255$  K

Block, yellow

$0.21 \times 0.08 \times 0.06$  mm

#### Data collection

Rigaku XtaLAB Synergy-R

diffractometer with a HyPix-Arc 100 detector

Radiation source: micro-focus sealed X-ray

tube, Mova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>−1</sup>

$\omega$  scans

Absorption correction: multi-scan

(CrysAlis PRO; Rigaku OD, 2025)

$T_{\min} = 0.758$ ,  $T_{\max} = 1.000$

38477 measured reflections

5349 independent reflections

4017 reflections with  $I \geq 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -18 \rightarrow 18$

$k = -14 \rightarrow 14$

$l = -19 \rightarrow 19$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.095$

$S = 1.08$

5349 reflections

354 parameters

0 restraints

25 constraints

Primary atom site location: dual

H atoms treated by a mixture of independent

and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.8343P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.52$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.35$  e Å<sup>−3</sup>

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| Fe1 | 0.69649 (2)  | 0.66196 (3)  | 0.33850 (2)   | 0.03374 (10)                     |
| S2  | 0.80799 (13) | 0.99110 (14) | 0.52297 (11)  | 0.0635 (2)                       |
| S1  | 0.95764 (12) | 0.63441 (16) | 0.22158 (14)  | 0.0798 (3)                       |
| N4  | 0.61138 (13) | 0.51911 (14) | 0.25577 (12)  | 0.0353 (4)                       |
| N6  | 0.74431 (13) | 0.51610 (15) | 0.42887 (12)  | 0.0375 (4)                       |
| N5  | 0.60705 (12) | 0.73914 (15) | 0.20856 (12)  | 0.0359 (4)                       |
| N3  | 0.57435 (13) | 0.65725 (17) | 0.38603 (13)  | 0.0418 (5)                       |
| N1  | 0.80709 (15) | 0.63463 (17) | 0.28500 (15)  | 0.0476 (5)                       |
| N2  | 0.75405 (16) | 0.80023 (19) | 0.41554 (16)  | 0.0553 (6)                       |
| C19 | 0.72268 (15) | 0.41648 (18) | 0.38714 (15)  | 0.0367 (5)                       |
| C18 | 0.75011 (17) | 0.3164 (2)   | 0.43513 (17)  | 0.0452 (6)                       |
| H18 | 0.73081 (17) | 0.2371 (2)   | 0.39996 (17)  | 0.060 (7)*                       |
| C7  | 0.51148 (16) | 0.5764 (2)   | 0.35015 (16)  | 0.0413 (5)                       |
| C1  | 0.87007 (17) | 0.63340 (19) | 0.25849 (17)  | 0.0432 (6)                       |
| C11 | 0.49346 (18) | 0.8174 (2)   | 0.03683 (18)  | 0.0497 (6)                       |
| H11 | 0.44978 (18) | 0.8476 (2)   | −0.03061 (18) | 0.076 (9)*                       |
| C12 | 0.51184 (18) | 0.7041 (2)   | 0.05205 (17)  | 0.0457 (6)                       |
| H12 | 0.48162 (18) | 0.6446 (2)   | −0.00324 (17) | 0.072 (9)*                       |
| C13 | 0.56927 (16) | 0.66778 (19) | 0.13876 (15)  | 0.0374 (5)                       |
| C2  | 0.77706 (18) | 0.8803 (2)   | 0.46087 (16)  | 0.0434 (6)                       |
| C20 | 0.67089 (18) | 0.41873 (19) | 0.28452 (16)  | 0.0446 (6)                       |
| H19 | 0.62675 (18) | 0.34449 (19) | 0.26449 (16)  | 0.083 (10)*                      |
| H20 | 0.72173 (18) | 0.41671 (19) | 0.24858 (16)  | 0.072 (9)*                       |
| C10 | 0.53197 (17) | 0.8910 (2)   | 0.10936 (18)  | 0.0487 (6)                       |
| H10 | 0.51874 (17) | 0.9797 (2)   | 0.09995 (18)  | 0.077 (9)*                       |
| C14 | 0.59554 (19) | 0.54692 (19) | 0.15847 (15)  | 0.0441 (6)                       |
| H14 | 0.65942 (19) | 0.52955 (19) | 0.14307 (15)  | 0.056 (7)*                       |
| H13 | 0.53925 (19) | 0.49455 (19) | 0.11364 (15)  | 0.072 (8)*                       |
| C9  | 0.58780 (16) | 0.84860 (19) | 0.19422 (17)  | 0.0421 (5)                       |
| H9  | 0.61714 (16) | 0.90590 (19) | 0.25135 (17)  | 0.057 (7)*                       |
| C17 | 0.80265 (18) | 0.3205 (2)   | 0.52889 (18)  | 0.0504 (6)                       |
| H17 | 0.82643 (18) | 0.2444 (2)   | 0.56791 (18)  | 0.082 (9)*                       |
| C15 | 0.79309 (17) | 0.5191 (2)   | 0.51962 (16)  | 0.0473 (6)                       |
| H15 | 0.80895 (17) | 0.5994 (2)   | 0.55380 (16)  | 0.065 (8)*                       |
| C8  | 0.52089 (18) | 0.5078 (2)   | 0.27208 (18)  | 0.0497 (6)                       |
| H8  | 0.46403 (18) | 0.5308 (2)   | 0.20866 (18)  | 0.095 (11)*                      |
| H7  | 0.51118 (18) | 0.4202 (2)   | 0.28586 (18)  | 0.079 (9)*                       |
| C16 | 0.82407 (18) | 0.4233 (2)   | 0.57154 (18)  | 0.0533 (7)                       |
| H16 | 0.86461 (18) | 0.4290 (2)   | 0.64469 (18)  | 0.091 (10)*                      |
| C3  | 0.5637 (2)   | 0.7217 (3)   | 0.45282 (19)  | 0.0593 (7)                       |
| H3  | 0.6148 (2)   | 0.7869 (3)   | 0.48213 (19)  | 0.077 (9)*                       |
| C6  | 0.4365 (2)   | 0.5578 (3)   | 0.3803 (2)    | 0.0625 (8)                       |
| H6  | 0.3865 (2)   | 0.4919 (3)   | 0.3499 (2)    | 0.086 (10)*                      |
| C23 | 0.3487 (3)   | 0.7737 (3)   | 0.2104 (3)    | 0.0833 (11)                      |
| H23 | 0.4009 (3)   | 0.7453 (3)   | 0.1818 (3)    | 0.0999 (13)*                     |

|     |            |            |            |              |
|-----|------------|------------|------------|--------------|
| C4  | 0.4904 (2) | 0.7084 (3) | 0.4858 (2) | 0.0789 (10)  |
| H4  | 0.4835 (2) | 0.7631 (3) | 0.5391 (2) | 0.114 (13)*  |
| C22 | 0.2646 (3) | 0.7217 (3) | 0.1890 (2) | 0.0865 (11)  |
| H22 | 0.2481 (3) | 0.6509 (3) | 0.1424 (2) | 0.1038 (13)* |
| N7  | 0.2171 (3) | 0.8448 (4) | 0.2840 (3) | 0.1129 (13)  |
| C5  | 0.4259 (2) | 0.6240 (4) | 0.4496 (2) | 0.0832 (11)  |
| H5  | 0.3683 (2) | 0.6101 (4) | 0.4750 (2) | 0.109 (12)*  |
| C24 | 0.3662 (3) | 0.8627 (4) | 0.2687 (3) | 0.0929 (12)  |
| H24 | 0.4323 (3) | 0.9075 (4) | 0.2864 (3) | 0.1115 (14)* |
| C25 | 0.2999 (4) | 0.8944 (3) | 0.3042 (3) | 0.1003 (13)  |
| H25 | 0.3154 (4) | 0.9641 (3) | 0.3518 (3) | 0.1203 (15)* |
| C21 | 0.2008 (3) | 0.7593 (4) | 0.2267 (3) | 0.0914 (11)  |
| H21 | 0.1336 (3) | 0.7171 (4) | 0.2088 (3) | 0.1096 (14)* |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|--------------|---------------|
| Fe1 | 0.03465 (17) | 0.02927 (17) | 0.03727 (18) | −0.00125 (14) | 0.01235 (13) | −0.00411 (13) |
| S2  | 0.0921 (6)   | 0.0447 (4)   | 0.0584 (4)   | −0.0145 (4)   | 0.0319 (4)   | −0.0183 (3)   |
| S1  | 0.0558 (5)   | 0.0905 (6)   | 0.1099 (7)   | 0.0279 (4)    | 0.0502 (5)   | 0.0499 (6)    |
| N4  | 0.0432 (11)  | 0.0258 (9)   | 0.0351 (10)  | 0.0000 (8)    | 0.0112 (8)   | −0.0043 (7)   |
| N6  | 0.0385 (10)  | 0.0341 (11)  | 0.0388 (10)  | −0.0033 (8)   | 0.0118 (8)   | −0.0009 (8)   |
| N5  | 0.0366 (10)  | 0.0293 (10)  | 0.0393 (10)  | 0.0017 (8)    | 0.0098 (8)   | −0.0027 (8)   |
| N3  | 0.0385 (11)  | 0.0489 (12)  | 0.0417 (10)  | 0.0002 (9)    | 0.0184 (8)   | −0.0068 (9)   |
| N1  | 0.0454 (13)  | 0.0431 (12)  | 0.0615 (13)  | 0.0057 (9)    | 0.0277 (11)  | 0.0058 (10)   |
| N2  | 0.0600 (14)  | 0.0447 (13)  | 0.0575 (13)  | −0.0125 (11)  | 0.0155 (11)  | −0.0166 (11)  |
| C19 | 0.0398 (12)  | 0.0297 (12)  | 0.0399 (12)  | 0.0002 (9)    | 0.0129 (10)  | −0.0012 (9)   |
| C18 | 0.0494 (14)  | 0.0357 (14)  | 0.0490 (14)  | 0.0035 (11)   | 0.0149 (11)  | 0.0039 (11)   |
| C7  | 0.0336 (12)  | 0.0492 (14)  | 0.0415 (12)  | −0.0010 (11)  | 0.0135 (10)  | 0.0043 (11)   |
| C1  | 0.0376 (13)  | 0.0379 (13)  | 0.0551 (14)  | 0.0064 (10)   | 0.0174 (11)  | 0.0130 (11)   |
| C11 | 0.0523 (15)  | 0.0432 (15)  | 0.0460 (14)  | 0.0037 (11)   | 0.0073 (12)  | 0.0096 (11)   |
| C12 | 0.0526 (15)  | 0.0392 (13)  | 0.0400 (13)  | 0.0000 (11)   | 0.0092 (11)  | −0.0001 (11)  |
| C13 | 0.0419 (12)  | 0.0328 (12)  | 0.0348 (12)  | 0.0011 (10)   | 0.0098 (10)  | −0.0035 (10)  |
| C2  | 0.0540 (14)  | 0.0371 (13)  | 0.0405 (13)  | −0.0080 (11)  | 0.0181 (11)  | −0.0060 (11)  |
| C20 | 0.0586 (16)  | 0.0295 (12)  | 0.0410 (13)  | 0.0038 (11)   | 0.0111 (12)  | −0.0044 (10)  |
| C10 | 0.0478 (14)  | 0.0347 (14)  | 0.0581 (16)  | 0.0038 (11)   | 0.0112 (12)  | 0.0049 (12)   |
| C14 | 0.0615 (16)  | 0.0320 (12)  | 0.0367 (12)  | 0.0020 (11)   | 0.0141 (11)  | −0.0060 (10)  |
| C9  | 0.0432 (13)  | 0.0284 (12)  | 0.0515 (14)  | −0.0012 (10)  | 0.0124 (11)  | −0.0024 (11)  |
| C17 | 0.0502 (15)  | 0.0499 (16)  | 0.0473 (15)  | 0.0052 (12)   | 0.0120 (12)  | 0.0110 (12)   |
| C15 | 0.0487 (14)  | 0.0463 (15)  | 0.0413 (13)  | −0.0083 (11)  | 0.0082 (11)  | −0.0043 (11)  |
| C8  | 0.0457 (14)  | 0.0490 (15)  | 0.0540 (15)  | −0.0109 (12)  | 0.0167 (12)  | −0.0096 (12)  |
| C16 | 0.0505 (15)  | 0.0599 (18)  | 0.0405 (14)  | −0.0018 (13)  | 0.0042 (11)  | 0.0041 (13)   |
| C3  | 0.0581 (17)  | 0.072 (2)    | 0.0546 (16)  | 0.0059 (15)   | 0.0280 (13)  | −0.0175 (14)  |
| C6  | 0.0466 (16)  | 0.084 (2)    | 0.0629 (17)  | −0.0066 (15)  | 0.0267 (13)  | 0.0060 (16)   |
| C23 | 0.097 (3)    | 0.084 (3)    | 0.088 (2)    | 0.030 (2)     | 0.056 (2)    | 0.026 (2)     |
| C4  | 0.075 (2)    | 0.112 (3)    | 0.064 (2)    | 0.016 (2)     | 0.0425 (17)  | −0.0124 (19)  |
| C22 | 0.120 (3)    | 0.083 (3)    | 0.061 (2)    | 0.006 (2)     | 0.036 (2)    | −0.0037 (18)  |
| N7  | 0.119 (3)    | 0.130 (3)    | 0.111 (3)    | 0.035 (3)     | 0.066 (3)    | 0.007 (3)     |

|     |           |           |           |            |             |            |
|-----|-----------|-----------|-----------|------------|-------------|------------|
| C5  | 0.058 (2) | 0.133 (3) | 0.074 (2) | 0.002 (2)  | 0.0421 (17) | 0.000 (2)  |
| C24 | 0.091 (3) | 0.086 (3) | 0.101 (3) | −0.007 (2) | 0.032 (2)   | 0.023 (2)  |
| C25 | 0.138 (4) | 0.071 (3) | 0.101 (3) | 0.010 (3)  | 0.052 (3)   | −0.011 (2) |
| C21 | 0.072 (2) | 0.118 (3) | 0.084 (3) | −0.012 (2) | 0.028 (2)   | 0.009 (2)  |

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| Fe1—N4    | 2.2538 (18) | C20—H20     | 1.0910      |
| Fe1—N6    | 2.1955 (19) | C10—H10     | 1.0830      |
| Fe1—N5    | 2.1961 (18) | C10—C9      | 1.385 (3)   |
| Fe1—N3    | 2.2088 (19) | C14—H14     | 1.0910      |
| Fe1—N1    | 2.128 (2)   | C14—H13     | 1.0910      |
| Fe1—N2    | 2.049 (2)   | C9—H9       | 1.0830      |
| S2—C2     | 1.609 (3)   | C17—H17     | 1.0830      |
| S1—C1     | 1.610 (3)   | C17—C16     | 1.381 (4)   |
| N4—C20    | 1.476 (3)   | C15—H15     | 1.0830      |
| N4—C14    | 1.471 (3)   | C15—C16     | 1.385 (4)   |
| N4—C8     | 1.480 (3)   | C8—H8       | 1.0910      |
| N6—C19    | 1.342 (3)   | C8—H7       | 1.0910      |
| N6—C15    | 1.337 (3)   | C16—H16     | 1.0830      |
| N5—C13    | 1.338 (3)   | C3—H3       | 1.0830      |
| N5—C9     | 1.345 (3)   | C3—C4       | 1.377 (4)   |
| N3—C7     | 1.336 (3)   | C6—H6       | 1.0830      |
| N3—C3     | 1.339 (3)   | C6—C5       | 1.382 (5)   |
| N1—C1     | 1.158 (3)   | C23—H23     | 1.0830      |
| N2—C2     | 1.166 (3)   | C23—C22     | 1.351 (5)   |
| C19—C18   | 1.394 (3)   | C23—C24     | 1.360 (5)   |
| C19—C20   | 1.502 (3)   | C4—H4       | 1.0830      |
| C18—H18   | 1.0830      | C4—C5       | 1.382 (5)   |
| C18—C17   | 1.388 (3)   | C22—H22     | 1.0830      |
| C7—C8     | 1.503 (3)   | C22—C21     | 1.363 (5)   |
| C7—C6     | 1.383 (3)   | N7—C25      | 1.323 (6)   |
| C11—H11   | 1.0830      | N7—C21      | 1.318 (5)   |
| C11—C12   | 1.390 (3)   | C5—H5       | 1.0830      |
| C11—C10   | 1.384 (4)   | C24—H24     | 1.0830      |
| C12—H12   | 1.0830      | C24—C25     | 1.350 (6)   |
| C12—C13   | 1.388 (3)   | C25—H25     | 1.0830      |
| C13—C14   | 1.505 (3)   | C21—H21     | 1.0830      |
| C20—H19   | 1.0910      |             |             |
| N6—Fe1—N4 | 75.69 (7)   | H20—C20—H19 | 107.9       |
| N5—Fe1—N4 | 75.67 (6)   | H10—C10—C11 | 120.72 (14) |
| N5—Fe1—N6 | 151.35 (7)  | C9—C10—C11  | 118.6 (2)   |
| N3—Fe1—N4 | 77.75 (7)   | C9—C10—H10  | 120.72 (14) |
| N3—Fe1—N6 | 84.28 (7)   | C13—C14—N4  | 111.25 (18) |
| N3—Fe1—N5 | 89.39 (7)   | H14—C14—N4  | 109.38 (13) |
| N1—Fe1—N4 | 92.09 (7)   | H14—C14—C13 | 109.38 (13) |
| N1—Fe1—N6 | 89.50 (7)   | H13—C14—N4  | 109.38 (12) |

|             |             |             |             |
|-------------|-------------|-------------|-------------|
| N1—Fe1—N5   | 91.84 (7)   | H13—C14—C13 | 109.38 (13) |
| N1—Fe1—N3   | 169.13 (8)  | H13—C14—H14 | 108.0       |
| N2—Fe1—N4   | 170.86 (8)  | C10—C9—N5   | 122.6 (2)   |
| N2—Fe1—N6   | 107.18 (8)  | H9—C9—N5    | 118.68 (13) |
| N2—Fe1—N5   | 101.08 (8)  | H9—C9—C10   | 118.68 (14) |
| N2—Fe1—N3   | 93.78 (8)   | H17—C17—C18 | 120.57 (15) |
| N2—Fe1—N1   | 96.58 (9)   | C16—C17—C18 | 118.9 (2)   |
| C20—N4—Fe1  | 106.20 (13) | C16—C17—H17 | 120.57 (15) |
| C14—N4—Fe1  | 105.33 (13) | H15—C15—N6  | 118.79 (14) |
| C14—N4—C20  | 111.02 (17) | C16—C15—N6  | 122.4 (2)   |
| C8—N4—Fe1   | 110.80 (13) | C16—C15—H15 | 118.79 (15) |
| C8—N4—C20   | 112.24 (19) | C7—C8—N4    | 115.67 (19) |
| C8—N4—C14   | 110.93 (19) | H8—C8—N4    | 108.36 (12) |
| C19—N6—Fe1  | 115.61 (14) | H8—C8—C7    | 108.36 (13) |
| C15—N6—Fe1  | 125.70 (16) | H7—C8—N4    | 108.36 (12) |
| C15—N6—C19  | 118.7 (2)   | H7—C8—C7    | 108.36 (14) |
| C13—N5—Fe1  | 114.90 (14) | H7—C8—H8    | 107.4       |
| C9—N5—Fe1   | 126.21 (15) | C15—C16—C17 | 119.2 (2)   |
| C9—N5—C13   | 118.89 (19) | H16—C16—C17 | 120.41 (15) |
| C7—N3—Fe1   | 116.02 (15) | H16—C16—C15 | 120.41 (15) |
| C3—N3—Fe1   | 125.11 (18) | H3—C3—N3    | 118.61 (16) |
| C3—N3—C7    | 118.6 (2)   | C4—C3—N3    | 122.8 (3)   |
| C1—N1—Fe1   | 171.6 (2)   | C4—C3—H3    | 118.6 (2)   |
| C2—N2—Fe1   | 172.3 (2)   | H6—C6—C7    | 120.23 (17) |
| C18—C19—N6  | 122.2 (2)   | C5—C6—C7    | 119.5 (3)   |
| C20—C19—N6  | 116.12 (19) | C5—C6—H6    | 120.2 (2)   |
| C20—C19—C18 | 121.7 (2)   | C22—C23—H23 | 120.8 (2)   |
| H18—C18—C19 | 120.67 (14) | C24—C23—H23 | 120.8 (3)   |
| C17—C18—C19 | 118.7 (2)   | C24—C23—C22 | 118.5 (4)   |
| C17—C18—H18 | 120.67 (15) | H4—C4—C3    | 120.6 (2)   |
| C8—C7—N3    | 118.5 (2)   | C5—C4—C3    | 118.8 (3)   |
| C6—C7—N3    | 121.8 (2)   | C5—C4—H4    | 120.62 (18) |
| C6—C7—C8    | 119.6 (2)   | H22—C22—C23 | 120.4 (2)   |
| N1—C1—S1    | 178.8 (2)   | C21—C22—C23 | 119.2 (4)   |
| C12—C11—H11 | 120.59 (15) | C21—C22—H22 | 120.4 (3)   |
| C10—C11—H11 | 120.59 (14) | C21—N7—C25  | 116.8 (4)   |
| C10—C11—C12 | 118.8 (2)   | C4—C5—C6    | 118.5 (3)   |
| H12—C12—C11 | 120.30 (15) | H5—C5—C6    | 120.7 (2)   |
| C13—C12—C11 | 119.4 (2)   | H5—C5—C4    | 120.73 (18) |
| C13—C12—H12 | 120.30 (14) | H24—C24—C23 | 120.5 (3)   |
| C12—C13—N5  | 121.7 (2)   | C25—C24—C23 | 118.9 (4)   |
| C14—C13—N5  | 116.31 (19) | C25—C24—H24 | 120.5 (3)   |
| C14—C13—C12 | 122.0 (2)   | C24—C25—N7  | 123.6 (4)   |
| N2—C2—S2    | 179.5 (3)   | H25—C25—N7  | 118.2 (3)   |
| C19—C20—N4  | 111.92 (18) | H25—C25—C24 | 118.2 (3)   |
| H19—C20—N4  | 109.23 (12) | N7—C21—C22  | 123.1 (4)   |
| H19—C20—C19 | 109.23 (13) | H21—C21—C22 | 118.5 (3)   |
| H20—C20—N4  | 109.23 (12) | H21—C21—N7  | 118.5 (3)   |



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| H20—C20—C19     | 109.23 (13)  |                 |            |
| Fe1—N4—C20—C19  | 40.83 (14)   | C19—C20—N4—C8   | −80.4 (2)  |
| Fe1—N4—C14—C13  | −43.40 (14)  | C18—C19—N6—C15  | −0.6 (3)   |
| Fe1—N4—C8—C7    | −10.53 (14)  | C18—C17—C16—C15 | −0.4 (3)   |
| Fe1—N6—C19—C18  | 178.57 (16)  | C7—N3—C3—C4     | −0.3 (3)   |
| Fe1—N6—C19—C20  | 1.29 (17)    | C7—C8—N4—C20    | 108.0 (2)  |
| Fe1—N6—C15—C16  | −177.4 (2)   | C7—C8—N4—C14    | −127.1 (2) |
| Fe1—N5—C13—C12  | −179.39 (16) | C7—C6—C5—C4     | 0.9 (3)    |
| Fe1—N5—C13—C14  | −1.70 (17)   | C11—C12—C13—C14 | −177.1 (2) |
| Fe1—N5—C9—C10   | 178.70 (19)  | C12—C11—C10—C9  | 0.3 (3)    |
| Fe1—N3—C7—C8    | −8.78 (19)   | C12—C13—N5—C9   | 0.8 (3)    |
| Fe1—N3—C7—C6    | 173.95 (18)  | C13—N5—C9—C10   | −1.5 (2)   |
| Fe1—N3—C3—C4    | −173.8 (2)   | C13—C12—C11—C10 | −1.0 (3)   |
| N4—C20—C19—N6   | −29.8 (2)    | C13—C14—N4—C20  | −157.9 (2) |
| N4—C20—C19—C18  | 152.93 (19)  | C13—C14—N4—C8   | 76.5 (2)   |
| N4—C14—C13—N5   | 32.0 (2)     | C20—C19—N6—C15  | −177.9 (2) |
| N4—C14—C13—C12  | −150.3 (2)   | C20—C19—C18—C17 | 176.2 (2)  |
| N4—C8—C7—N3     | 13.3 (3)     | C14—C13—N5—C9   | 178.4 (2)  |
| N4—C8—C7—C6     | −169.4 (2)   | C8—C7—N3—C3     | 177.1 (2)  |
| N6—C19—C18—C17  | −0.9 (3)     | C8—C7—C6—C5     | −177.4 (3) |
| N6—C15—C16—C17  | −1.2 (3)     | C3—N3—C7—C6     | −0.2 (3)   |
| N5—C13—C12—C11  | 0.5 (3)      | C3—C4—C5—C6     | −1.3 (4)   |
| N5—C9—C10—C11   | 0.9 (3)      | C23—C22—C21—N7  | −0.2 (4)   |
| N3—C7—C6—C5     | −0.2 (3)     | C23—C24—C25—N7  | −1.4 (4)   |
| N3—C3—C4—C5     | 1.0 (4)      | C22—C23—C24—C25 | 1.1 (4)    |
| C19—N6—C15—C16  | 1.7 (2)      | C22—C21—N7—C25  | −0.0 (5)   |
| C19—C18—C17—C16 | 1.4 (3)      | C24—C23—C22—C21 | −0.4 (4)   |
| C19—C20—N4—C14  | 154.8 (2)    | C24—C25—N7—C21  | 0.8 (5)    |

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