



# Crystal structure of bis{3-(3-bromo-4-methoxyphenyl)-5-[6-(1*H*-pyrazol-1-yl)pyridin-2-yl]-1,2,4-triazol-3-ato}iron(II) methanol disolvate

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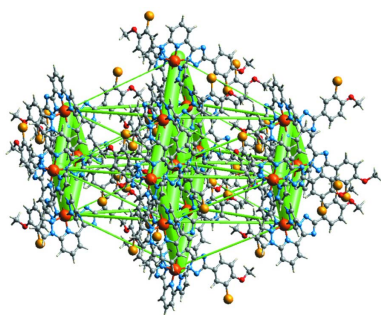
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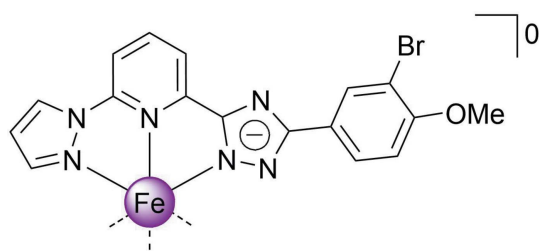
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The unit cell of the title compound, [Fe<sup>II</sup>(C<sub>17</sub>H<sub>12</sub>BrN<sub>6</sub>O)<sub>2</sub>].2MeOH, consists of a charge-neutral complex molecule and two independent molecules of methanol. In the complex molecule, the two tridentate ligand molecules 2-[5-(3-bromo-4-methoxyphenyl)-4*H*-1,2,4-triazol-3-yl]-6-(1*H*-pyrazol-1-yl)pyridine coordinate to the Fe<sup>II</sup> ion through the N atoms of the pyrazole, pyridine and triazole groups, forming a pseudo-octahedral coordination sphere around the central ion. In the crystal, neighbouring asymmetric molecules are linked through weak C—H(pz) ···π(ph) interactions into chains, which are then linked into layers by weak C—H ···N/C interactions. Finally, the layers stack into a three-dimensional network linked by weak interlayer C—H ···π interactions between the methoxy groups and the phenyl rings. The intermolecular contacts were quantified using Hirshfeld surface analysis and two-dimensional fingerprint plots, revealing the relative contributions of the contacts to the crystal packing to be H ···H 34.2%, H ···C/C ···H 25.2%, H ···Br/Br ···H 13.2%, H ···N/N ···H 12.2% and H ···O/O ···H 4.0%. The average Fe—N bond distance is 1.949 Å, indicating the low-spin state of the Fe<sup>II</sup> ion. Energy framework analysis at the HF/3–21 G theory level was performed to quantify the interaction energies in the crystal structure.

## 1. Chemical context

A broad class of coordination compounds exhibiting spin-state switching between low- (total spin  $S = 0$ ) and high-spin states (total spin  $S = 2$ ) is represented by Fe<sup>II</sup> complexes based on tridentate bisazolepyridine ligands (Halcrow, 2014; Suryadevara *et al.*, 2022; Halcrow *et al.*, 2019). In the case of asymmetric ligand design, where one of the azole groups carries a hydrogen on a nitrogen heteroatom and acts as a Brønsted acid, deprotonation can produce neutral complexes that can be either high-spin (Schäfer *et al.*, 2013) or low-spin (Shiga *et al.*, 2019) or exhibit temperature-induced transitions between the spin states of the central atom (Seredyuk *et al.*, 2014), depending on the ligand field strength. The periphery of the molecule, *i.e.* ligand substituents, also plays an important role in the behaviour, determining the way in which molecules are packed in the lattice and their interactions with each other, and therefore further influencing the spin state adopted by the central atom. As we have recently demonstrated, the dynamic rearrangement of the methoxy group between the bent and extended configurations can lead to a highly hysteretic spin transition *via* a supramolecular blocking mechanism (Seredyuk *et al.*, 2022).





Having interest in spin-transition 3d-metal complexes formed by polydentate ligands (Bartual-Murgui *et al.*, 2017; Bonhommeau *et al.*, 2012; Valverde-Muñoz *et al.*, 2020), we report here on our current structural exploration of a new complex  $[\text{Fe}^{\text{II}}L_2]$  based on an asymmetric deprotonable ligand with two substituents on the phenyl group,  $L = 2\text{-}[5\text{-}(3\text{-bromo-4-methoxyphenyl})\text{-}4H\text{-}1,2,4\text{-triazol-}3\text{-yl}]\text{-}6\text{-}(1H\text{-pyrazol-}1\text{-yl})\text{-pyridine}$ .

## 2. Structural commentary

The title complex has a asymmetric molecule with divergent phenyl groups. The ligand molecules are almost planar (r.m.s. deviation = 0.330 Å), including the methoxy substituents, which also lie in the plane of the aromatic groups [atoms C17 and C35 are 0.514 (1) and 0.116 (1) Å, respectively, away from the planes passing through their respective ligand molecules]. The two independent methanol molecules form O—H···N hydrogen bonds with the triazole (trz) rings of the ligand molecules (Fig. 1, Table 1). The central  $\text{Fe}^{\text{II}}$  ion of the complex has a distorted octahedral  $N_6$  coordination environment formed by the nitrogen donor atoms of two tridentate ligands (Fig. 1).

The average bond length,  $\langle \text{Fe}-\text{N} \rangle = 1.949$  Å, is typical for low-spin complexes with an  $N_6$  coordination environment (Gütlich & Goodwin, 2004). The average trigonal distortion parameters  $\Sigma = \Sigma_1^{12}(|90 - \varphi_i|)$ , where  $\varphi_i$  is the angle  $\text{N}-\text{Fe}-\text{N}'$  (Drew *et al.*, 1995), and  $\Theta = \Sigma_1^{24}(|60 - \theta_i|)$ , where  $\theta_i$  is the angle generated by superposition of two opposite faces of an

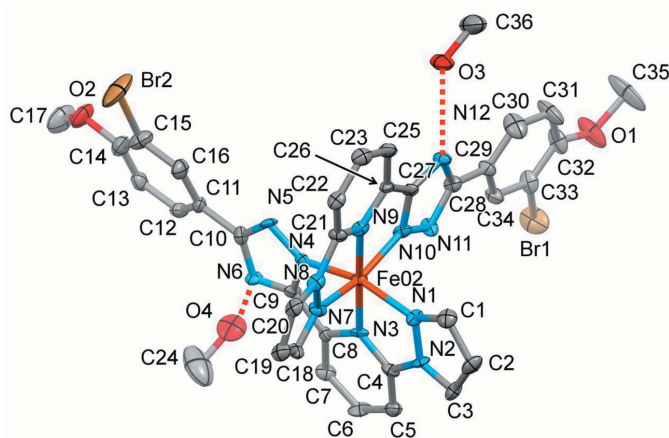


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity. Hydrogen bonds are indicated by dashed lines.

Table 1

Geometry (Å, °) of hydrogen bonds and C···N interactions in the title compound.

Cg1 and Cg2 are the centroids of the C11–C16 and C29–C34 rings, respectively.

| D—H···A                      | D—H  | H···A | D···A      | D—H···A |
|------------------------------|------|-------|------------|---------|
| C17···N6 <sup>i</sup>        |      |       | 3.201 (16) |         |
| O3—H3A···N12                 | 0.84 | 2.02  | 2.820 (12) | 160     |
| O4—H4···N6                   | 0.84 | 2.06  | 2.855 (11) | 158     |
| C1—H1···O4 <sup>ii</sup>     | 0.95 | 2.22  | 3.128 (14) | 161     |
| C18—H18···O3 <sup>iii</sup>  | 0.95 | 2.27  | 3.192 (14) | 163     |
| C35—H35A···C30 <sup>iv</sup> | 0.98 | 2.62  | 3.233 (16) | 121     |
| C3—H3···N5 <sup>iii</sup>    | 0.95 | 2.45  | 3.301 (13) | 148     |
| C7—H7···O4                   | 0.95 | 2.46  | 3.310 (11) | 148     |
| C22—H22···N11 <sup>ii</sup>  | 0.95 | 2.39  | 3.317 (13) | 166     |
| C20—H20···N11 <sup>ii</sup>  | 0.95 | 2.55  | 3.389 (13) | 148     |
| C5—H5···N5 <sup>iii</sup>    | 0.95 | 2.53  | 3.440 (12) | 161     |
| C17—H17A···O4 <sup>i</sup>   | 0.98 | 2.52  | 3.451 (17) | 159     |
| C34—H34···C20 <sup>v</sup>   | 0.95 | 2.63  | 3.535 (15) | 159     |
| C25—H25···O3                 | 0.95 | 2.69  | 3.542 (13) | 150     |
| C18—H18···C36 <sup>iii</sup> | 0.95 | 2.88  | 3.65 (2)   | 138     |
| C2—H2···C31 <sup>vi</sup>    | 0.95 | 2.84  | 3.639 (15) | 143     |
| C2—H2···C32 <sup>vi</sup>    | 0.95 | 2.89  | 3.656 (15) | 139     |
| C2—H2···C30 <sup>vi</sup>    | 0.95 | 2.86  | 3.734 (11) | 154     |
| C2—H2···Cg2 <sup>vi</sup>    | 0.95 | 2.57  | 3.501 (11) | 168     |
| C19—H19···Cg1 <sup>vi</sup>  | 0.95 | 2.74  | 3.681 (11) | 169     |

Symmetry codes: (i)  $-x+1, -y+1, z-\frac{1}{2}$ ; (ii)  $-x+\frac{3}{2}, y+\frac{1}{2}, z+\frac{1}{2}$ ; (iii)  $-x+\frac{3}{2}, y-\frac{1}{2}, z+\frac{1}{2}$ ; (iv)  $-x+2, -y+1, z-\frac{1}{2}$ ; (v)  $-x+\frac{3}{2}, y-\frac{1}{2}, z-\frac{1}{2}$ ; (vi)  $x, y, z+1$ .

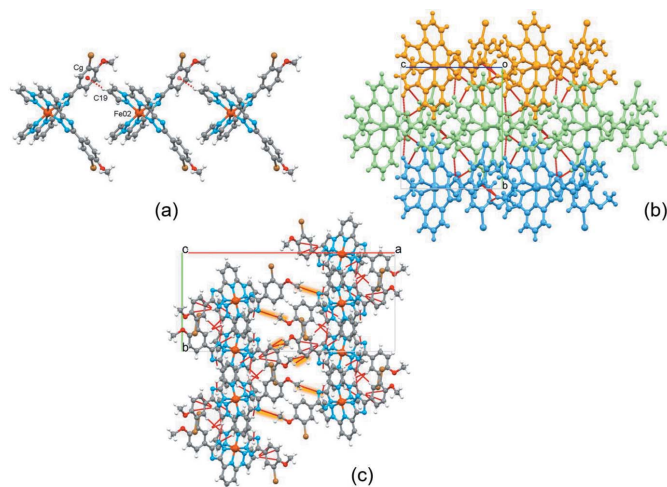
octahedron (Chang *et al.*, 1990) are 93.3 and 298.8°, respectively. The values reveal a deviation of the coordination environment from an ideal octahedron (where  $\Sigma = \Theta = 0$ ) but is, however, in the expected range for bisazolepyridines and similar ligands (see below). The calculated continuous shape measure (CSHM) value relative to the ideal  $O_h$  symmetry is 2.24 (Kershaw Cook *et al.*, 2015). The volume of the  $[\text{Fe}N_6]$  coordination polyhedron is 9.536 Å<sup>3</sup>.

## 3. Supramolecular features

As a result of their asymmetric shape, neighbouring complex molecules fit into each other and interact through a weak C—H(pz)··· $\pi$ (ph) intermolecular contact between the pyrazole (pz) and phenyl (ph) groups respectively (Table 1). The mono-periodic supramolecular chains formed extend along the *c*-axis direction with a stacking periodicity of 10.6434 (3) Å (equal to cell parameter *c*; Fig. 2a). Through weak intermolecular C—H(pz, py)···N/C(pz, trz) interactions in the range 3.128 (14)–3.734 (11) Å (Table 1), neighbouring chains are linked into corrugated layers in the *bc* plane (Fig. 2b,c). The layers stack with interlayer interactions limited to C—H···N(trz) and C—H··· $\pi$ (ph) contacts involving the methyl groups (Fig. 2c). The voids between the layers are occupied by methanol molecules, which also participate in bonding between neighbouring layers (see Table 1 for the complete list of intermolecular interactions).

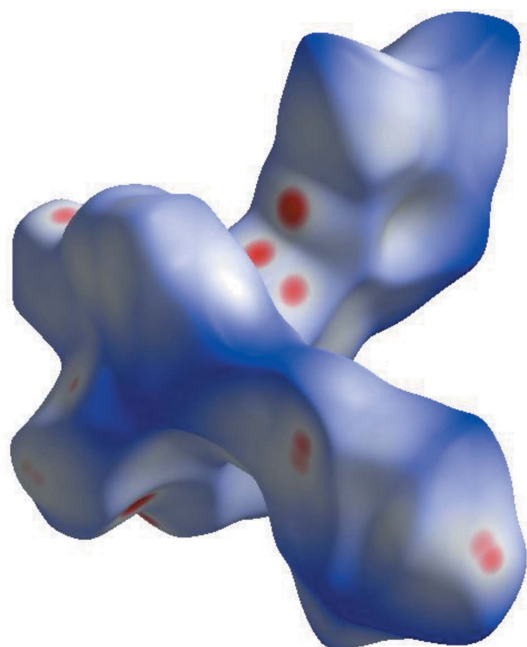
## 4. Hirshfeld surface and 2D fingerprint plots

Hirshfeld surface analysis was performed and the associated two-dimensional fingerprint plots were generated using



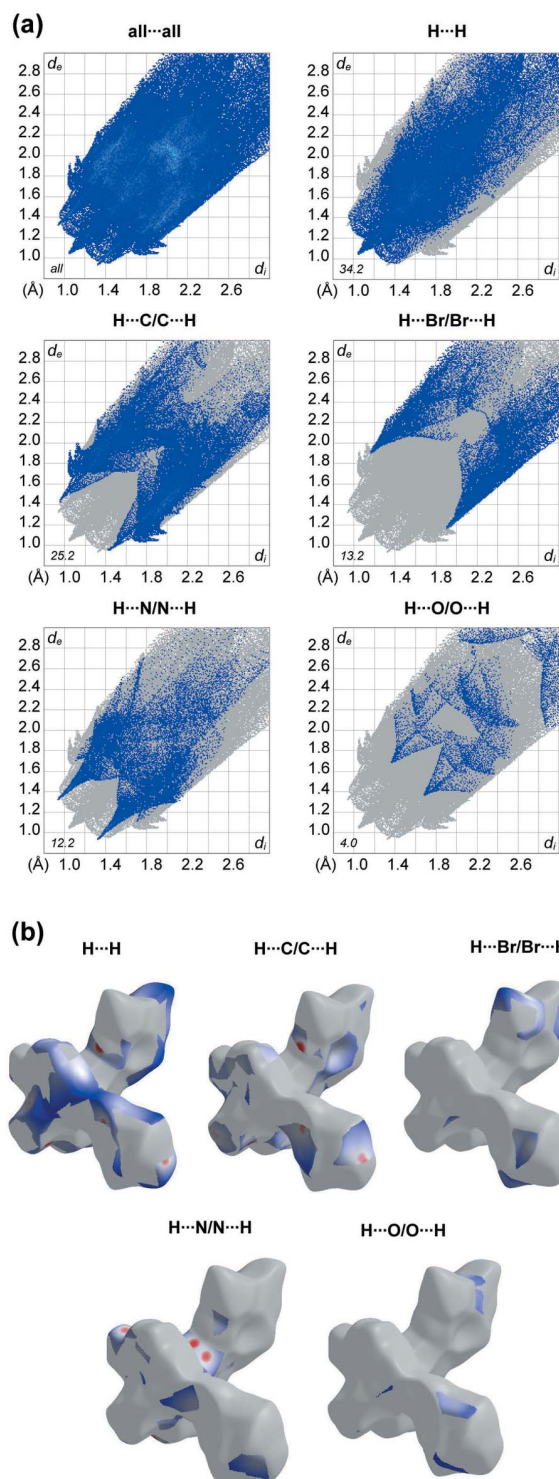
**Figure 2**  
 (a) Mono-periodic supramolecular chain formed by stacking of molecules of the title compound. (b) Di-periodic layers formed by supramolecular chains. For a better representation, each chain has a different colour. (c) Highlighted interactions of neighbouring layers in the three-dimensional supramolecular network of the title complex. The red dashed lines correspond to contacts below the sum of the van der Waals radii. The methanol molecules are not shown for clarity.

*CrystalExplorer* (Spackman *et al.*, 2021), with a standard resolution of the three-dimensional  $d_{\text{norm}}$  surfaces plotted over a fixed colour scale of  $-0.2869$  (red) to  $2.4335$  (blue) a.u. (Fig. 3). The pale-red spots represent short contacts and negative  $d_{\text{norm}}$  values on the surface corresponding to the interactions described above. The overall two-dimensional



**Figure 3**  
 A projection of  $d_{\text{norm}}$  mapped on the Hirshfeld surface, showing the intermolecular interactions within the molecule. Red areas represent regions where contacts are shorter than the sum of the van der Waals radii, blue areas represent regions where contacts are larger than the sum of van der Waals radii, and white areas are regions where contacts are close to the sum of van der Waals radii.

fingerprint plot is illustrated in Fig. 4. The Hirshfeld surfaces mapped over  $d_{\text{norm}}$  are shown for the  $\text{H}\cdots\text{H}$ ,  $\text{H}\cdots\text{C}/\text{C}\cdots\text{H}$ ,  $\text{H}\cdots\text{Br}/\text{Br}\cdots\text{H}$ ,  $\text{H}\cdots\text{N}/\text{N}\cdots\text{H}$  and  $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$  contacts together with the two-dimensional fingerprint plots associated with their relative contributions to the Hirshfeld surface. At 34.2%, the largest contribution to the overall crystal packing is



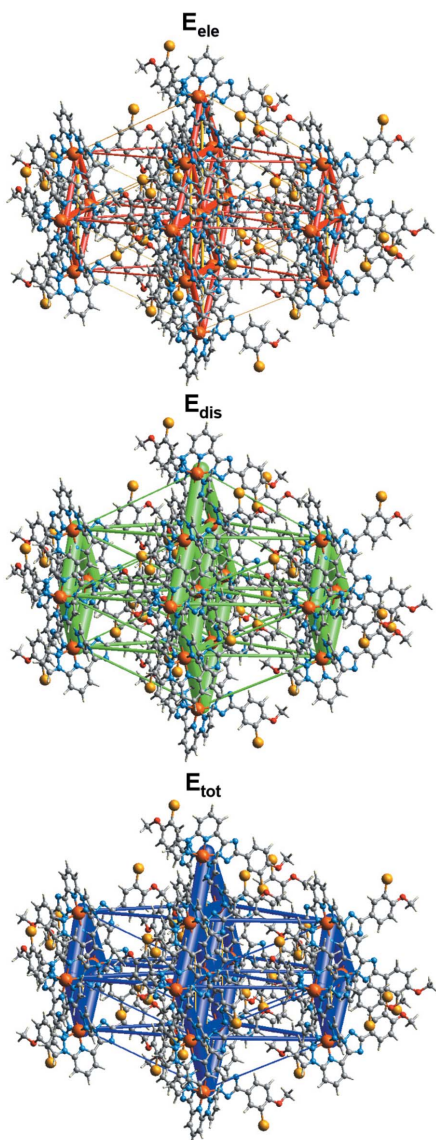
**Figure 4**  
 (a) The overall two-dimensional fingerprint plot and those decomposed into specified interactions. (b) Hirshfeld surface representations with the function  $d_{\text{norm}}$  plotted onto the surface for the different interactions.



from H···H interactions, which are located in the middle region of the fingerprint plot. H···C/C···H contacts contribute 25.2%, and the H···Br/Br···H contacts contribute 13.2% to the Hirshfeld surface and both result in a pair of characteristic wings. The H···N/N···H contacts, represented by a pair of sharp spikes in the fingerprint plot, make a 12.2% contribution to the Hirshfeld surface. Finally, H···O/O···H contacts, which account for 4.0% of the contribution, are mostly distributed in the middle part of the plot.

## 5. Energy framework analysis

The energy framework (Spackman *et al.*, 2021), calculated using the wave function at the HF/3-21G theory level, including the electrostatic potential forces ( $E_{\text{ele}}$ ), the dispersion forces ( $E_{\text{dis}}$ ) and the total energy diagrams ( $E_{\text{tot}}$ ), are shown in Fig. 5. The cylindrical radii, adjusted to the same



**Figure 5**  
The calculated energy frameworks, showing the electrostatic potential forces ( $E_{\text{ele}}$ ), dispersion forces ( $E_{\text{dis}}$ ) and total energy ( $E_{\text{tot}}$ ) diagrams. Tube size is set at 100 scale, cut-off is  $5 \text{ kJ mol}^{-1}$ .

**Table 2**

Computed distortion indices ( $\text{\AA},^\circ$ ) for the title compound and similar complexes reported in the literature.

| CSD refcode           | Spin state | $\langle \text{Fe}-\text{N} \rangle$ | $\Sigma$ | $\Theta$ | CShM( $O_h$ ) |
|-----------------------|------------|--------------------------------------|----------|----------|---------------|
| Title compound        | LS         | 1.949                                | 93.3     | 298.8    | 2.24          |
| IGERIX <sup>a</sup>   | HS         | 2.179                                | 149.7    | 553.2    | 6.06          |
| IGERIX01 <sup>a</sup> | LS         | 1.986                                | 105.6    | 350.6    | 2.85          |
| LUTGEO <sup>b</sup>   | LS         | 1.933                                | 85.0     | 309.6    | 2.10          |
| XODCEB <sup>c</sup>   | LS         | 1.950                                | 87.4     | 276.6    | 1.93          |
| DOMQIH <sup>d</sup>   | LS         | 1.962                                | 83.8     | 280.7    | 2.02          |
| QIDJET01 <sup>e</sup> | LS         | 1.970                                | 90.3     | 341.3    | 2.47          |
| QIDJET <sup>c</sup>   | HS         | 2.184                                | 145.5    | 553.3    | 5.88          |
| DOMQUT <sup>d</sup>   | LS         | 1.991                                | 88.5     | 320.0    | 2.48          |
| DOMQUT02 <sup>d</sup> | HS         | 2.183                                | 139.6    | 486.9    | 5.31          |

Notes: (a) Gentili *et al.* (2015); (b) Senthil Kumar *et al.* (2015); (c) Shiga *et al.* (2019); (d) Seredyuk *et al.* (2014); (e) Zhang *et al.* (2007).

scale factor of 100, are proportional to the relative strength of the corresponding energies. The major contribution to the intermolecular interactions is due to the dispersion forces ( $E_{\text{dis}}$ ), reflecting the dominating interactions in the lattice of the neutral asymmetric molecules. The topology of the energy framework resembles the topology of the interactions within and between the layers described above. The calculated values  $E_{\text{tot}}$  are in the range  $65.2\text{--}87.6 \text{ kJ mol}^{-1}$  for intrachain and intralayer interactions, whereas for the interlayer interactions they are within  $7.7\text{--}23.4 \text{ kJ mol}^{-1}$ . The colour-coded interaction mappings within a radius of  $3.8 \text{ \AA}$  of a central reference molecule for the title compound together with full details of the various contributions to the total energy ( $E_{\text{tot}}$ ) are given in the supporting information.

## 6. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42, last update February 2021; Groom *et al.*, 2016) reveals several similar neutral  $\text{Fe}^{\text{II}}$  complexes with a deprotonable azole group, for example, derivatives of a pyrazole-pyridine-tetrazole (IGERIX and LUTGEO; Gentili *et al.*, 2015; Senthil Kumar *et al.*, 2015) and a pyrazole-pyridine-benzimidazole (XODCEB; Shiga *et al.*, 2019). There are also related complexes based on phenanthroline-tetrazole, such as QIDJET (Zhang *et al.*, 2007) and phenanthroline-benzimidazole (DOMQUT; Seredyuk *et al.*, 2014). Schematic structures of the complexes are shown in Fig. S1 in the supporting information. The Fe–N distances of these complexes in the low-spin state are  $1.933\text{--}1.959 \text{ \AA}$ , while in the high-spin state they are in the range  $2.179\text{--}2.184 \text{ \AA}$ . The values of the trigonal distortion and CShM( $O_h$ ) change correspondingly, and in the low-spin state they are systematically lower than in the high-spin state. Table 2 collates the structural parameters of the complexes and of the title compound.

## 7. Synthesis and crystallization

The synthesis of the title compound is identical to that reported recently for a similar complex (Seredyuk *et al.*, 2022). It was produced by layering in a standard test tube. The

**Table 3**  
Experimental details.

|  |   |
|--|---|
| Crystal data   |   |
| Chemical formula   | [Fe(C <sub>17</sub> H <sub>12</sub> BrN <sub>6</sub> O) <sub>2</sub> ] <sub>2</sub> ·2CH <sub>4</sub> O   |
| <i>M</i> <sub>r</sub>  | 912.40  |
| Crystal system, space group  | Orthorhombic, <i>Pna</i> 2 <sub>1</sub>   |
| Temperature (K)  | 180   |
| <i>a</i> , <i>b</i> , <i>c</i> (Å)   | 27.4318 (10), 12.6723 (4),<br>10.6434 (3)   |
| <i>V</i> (Å <sup>3</sup> )   | 3699.9 (2)  |
| <i>Z</i>   | 4   |
| Radiation type   | Mo <i>K</i> α   |
| μ (mm <sup>-1</sup> )  | 2.63  |
| Crystal size (mm)  | 0.3 × 0.26 × 0.04   |
| Data collection  |   |
| Diffraction  | Xcalibur, Eos   |
| Absorption correction  | Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2022)   |
| <i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>  | 0.772, 1.000  |
| No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections                             | 14160, 6227, 4361   |
| <i>R</i> <sub>int</sub>  | 0.061   |
| (sin θ/λ) <sub>max</sub> (Å <sup>-1</sup> )  | 0.595   |
| Refinement   |   |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i> | 0.057, 0.125, 1.03  |
| No. of reflections   | 6227  |
| No. of parameters  | 502   |
| No. of restraints  | 7   |
| H-atom treatment   | H-atom parameters constrained   |
| Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )   | 1.25, -0.62   |
| Absolute structure   | Flack <i>x</i> determined using 1444<br>quotients [( <i>I</i> <sup>+</sup> ) - ( <i>I</i> <sup>-</sup> )] / [( <i>I</i> <sup>+</sup> ) + ( <i>I</i> <sup>-</sup> )]<br>(Parsons <i>et al.</i> , 2013) |
| Absolute structure parameter   | -0.009 (8)  |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

layering sequence was as follows: the bottom layer contains a solution of [Fe(*L*<sub>2</sub>)](BF<sub>4</sub>)<sub>2</sub> prepared by dissolving *L* = 2-[5-(3-bromo-4-methoxyphenyl)-4*H*-1,2,4-triazol-3-yl]-6-(1*H*-pyrazol-1-yl)pyridine (100 mg, 0.252 mmol) and Fe(BF<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O (43 mg, 0.126 mmol) in boiling acetone, to which chloroform (5 ml) was then added. The middle layer was a methanol-chloroform mixture (1:10, 10 ml), which was covered by a layer of methanol (10 ml), to which 100 μl of NEt<sub>3</sub> was added dropwise. The tube was sealed, and black cubic single crystals appeared in 3–4 weeks (yield *ca* 60%). Elemental analysis calculated for C<sub>36</sub>H<sub>32</sub>Br<sub>2</sub>FeN<sub>12</sub>O<sub>4</sub>: C, 47.39; H, 3.54; N, 18.42. Found: C, 47.11; H, 3.74; N, 18.40.

## 8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The highest and lowest remaining electron density peaks are located 1.01 and 0.88 Å, respectively, from the Br2 atom. H atoms were refined as riding [C–H = 0.95–0.98 Å with *U*<sub>iso</sub>(H) = 1.2–1.5*U*<sub>eq</sub>(C)]. O-bound H atoms were refined with *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(O).

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Author contributions are as follows: Conceptualization, KZ and MS; methodology, KZ; formal analysis, IOF; synthesis,

SOM; single-crystal measurements, SS; writing (original draft), MS; writing (review and editing of the manuscript), TYS, MS; visualization and calculations, KZ, VMA; funding acquisition, MS, IOF, VMA.

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## References

- Bartual-Murgui, C., Piñeiro-López, L., Valverde-Muñoz, F. J., Muñoz, M. C., Seredyuk, M. & Real, J. A. (2017). *Inorg. Chem.* **56**, 13535–13546.
- Bonhommeau, S., Lacroix, P. G., Talaga, D., Bousseksou, A., Seredyuk, M., Fritsky, I. O. & Rodriguez, V. (2012). *J. Phys. Chem. C*, **116**, 11251–11255.
- Chang, H. R., McCusker, J. K., Toftlund, H., Wilson, S. R., Trautwein, A. X., Winkler, H. & Hendrickson, D. N. (1990). *J. Am. Chem. Soc.* **112**, 6814–6827.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Drew, M. G. B., Harding, C. J., McKee, V., Morgan, G. G. & Nelson, J. (1995). *J. Chem. Soc. Chem. Commun.* pp. 1035–1038.
- Gentili, D., Demitri, N., Schäfer, B., Liscio, F., Bergenti, I., Ruani, G., Ruben, M. & Cavallini, M. (2015). *J. Mater. Chem. C*, **3**, 7836–7844.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Gütlich, P. & Goodwin, H. A. (2004). *Top. Curr. Chem.* **233**, 1–47.
- Halcrow, M. A. (2014). *New J. Chem.* **38**, 1868–1882.
- Halcrow, M. A., Capel Berdiell, I., Pask, C. M. & Kulmaczewski, R. (2019). *Inorg. Chem.* **58**, 9811–9821.
- Kershaw Cook, L. J., Mohammed, R., Sherborne, G., Roberts, T. D., Alvarez, S. & Halcrow, M. A. (2015). *Coord. Chem. Rev.* **289–290**, 2–12.
- Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* **B69**, 249–259.
- Rigaku OD (2022). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
- Schäfer, B., Rajnák, C., Šalitraš, I., Fuhr, O., Klar, D., Schmitz-Antoniak, C., Weschke, E., Wende, H. & Ruben, M. (2013). *Chem. Commun.* **49**, 10986.
- Senthil Kumar, K., Šalitraš, I., Heinrich, B., Fuhr, O. & Ruben, M. (2015). *J. Mater. Chem. C*, **3**, 11635–11644.
- Seredyuk, M., Znovjyak, K., Valverde-Muñoz, F. J., da Silva, I., Muñoz, M. C., Moroz, Y. S. & Real, J. A. (2022). *J. Am. Chem. Soc.* <https://doi.org/10.1021/jacs.2c05417>.
- Seredyuk, M., Znovjyak, K. O., Kusz, J., Nowak, M., Muñoz, M. C. & Real, J. A. (2014). *Dalton Trans.* **43**, 16387–16394.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Shiga, T., Saiki, R., Akiyama, L., Kumai, R., Natke, D., Renz, F., Cameron, J. M., Newton, G. N. & Oshio, H. (2019). *Angew. Chem. Int. Ed.* **58**, 5658–5662.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Suryadevara, N., Mizuno, A., Spieker, L., Salamon, S., Sleziona, S., Maas, A., Pollmann, E., Heinrich, B., Schleberger, M., Wende, H., Kuppasamy, S. K. & Ruben, M. (2022). *Chem. Eur. J.* **28**, e202103853.
- Valverde-Muñoz, F., Seredyuk, M., Muñoz, M. C., Molnár, G., Bibik, Y. S. & Real, J. A. (2020). *Angew. Chem. Int. Ed.* **59**, 18632–18638.
- Zhang, W., Zhao, F., Liu, T., Yuan, M., Wang, Z. M. & Gao, S. (2007). *Inorg. Chem.* **46**, 2541–2555.

## supporting information

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## Crystal structure of bis{3-(3-bromo-4-methoxyphenyl)-5-[6-(1*H*-pyrazol-1-yl)pyridin-2-yl]-1,2,4-triazol-3-ato}iron(II) methanol disolvate

**Kateryna Znovjyak, Igor O. Fritsky, Tatiana Y. Sliva, Vladimir M. Amirkhanov, Sergey O. Malinkin, Sergiu Shova and Maksym Seredyuk**

### Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2022); cell refinement: *CrysAlis PRO* (Rigaku OD, 2022); data reduction: *CrysAlis PRO* (Rigaku OD, 2022); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### Bis{3-(3-bromo-4-methoxyphenyl)-5-[6-(1*H*-pyrazol-1-yl)pyridin-2-yl]-1,2,4-triazol-3-ato}iron(II) methanol disolvate

#### Crystal data

[Fe(C<sub>17</sub>H<sub>12</sub>BrN<sub>6</sub>O)<sub>2</sub>]·2CH<sub>4</sub>O

*M<sub>r</sub>* = 912.40

Orthorhombic, *Pna*2<sub>1</sub>

*a* = 27.4318 (10) Å

*b* = 12.6723 (4) Å

*c* = 10.6434 (3) Å

*V* = 3699.9 (2) Å<sup>3</sup>

*Z* = 4

*F*(000) = 1840

*D<sub>x</sub>* = 1.638 Mg m<sup>-3</sup>

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 3167 reflections

θ = 2.2–25.7°

μ = 2.63 mm<sup>-1</sup>

*T* = 180 K

Plate, clear dark red

0.3 × 0.26 × 0.04 mm

#### Data collection

Xcalibur, Eos  
diffractometer

Radiation source: fine-focus sealed X-ray tube,  
Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.1593 pixels mm<sup>-1</sup>

ω scans

Absorption correction: multi-scan  
(*CrysAlisPro*; Rigaku OD, 2022)

*T<sub>min</sub>* = 0.772, *T<sub>max</sub>* = 1.000

14160 measured reflections

6227 independent reflections

4361 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.061

θ<sub>max</sub> = 25.0°, θ<sub>min</sub> = 1.8°

*h* = -29→32

*k* = -15→9

*l* = -11→12

#### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.057

*wR*(*F*<sup>2</sup>) = 0.125

*S* = 1.03

6227 reflections

502 parameters

7 restraints

Primary atom site location: dual

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.25 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.62 \text{ e } \text{Å}^{-3}$

Absolute structure: Flack  $x$  determined using  
 1444 quotients  $[(F^+)-(F^-)]/[(F^+)+(F^-)]$  (Parsons *et al.*, 2013)  
 Absolute structure parameter:  $-0.009$  (8)

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

|     | <i>x</i>    | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| Br1 | 0.93947 (6) | 0.18179 (10) | 0.18738 (16) | 0.0755 (6)                       |
| Br2 | 0.58117 (5) | 0.86423 (9)  | 0.16575 (14) | 0.0533 (4)                       |
| Fe1 | 0.74880 (4) | 0.51357 (9)  | 0.66488 (14) | 0.0165 (3)                       |
| O1  | 0.9850 (3)  | 0.3512 (6)   | 0.0344 (7)   | 0.046 (2)                        |
| O2  | 0.5033 (3)  | 0.7175 (7)   | 0.0845 (8)   | 0.059 (3)                        |
| N1  | 0.7925 (3)  | 0.4642 (7)   | 0.7986 (7)   | 0.021 (2)                        |
| N2  | 0.7854 (3)  | 0.3616 (6)   | 0.8310 (6)   | 0.0190 (19)                      |
| N3  | 0.7331 (2)  | 0.3663 (5)   | 0.6688 (8)   | 0.0169 (16)                      |
| N4  | 0.6980 (3)  | 0.5090 (7)   | 0.5358 (7)   | 0.019 (2)                        |
| N5  | 0.6735 (3)  | 0.5777 (6)   | 0.4591 (7)   | 0.021 (2)                        |
| N6  | 0.6424 (3)  | 0.4144 (6)   | 0.4290 (7)   | 0.021 (2)                        |
| N7  | 0.7030 (3)  | 0.5599 (6)   | 0.7956 (7)   | 0.0179 (19)                      |
| N8  | 0.7095 (3)  | 0.6637 (6)   | 0.8297 (7)   | 0.0203 (19)                      |
| N9  | 0.7639 (2)  | 0.6605 (5)   | 0.6733 (7)   | 0.0157 (16)                      |
| N10 | 0.8015 (3)  | 0.5208 (7)   | 0.5376 (7)   | 0.0167 (19)                      |
| N11 | 0.8252 (3)  | 0.4538 (7)   | 0.4581 (7)   | 0.020 (2)                        |
| N12 | 0.8561 (3)  | 0.6197 (6)   | 0.4353 (7)   | 0.020 (2)                        |
| C1  | 0.8232 (4)  | 0.5020 (9)   | 0.8838 (9)   | 0.022 (3)                        |
| H1  | 0.836027    | 0.571689     | 0.884392     | 0.027*                           |
| C2  | 0.8342 (4)  | 0.4225 (8)   | 0.9744 (9)   | 0.027 (3)                        |
| H2  | 0.854203    | 0.429649     | 1.046766     | 0.032*                           |
| C3  | 0.8103 (3)  | 0.3351 (8)   | 0.9362 (9)   | 0.022 (2)                        |
| H3  | 0.810827    | 0.267895     | 0.975646     | 0.027*                           |
| C4  | 0.7534 (3)  | 0.3030 (8)   | 0.7529 (8)   | 0.020 (2)                        |
| C5  | 0.7444 (3)  | 0.1970 (8)   | 0.7604 (8)   | 0.020 (2)                        |
| H5  | 0.760428    | 0.153348     | 0.819944     | 0.024*                           |
| C6  | 0.7104 (3)  | 0.1564 (7)   | 0.6761 (9)   | 0.026 (2)                        |
| H6  | 0.703580    | 0.082904     | 0.675727     | 0.032*                           |
| C7  | 0.6865 (3)  | 0.2219 (8)   | 0.5934 (8)   | 0.023 (2)                        |
| H7  | 0.662303    | 0.194369     | 0.538543     | 0.028*                           |
| C8  | 0.6979 (3)  | 0.3278 (8)   | 0.5907 (8)   | 0.018 (2)                        |
| C9  | 0.6790 (3)  | 0.4128 (8)   | 0.5154 (7)   | 0.016 (2)                        |
| C10 | 0.6407 (4)  | 0.5181 (8)   | 0.3985 (8)   | 0.020 (2)                        |
| C11 | 0.6061 (3)  | 0.5658 (8)   | 0.3117 (8)   | 0.021 (2)                        |

|      |            |             |              |           |
|------|------------|-------------|--------------|-----------|
| C12  | 0.5688 (4) | 0.5087 (9)  | 0.2563 (10)  | 0.042 (3) |
| H12  | 0.566690   | 0.435012    | 0.271727     | 0.051*    |
| C13  | 0.5339 (4) | 0.5569 (9)  | 0.1775 (13)  | 0.049 (3) |
| H13  | 0.508661   | 0.515389    | 0.141389     | 0.059*    |
| C14  | 0.5360 (4) | 0.6632 (9)  | 0.1525 (11)  | 0.034 (3) |
| C15  | 0.5737 (3) | 0.7203 (9)  | 0.2037 (9)   | 0.029 (3) |
| C16  | 0.6080 (4) | 0.6728 (8)  | 0.2824 (9)   | 0.026 (3) |
| H16  | 0.633427   | 0.714686    | 0.316951     | 0.031*    |
| C17  | 0.4597 (5) | 0.6610 (12) | 0.0437 (14)  | 0.085 (6) |
| H17A | 0.436960   | 0.710675    | 0.004257     | 0.128*    |
| H17B | 0.443970   | 0.628133    | 0.116611     | 0.128*    |
| H17C | 0.468752   | 0.606305    | -0.017004    | 0.128*    |
| C18  | 0.6699 (4) | 0.5218 (9)  | 0.8754 (9)   | 0.027 (3) |
| H18  | 0.656975   | 0.452322    | 0.872907     | 0.033*    |
| C19  | 0.6568 (4) | 0.6007 (9)  | 0.9651 (9)   | 0.028 (3) |
| H19  | 0.634576   | 0.593789    | 1.033020     | 0.033*    |
| C20  | 0.6829 (4) | 0.6878 (8)  | 0.9327 (9)   | 0.022 (2) |
| H20  | 0.682436   | 0.753910    | 0.974832     | 0.026*    |
| C21  | 0.7426 (3) | 0.7227 (8)  | 0.7576 (8)   | 0.019 (2) |
| C22  | 0.7510 (4) | 0.8292 (8)  | 0.7670 (9)   | 0.024 (2) |
| H22  | 0.734218   | 0.871698    | 0.826391     | 0.029*    |
| C23  | 0.7855 (3) | 0.8719 (7)  | 0.6848 (9)   | 0.028 (2) |
| H23  | 0.792262   | 0.945451    | 0.686681     | 0.033*    |
| C25  | 0.8098 (4) | 0.8074 (8)  | 0.6009 (9)   | 0.024 (2) |
| H25  | 0.833605   | 0.836305    | 0.545977     | 0.029*    |
| C26  | 0.7993 (3) | 0.7007 (8)  | 0.5971 (8)   | 0.015 (2) |
| C27  | 0.8201 (3) | 0.6167 (8)  | 0.5207 (8)   | 0.017 (2) |
| C28  | 0.8572 (3) | 0.5168 (9)  | 0.3993 (8)   | 0.022 (2) |
| C29  | 0.8908 (3) | 0.4756 (8)  | 0.3034 (9)   | 0.021 (2) |
| C30  | 0.9141 (4) | 0.5434 (9)  | 0.2193 (8)   | 0.028 (3) |
| H30  | 0.908122   | 0.617120    | 0.223076     | 0.034*    |
| C31  | 0.9464 (4) | 0.5030 (10) | 0.1295 (9)   | 0.029 (3) |
| H31  | 0.962898   | 0.550064    | 0.074511     | 0.034*    |
| C32  | 0.9544 (4) | 0.3969 (10) | 0.1196 (9)   | 0.034 (3) |
| C33  | 0.9309 (4) | 0.3287 (9)  | 0.2023 (10)  | 0.037 (3) |
| C34  | 0.8993 (4) | 0.3676 (9)  | 0.2928 (9)   | 0.029 (3) |
| H34  | 0.883359   | 0.320139    | 0.348246     | 0.035*    |
| C35  | 1.0089 (5) | 0.4170 (10) | -0.0556 (11) | 0.062 (4) |
| H35A | 1.026381   | 0.372877    | -0.116380    | 0.093*    |
| H35B | 1.032148   | 0.463288    | -0.012460    | 0.093*    |
| H35C | 0.984662   | 0.460036    | -0.099718    | 0.093*    |
| O3   | 0.8946 (3) | 0.8132 (7)  | 0.3501 (8)   | 0.054 (2) |
| H3A  | 0.889770   | 0.749515    | 0.368323     | 0.080*    |
| C36  | 0.9355 (6) | 0.8495 (13) | 0.4134 (18)  | 0.101 (6) |
| H36A | 0.964669   | 0.816116    | 0.378324     | 0.151*    |
| H36B | 0.932720   | 0.831820    | 0.502734     | 0.151*    |
| H36C | 0.937869   | 0.926235    | 0.403763     | 0.151*    |
| O4   | 0.6173 (3) | 0.2092 (6)  | 0.3391 (7)   | 0.040 (2) |



|      |            |             |             |           |
|------|------------|-------------|-------------|-----------|
| H4   | 0.620155   | 0.274742    | 0.348513    | 0.059*    |
| C24  | 0.5796 (4) | 0.1885 (10) | 0.2562 (11) | 0.054 (4) |
| H24A | 0.581928   | 0.115298    | 0.227028    | 0.082*    |
| H24B | 0.581953   | 0.236323    | 0.184082    | 0.082*    |
| H24C | 0.548314   | 0.199154    | 0.298680    | 0.082*    |

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

|     | $U^{11}$    | $U^{22}$   | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|-------------|------------|-------------|-------------|-------------|-------------|
| Br1 | 0.0982 (11) | 0.0278 (8) | 0.1007 (13) | 0.0159 (8)  | 0.0583 (11) | -0.0030 (9) |
| Br2 | 0.0659 (8)  | 0.0299 (7) | 0.0640 (8)  | 0.0033 (6)  | -0.0228 (9) | 0.0170 (8)  |
| Fe1 | 0.0203 (6)  | 0.0113 (7) | 0.0179 (6)  | -0.0009 (6) | -0.0005 (7) | 0.0006 (8)  |
| O1  | 0.048 (5)   | 0.041 (6)  | 0.050 (5)   | 0.001 (4)   | 0.030 (4)   | -0.010 (4)  |
| O2  | 0.048 (5)   | 0.050 (6)  | 0.077 (6)   | -0.010 (5)  | -0.044 (4)  | 0.030 (5)   |
| N1  | 0.023 (5)   | 0.016 (5)  | 0.023 (5)   | -0.008 (4)  | 0.003 (4)   | 0.001 (4)   |
| N2  | 0.028 (5)   | 0.011 (5)  | 0.019 (4)   | -0.001 (4)  | 0.007 (3)   | 0.007 (4)   |
| N3  | 0.027 (4)   | 0.011 (4)  | 0.013 (4)   | -0.003 (3)  | 0.008 (4)   | 0.004 (4)   |
| N4  | 0.028 (5)   | 0.015 (5)  | 0.013 (4)   | 0.001 (4)   | 0.000 (3)   | -0.001 (4)  |
| N5  | 0.021 (5)   | 0.010 (5)  | 0.031 (5)   | -0.005 (4)  | -0.001 (4)  | 0.009 (4)   |
| N6  | 0.025 (5)   | 0.015 (5)  | 0.022 (5)   | -0.002 (4)  | -0.004 (4)  | 0.000 (4)   |
| N7  | 0.024 (5)   | 0.010 (5)  | 0.020 (4)   | -0.002 (4)  | 0.008 (4)   | 0.004 (4)   |
| N8  | 0.030 (5)   | 0.009 (5)  | 0.022 (5)   | 0.002 (4)   | 0.001 (4)   | -0.007 (4)  |
| N9  | 0.026 (4)   | 0.009 (4)  | 0.013 (4)   | -0.001 (3)  | -0.008 (4)  | -0.002 (4)  |
| N10 | 0.015 (4)   | 0.016 (5)  | 0.018 (4)   | 0.000 (4)   | -0.004 (3)  | 0.002 (4)   |
| N11 | 0.027 (5)   | 0.014 (5)  | 0.017 (5)   | -0.005 (4)  | 0.005 (4)   | -0.010 (4)  |
| N12 | 0.024 (5)   | 0.010 (5)  | 0.026 (5)   | -0.004 (4)  | 0.004 (4)   | -0.003 (4)  |
| C1  | 0.019 (6)   | 0.013 (6)  | 0.034 (6)   | -0.003 (5)  | -0.003 (5)  | -0.002 (5)  |
| C2  | 0.033 (6)   | 0.022 (7)  | 0.025 (6)   | 0.008 (6)   | -0.006 (5)  | 0.001 (5)   |
| C3  | 0.029 (6)   | 0.018 (6)  | 0.020 (5)   | 0.011 (5)   | -0.005 (4)  | 0.005 (4)   |
| C4  | 0.025 (6)   | 0.018 (6)  | 0.015 (5)   | -0.001 (5)  | -0.004 (4)  | 0.001 (4)   |
| C5  | 0.027 (6)   | 0.013 (6)  | 0.020 (5)   | 0.003 (5)   | 0.003 (4)   | 0.007 (4)   |
| C6  | 0.034 (5)   | 0.020 (5)  | 0.026 (6)   | -0.005 (5)  | 0.007 (6)   | 0.001 (6)   |
| C7  | 0.028 (6)   | 0.024 (7)  | 0.018 (5)   | 0.000 (5)   | -0.005 (4)  | -0.005 (5)  |
| C8  | 0.018 (5)   | 0.013 (6)  | 0.022 (5)   | -0.003 (5)  | 0.004 (4)   | -0.006 (4)  |
| C9  | 0.021 (6)   | 0.016 (6)  | 0.009 (5)   | 0.001 (5)   | -0.002 (4)  | 0.000 (4)   |
| C10 | 0.025 (6)   | 0.018 (7)  | 0.018 (5)   | 0.001 (5)   | -0.002 (4)  | 0.003 (4)   |
| C11 | 0.022 (6)   | 0.021 (6)  | 0.021 (5)   | -0.005 (5)  | -0.002 (4)  | 0.003 (5)   |
| C12 | 0.039 (8)   | 0.023 (7)  | 0.064 (8)   | -0.007 (6)  | -0.007 (6)  | 0.015 (6)   |
| C13 | 0.039 (6)   | 0.042 (8)  | 0.066 (8)   | -0.014 (6)  | -0.033 (7)  | 0.012 (8)   |
| C14 | 0.026 (5)   | 0.041 (7)  | 0.035 (6)   | -0.002 (5)  | -0.009 (5)  | 0.021 (6)   |
| C15 | 0.026 (6)   | 0.031 (7)  | 0.028 (6)   | 0.001 (5)   | -0.002 (5)  | 0.013 (5)   |
| C16 | 0.026 (6)   | 0.021 (6)  | 0.031 (6)   | -0.003 (5)  | -0.007 (5)  | -0.001 (5)  |
| C17 | 0.069 (10)  | 0.081 (12) | 0.105 (11)  | -0.027 (9)  | -0.065 (9)  | 0.032 (9)   |
| C18 | 0.031 (7)   | 0.018 (7)  | 0.032 (6)   | 0.000 (6)   | 0.001 (5)   | 0.004 (5)   |
| C19 | 0.039 (7)   | 0.028 (7)  | 0.017 (5)   | 0.006 (6)   | 0.009 (5)   | 0.000 (5)   |
| C20 | 0.027 (6)   | 0.019 (6)  | 0.019 (5)   | 0.009 (5)   | 0.007 (4)   | 0.003 (5)   |
| C21 | 0.022 (6)   | 0.015 (6)  | 0.019 (5)   | -0.005 (5)  | -0.006 (4)  | 0.003 (4)   |
| C22 | 0.026 (6)   | 0.023 (6)  | 0.023 (5)   | 0.001 (5)   | -0.005 (4)  | -0.005 (5)  |

|     |            |            |            |             |             |            |
|-----|------------|------------|------------|-------------|-------------|------------|
| C23 | 0.046 (6)  | 0.009 (5)  | 0.028 (6)  | -0.002 (5)  | -0.001 (6)  | -0.002 (5) |
| C25 | 0.030 (6)  | 0.018 (6)  | 0.023 (5)  | -0.007 (5)  | 0.001 (4)   | 0.005 (5)  |
| C26 | 0.013 (5)  | 0.014 (6)  | 0.020 (5)  | -0.002 (5)  | -0.002 (4)  | 0.002 (4)  |
| C27 | 0.021 (5)  | 0.021 (6)  | 0.010 (5)  | -0.007 (5)  | 0.003 (4)   | -0.001 (4) |
| C28 | 0.016 (5)  | 0.025 (7)  | 0.023 (6)  | 0.002 (5)   | 0.000 (4)   | 0.002 (5)  |
| C29 | 0.018 (5)  | 0.025 (7)  | 0.020 (5)  | 0.005 (5)   | 0.000 (4)   | -0.004 (5) |
| C30 | 0.030 (6)  | 0.030 (7)  | 0.026 (5)  | -0.008 (6)  | 0.004 (5)   | -0.011 (5) |
| C31 | 0.020 (6)  | 0.038 (8)  | 0.028 (6)  | -0.007 (6)  | 0.003 (4)   | -0.001 (5) |
| C32 | 0.033 (7)  | 0.041 (8)  | 0.028 (6)  | 0.010 (6)   | 0.001 (5)   | -0.005 (5) |
| C33 | 0.050 (7)  | 0.028 (7)  | 0.033 (7)  | 0.007 (6)   | 0.004 (6)   | -0.008 (5) |
| C34 | 0.025 (6)  | 0.031 (7)  | 0.032 (6)  | -0.004 (6)  | 0.012 (5)   | -0.001 (5) |
| C35 | 0.072 (10) | 0.057 (11) | 0.056 (8)  | -0.007 (9)  | 0.038 (8)   | -0.005 (8) |
| O3  | 0.067 (6)  | 0.023 (5)  | 0.070 (6)  | -0.009 (5)  | 0.009 (5)   | 0.013 (4)  |
| C36 | 0.058 (11) | 0.065 (13) | 0.179 (18) | -0.023 (10) | -0.015 (12) | 0.025 (12) |
| O4  | 0.044 (5)  | 0.021 (5)  | 0.053 (5)  | 0.003 (4)   | -0.018 (4)  | -0.012 (4) |
| C24 | 0.060 (9)  | 0.047 (10) | 0.056 (9)  | 0.012 (8)   | -0.019 (7)  | -0.022 (7) |

*Geometric parameters (Å, °)*

|         |            |          |            |
|---------|------------|----------|------------|
| Br1—C33 | 1.883 (11) | C11—C12  | 1.384 (14) |
| Br2—C15 | 1.879 (11) | C11—C16  | 1.392 (13) |
| Fe1—N1  | 1.964 (8)  | C12—H12  | 0.9500     |
| Fe1—N3  | 1.916 (7)  | C12—C13  | 1.412 (14) |
| Fe1—N4  | 1.958 (8)  | C13—H13  | 0.9500     |
| Fe1—N7  | 1.964 (8)  | C13—C14  | 1.374 (14) |
| Fe1—N9  | 1.909 (6)  | C14—C15  | 1.375 (13) |
| Fe1—N10 | 1.982 (8)  | C15—C16  | 1.396 (13) |
| O1—C32  | 1.364 (12) | C16—H16  | 0.9500     |
| O1—C35  | 1.430 (13) | C17—H17A | 0.9800     |
| O2—C14  | 1.342 (11) | C17—H17B | 0.9800     |
| O2—C17  | 1.462 (14) | C17—H17C | 0.9800     |
| N1—N2   | 1.359 (10) | C18—H18  | 0.9500     |
| N1—C1   | 1.328 (12) | C18—C19  | 1.429 (14) |
| N2—C3   | 1.354 (11) | C19—H19  | 0.9500     |
| N2—C4   | 1.418 (12) | C19—C20  | 1.360 (14) |
| N3—C4   | 1.324 (11) | C20—H20  | 0.9500     |
| N3—C8   | 1.363 (11) | C21—C22  | 1.373 (13) |
| N4—N5   | 1.370 (11) | C22—H22  | 0.9500     |
| N4—C9   | 1.344 (12) | C22—C23  | 1.398 (13) |
| N5—C10  | 1.340 (12) | C23—H23  | 0.9500     |
| N6—C9   | 1.361 (11) | C23—C25  | 1.382 (13) |
| N6—C10  | 1.354 (12) | C25—H25  | 0.9500     |
| N7—N8   | 1.376 (10) | C25—C26  | 1.384 (13) |
| N7—C18  | 1.334 (12) | C26—C27  | 1.455 (13) |
| N8—C20  | 1.352 (11) | C28—C29  | 1.472 (13) |
| N8—C21  | 1.404 (11) | C29—C30  | 1.395 (14) |
| N9—C21  | 1.329 (11) | C29—C34  | 1.393 (13) |
| N9—C26  | 1.365 (11) | C30—H30  | 0.9500     |

|            |            |               |            |
|------------|------------|---------------|------------|
| N10—N11    | 1.364 (11) | C30—C31       | 1.399 (12) |
| N10—C27    | 1.331 (12) | C31—H31       | 0.9500     |
| N11—C28    | 1.341 (12) | C31—C32       | 1.367 (15) |
| N12—C27    | 1.341 (11) | C32—C33       | 1.393 (15) |
| N12—C28    | 1.359 (12) | C33—C34       | 1.385 (14) |
| C1—H1      | 0.9500     | C34—H34       | 0.9500     |
| C1—C2      | 1.426 (13) | C35—H35A      | 0.9800     |
| C2—H2      | 0.9500     | C35—H35B      | 0.9800     |
| C2—C3      | 1.350 (14) | C35—H35C      | 0.9800     |
| C3—H3      | 0.9500     | O3—H3A        | 0.8400     |
| C4—C5      | 1.369 (12) | O3—C36        | 1.387 (16) |
| C5—H5      | 0.9500     | C36—H36A      | 0.9800     |
| C5—C6      | 1.392 (13) | C36—H36B      | 0.9800     |
| C6—H6      | 0.9500     | C36—H36C      | 0.9800     |
| C6—C7      | 1.376 (13) | O4—H4         | 0.8400     |
| C7—H7      | 0.9500     | O4—C24        | 1.384 (12) |
| C7—C8      | 1.379 (13) | C24—H24A      | 0.9800     |
| C8—C9      | 1.440 (13) | C24—H24B      | 0.9800     |
| C10—C11    | 1.456 (13) | C24—H24C      | 0.9800     |
|            |            |               |            |
| N1—Fe1—N7  | 88.4 (3)   | O2—C14—C13    | 125.4 (10) |
| N1—Fe1—N10 | 93.7 (3)   | O2—C14—C15    | 116.5 (10) |
| N3—Fe1—N1  | 79.1 (3)   | C13—C14—C15   | 118.1 (10) |
| N3—Fe1—N4  | 80.0 (3)   | C14—C15—Br2   | 120.5 (7)  |
| N3—Fe1—N7  | 97.6 (3)   | C14—C15—C16   | 121.2 (10) |
| N3—Fe1—N10 | 102.9 (3)  | C16—C15—Br2   | 118.3 (8)  |
| N4—Fe1—N1  | 159.1 (4)  | C11—C16—C15   | 121.9 (9)  |
| N4—Fe1—N7  | 93.0 (3)   | C11—C16—H16   | 119.1      |
| N4—Fe1—N10 | 92.3 (3)   | C15—C16—H16   | 119.1      |
| N7—Fe1—N10 | 159.4 (3)  | O2—C17—H17A   | 109.5      |
| N9—Fe1—N1  | 98.3 (3)   | O2—C17—H17B   | 109.5      |
| N9—Fe1—N3  | 176.0 (4)  | O2—C17—H17C   | 109.5      |
| N9—Fe1—N4  | 102.5 (3)  | H17A—C17—H17B | 109.5      |
| N9—Fe1—N7  | 79.3 (3)   | H17A—C17—H17C | 109.5      |
| N9—Fe1—N10 | 80.2 (3)   | H17B—C17—H17C | 109.5      |
| C32—O1—C35 | 118.7 (10) | N7—C18—H18    | 125.0      |
| C14—O2—C17 | 117.1 (10) | N7—C18—C19    | 110.1 (10) |
| N2—N1—Fe1  | 113.6 (6)  | C19—C18—H18   | 125.0      |
| C1—N1—Fe1  | 140.1 (8)  | C18—C19—H19   | 127.3      |
| C1—N1—N2   | 105.3 (8)  | C20—C19—C18   | 105.4 (9)  |
| N1—N2—C4   | 116.2 (7)  | C20—C19—H19   | 127.3      |
| C3—N2—N1   | 111.9 (8)  | N8—C20—C19    | 107.8 (9)  |
| C3—N2—C4   | 131.9 (8)  | N8—C20—H20    | 126.1      |
| C4—N3—Fe1  | 120.6 (6)  | C19—C20—H20   | 126.1      |
| C4—N3—C8   | 119.6 (8)  | N9—C21—N8     | 109.7 (8)  |
| C8—N3—Fe1  | 119.6 (6)  | N9—C21—C22    | 123.9 (9)  |
| N5—N4—Fe1  | 138.5 (7)  | C22—C21—N8    | 126.3 (9)  |
| C9—N4—Fe1  | 114.6 (6)  | C21—C22—H22   | 121.6      |

|             |            |               |            |
|-------------|------------|---------------|------------|
| C9—N4—N5    | 106.8 (8)  | C21—C22—C23   | 116.7 (9)  |
| C10—N5—N4   | 105.0 (8)  | C23—C22—H22   | 121.6      |
| C10—N6—C9   | 101.7 (8)  | C22—C23—H23   | 120.0      |
| N8—N7—Fe1   | 113.0 (6)  | C25—C23—C22   | 120.1 (9)  |
| C18—N7—Fe1  | 141.1 (7)  | C25—C23—H23   | 120.0      |
| C18—N7—N8   | 105.5 (8)  | C23—C25—H25   | 120.1      |
| N7—N8—C21   | 116.6 (7)  | C23—C25—C26   | 119.8 (9)  |
| C20—N8—N7   | 111.1 (8)  | C26—C25—H25   | 120.1      |
| C20—N8—C21  | 132.2 (8)  | N9—C26—C25    | 119.7 (8)  |
| C21—N9—Fe1  | 121.0 (6)  | N9—C26—C27    | 109.8 (8)  |
| C21—N9—C26  | 119.5 (8)  | C25—C26—C27   | 130.5 (9)  |
| C26—N9—Fe1  | 119.4 (6)  | N10—C27—N12   | 113.6 (9)  |
| N11—N10—Fe1 | 138.0 (7)  | N10—C27—C26   | 116.2 (8)  |
| C27—N10—Fe1 | 114.5 (6)  | N12—C27—C26   | 130.3 (9)  |
| C27—N10—N11 | 107.5 (8)  | N11—C28—N12   | 115.1 (9)  |
| C28—N11—N10 | 103.3 (8)  | N11—C28—C29   | 121.5 (10) |
| C27—N12—C28 | 100.4 (8)  | N12—C28—C29   | 123.3 (9)  |
| N1—C1—H1    | 125.1      | C30—C29—C28   | 121.0 (10) |
| N1—C1—C2    | 109.9 (10) | C34—C29—C28   | 120.6 (9)  |
| C2—C1—H1    | 125.1      | C34—C29—C30   | 118.4 (9)  |
| C1—C2—H2    | 127.1      | C29—C30—H30   | 119.9      |
| C3—C2—C1    | 105.9 (9)  | C29—C30—C31   | 120.2 (11) |
| C3—C2—H2    | 127.1      | C31—C30—H30   | 119.9      |
| N2—C3—H3    | 126.5      | C30—C31—H31   | 119.5      |
| C2—C3—N2    | 106.9 (9)  | C32—C31—C30   | 120.9 (10) |
| C2—C3—H3    | 126.5      | C32—C31—H31   | 119.5      |
| N3—C4—N2    | 109.9 (8)  | O1—C32—C31    | 124.6 (10) |
| N3—C4—C5    | 123.8 (8)  | O1—C32—C33    | 116.2 (11) |
| C5—C4—N2    | 126.3 (8)  | C31—C32—C33   | 119.2 (10) |
| C4—C5—H5    | 121.7      | C32—C33—Br1   | 120.2 (8)  |
| C4—C5—C6    | 116.5 (9)  | C34—C33—Br1   | 119.3 (9)  |
| C6—C5—H5    | 121.7      | C34—C33—C32   | 120.5 (11) |
| C5—C6—H6    | 119.7      | C29—C34—H34   | 119.6      |
| C7—C6—C5    | 120.6 (9)  | C33—C34—C29   | 120.7 (10) |
| C7—C6—H6    | 119.7      | C33—C34—H34   | 119.6      |
| C6—C7—H7    | 120.3      | O1—C35—H35A   | 109.5      |
| C6—C7—C8    | 119.5 (9)  | O1—C35—H35B   | 109.5      |
| C8—C7—H7    | 120.3      | O1—C35—H35C   | 109.5      |
| N3—C8—C7    | 119.8 (9)  | H35A—C35—H35B | 109.5      |
| N3—C8—C9    | 109.1 (8)  | H35A—C35—H35C | 109.5      |
| C7—C8—C9    | 131.1 (9)  | H35B—C35—H35C | 109.5      |
| N4—C9—N6    | 112.4 (8)  | C36—O3—H3A    | 109.5      |
| N4—C9—C8    | 116.6 (8)  | O3—C36—H36A   | 109.5      |
| N6—C9—C8    | 130.8 (9)  | O3—C36—H36B   | 109.5      |
| N5—C10—N6   | 114.0 (9)  | O3—C36—H36C   | 109.5      |
| N5—C10—C11  | 120.6 (9)  | H36A—C36—H36B | 109.5      |
| N6—C10—C11  | 125.3 (9)  | H36A—C36—H36C | 109.5      |
| C12—C11—C10 | 122.3 (10) | H36B—C36—H36C | 109.5      |



|                 |            |                 |             |
|-----------------|------------|-----------------|-------------|
| C12—C11—C16     | 116.3 (9)  | C24—O4—H4       | 109.5       |
| C16—C11—C10     | 121.4 (9)  | O4—C24—H24A     | 109.5       |
| C11—C12—H12     | 119.1      | O4—C24—H24B     | 109.5       |
| C11—C12—C13     | 121.9 (10) | O4—C24—H24C     | 109.5       |
| C13—C12—H12     | 119.1      | H24A—C24—H24B   | 109.5       |
| C12—C13—H13     | 119.7      | H24A—C24—H24C   | 109.5       |
| C14—C13—C12     | 120.6 (10) | H24B—C24—H24C   | 109.5       |
| C14—C13—H13     | 119.7      |                 |             |
| Br1—C33—C34—C29 | -177.7 (8) | C3—N2—C4—N3     | 172.4 (9)   |
| Br2—C15—C16—C11 | -177.8 (7) | C3—N2—C4—C5     | -8.0 (16)   |
| Fe1—N1—N2—C3    | -169.9 (6) | C4—N2—C3—C2     | -178.2 (9)  |
| Fe1—N1—N2—C4    | 8.8 (10)   | C4—N3—C8—C7     | -4.9 (13)   |
| Fe1—N1—C1—C2    | 165.1 (8)  | C4—N3—C8—C9     | 175.6 (8)   |
| Fe1—N3—C4—N2    | 0.4 (10)   | C4—C5—C6—C7     | -2.0 (13)   |
| Fe1—N3—C4—C5    | -179.2 (7) | C5—C6—C7—C8     | 2.7 (14)    |
| Fe1—N3—C8—C7    | -179.9 (7) | C6—C7—C8—N3     | 0.7 (14)    |
| Fe1—N3—C8—C9    | 0.5 (10)   | C6—C7—C8—C9     | -179.8 (9)  |
| Fe1—N4—N5—C10   | -176.2 (7) | C7—C8—C9—N4     | 179.9 (9)   |
| Fe1—N4—C9—N6    | 177.2 (6)  | C7—C8—C9—N6     | 3.8 (17)    |
| Fe1—N4—C9—C8    | 0.5 (10)   | C8—N3—C4—N2     | -174.6 (7)  |
| Fe1—N7—N8—C20   | -170.3 (6) | C8—N3—C4—C5     | 5.7 (14)    |
| Fe1—N7—N8—C21   | 7.5 (9)    | C9—N4—N5—C10    | 0.7 (10)    |
| Fe1—N7—C18—C19  | 167.7 (8)  | C9—N6—C10—N5    | 0.4 (11)    |
| Fe1—N9—C21—N8   | -1.0 (10)  | C9—N6—C10—C11   | -177.5 (9)  |
| Fe1—N9—C21—C22  | -178.6 (7) | C10—N6—C9—N4    | 0.1 (10)    |
| Fe1—N9—C26—C25  | 178.7 (7)  | C10—N6—C9—C8    | 176.3 (10)  |
| Fe1—N9—C26—C27  | -0.7 (10)  | C10—C11—C12—C13 | 176.6 (10)  |
| Fe1—N10—N11—C28 | 180.0 (7)  | C10—C11—C16—C15 | -177.1 (9)  |
| Fe1—N10—C27—N12 | 179.3 (6)  | C11—C12—C13—C14 | 0.4 (19)    |
| Fe1—N10—C27—C26 | -0.1 (10)  | C12—C11—C16—C15 | 1.3 (15)    |
| O1—C32—C33—Br1  | -3.1 (13)  | C12—C13—C14—O2  | -176.3 (11) |
| O1—C32—C33—C34  | 179.6 (10) | C12—C13—C14—C15 | 1.6 (19)    |
| O2—C14—C15—Br2  | -5.6 (14)  | C13—C14—C15—Br2 | 176.3 (9)   |
| O2—C14—C15—C16  | 176.0 (10) | C13—C14—C15—C16 | -2.1 (17)   |
| N1—N2—C3—C2     | 0.3 (11)   | C14—C15—C16—C11 | 0.6 (16)    |
| N1—N2—C4—N3     | -6.1 (11)  | C16—C11—C12—C13 | -1.9 (16)   |
| N1—N2—C4—C5     | 173.6 (9)  | C17—O2—C14—C13  | 6.0 (19)    |
| N1—C1—C2—C3     | 2.4 (12)   | C17—O2—C14—C15  | -171.9 (11) |
| N2—N1—C1—C2     | -2.2 (11)  | C18—N7—N8—C20   | 3.1 (10)    |
| N2—C4—C5—C6     | 178.2 (8)  | C18—N7—N8—C21   | -179.0 (8)  |
| N3—C4—C5—C6     | -2.2 (14)  | C18—C19—C20—N8  | 0.6 (12)    |
| N3—C8—C9—N4     | -0.6 (11)  | C20—N8—C21—N9   | 172.9 (9)   |
| N3—C8—C9—N6     | -176.7 (8) | C20—N8—C21—C22  | -9.5 (16)   |
| N4—N5—C10—N6    | -0.7 (11)  | C21—N8—C20—C19  | -179.8 (9)  |
| N4—N5—C10—C11   | 177.2 (8)  | C21—N9—C26—C25  | -5.5 (12)   |
| N5—N4—C9—N6     | -0.5 (10)  | C21—N9—C26—C27  | 175.1 (8)   |
| N5—N4—C9—C8     | -177.3 (8) | C21—C22—C23—C25 | -1.0 (14)   |

|                 |            |                 |            |
|-----------------|------------|-----------------|------------|
| N5—C10—C11—C12  | -173.9 (9) | C22—C23—C25—C26 | 1.0 (14)   |
| N5—C10—C11—C16  | 4.5 (15)   | C23—C25—C26—N9  | 2.3 (13)   |
| N6—C10—C11—C12  | 3.9 (15)   | C23—C25—C26—C27 | -178.5 (9) |
| N6—C10—C11—C16  | -177.7 (9) | C25—C26—C27—N10 | -178.9 (9) |
| N7—N8—C20—C19   | -2.3 (11)  | C25—C26—C27—N12 | 1.9 (17)   |
| N7—N8—C21—N9    | -4.4 (11)  | C26—N9—C21—N8   | -176.6 (7) |
| N7—N8—C21—C22   | 173.2 (9)  | C26—N9—C21—C22  | 5.7 (14)   |
| N7—C18—C19—C20  | 1.4 (12)   | C27—N10—N11—C28 | 0.4 (10)   |
| N8—N7—C18—C19   | -2.7 (11)  | C27—N12—C28—N11 | -0.9 (11)  |
| N8—C21—C22—C23  | -179.6 (8) | C27—N12—C28—C29 | 179.4 (8)  |
| N9—C21—C22—C23  | -2.4 (14)  | C28—N12—C27—N10 | 1.1 (10)   |
| N9—C26—C27—N10  | 0.4 (11)   | C28—N12—C27—C26 | -179.6 (9) |
| N9—C26—C27—N12  | -178.8 (9) | C28—C29—C30—C31 | 179.4 (8)  |
| N10—N11—C28—N12 | 0.3 (11)   | C28—C29—C34—C33 | 179.8 (9)  |
| N10—N11—C28—C29 | -180.0 (8) | C29—C30—C31—C32 | 2.1 (15)   |
| N11—N10—C27—N12 | -1.0 (10)  | C30—C29—C34—C33 | 1.1 (15)   |
| N11—N10—C27—C26 | 179.6 (8)  | C30—C31—C32—O1  | 179.6 (9)  |
| N11—C28—C29—C30 | 162.3 (9)  | C30—C31—C32—C33 | -1.4 (15)  |
| N11—C28—C29—C34 | -16.3 (14) | C31—C32—C33—Br1 | 177.8 (8)  |
| N12—C28—C29—C30 | -18.0 (15) | C31—C32—C33—C34 | 0.6 (16)   |
| N12—C28—C29—C34 | 163.4 (9)  | C32—C33—C34—C29 | -0.4 (16)  |
| C1—N1—N2—C3     | 1.2 (10)   | C34—C29—C30—C31 | -1.9 (14)  |
| C1—N1—N2—C4     | 180.0 (8)  | C35—O1—C32—C31  | -3.0 (16)  |
| C1—C2—C3—N2     | -1.6 (11)  | C35—O1—C32—C33  | 178.0 (10) |

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

$Cg_1$  and  $Cg_2$  are the centroids of the C11–C16 and C29–C34 rings, respectively.

| $D-H\cdots A$                       | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| C17 $\cdots$ N6 <sup>i</sup>        |       |             | 3.201 (16)  |               |
| O3—H3A $\cdots$ N12                 | 0.84  | 2.02        | 2.820 (12)  | 160           |
| O4—H4 $\cdots$ N6                   | 0.84  | 2.06        | 2.855 (11)  | 158           |
| C1—H1 $\cdots$ O4 <sup>ii</sup>     | 0.95  | 2.22        | 3.128 (14)  | 161           |
| C18—H18 $\cdots$ O3 <sup>iii</sup>  | 0.95  | 2.27        | 3.192 (14)  | 163           |
| C35—H35A $\cdots$ C30 <sup>iv</sup> | 0.98  | 2.62        | 3.233 (16)  | 121           |
| C3—H3 $\cdots$ N5 <sup>iii</sup>    | 0.95  | 2.45        | 3.301 (13)  | 148           |
| C7—H7 $\cdots$ O4                   | 0.95  | 2.46        | 3.310 (11)  | 148           |
| C22—H22 $\cdots$ N11 <sup>ii</sup>  | 0.95  | 2.39        | 3.317 (13)  | 166           |
| C20—H20 $\cdots$ N11 <sup>ii</sup>  | 0.95  | 2.55        | 3.389 (13)  | 148           |
| C5—H5 $\cdots$ N5 <sup>iii</sup>    | 0.95  | 2.53        | 3.440 (12)  | 161           |
| C17—H17A $\cdots$ O4 <sup>i</sup>   | 0.98  | 2.52        | 3.451 (17)  | 159           |
| C34—H34 $\cdots$ C20 <sup>v</sup>   | 0.95  | 2.63        | 3.535 (15)  | 159           |
| C25—H25 $\cdots$ O3                 | 0.95  | 2.69        | 3.542 (13)  | 150           |
| C18—H18 $\cdots$ C36 <sup>iii</sup> | 0.95  | 2.88        | 3.65 (2)    | 138           |
| C2—H2 $\cdots$ C31 <sup>vi</sup>    | 0.95  | 2.84        | 3.639 (15)  | 143           |
| C2—H2 $\cdots$ C32 <sup>vi</sup>    | 0.95  | 2.89        | 3.656 (15)  | 139           |
| C2—H2 $\cdots$ C30 <sup>vi</sup>    | 0.95  | 2.86        | 3.734 (11)  | 154           |

|                             |      |      |            |     |
|-----------------------------|------|------|------------|-----|
| C2—H2...Cg2 <sup>vi</sup>   | 0.95 | 2.57 | 3.501 (11) | 168 |
| C19—H19...Cg1 <sup>vi</sup> | 0.95 | 2.74 | 3.681 (11) | 169 |

Symmetry codes: (i)  $-x+1, -y+1, z-1/2$ ; (ii)  $-x+3/2, y+1/2, z+1/2$ ; (iii)  $-x+3/2, y-1/2, z+1/2$ ; (iv)  $-x+2, -y+1, z-1/2$ ; (v)  $-x+3/2, y-1/2, z-1/2$ ; (vi)  $x, y, z+1$ .

*Geometry ( $\text{\AA}$ ,  $^\circ$ ) of hydrogen bonds and C...N interactions in the title compound.*

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | <i>D</i> ... <i>A</i> | H... <i>A</i> | <i>D</i> —H... <i>A</i> | Symmetry operation  |
|-------------------------|-------------|-----------------------|---------------|-------------------------|---------------------|
| C17...N6                | -           | 3.20 (2)              | -             | -                       | 1-x,1-y,-1/2+z      |
| O3-H...N12              | 0.84 (1)    | 2.82 (1)              | 2.02 (1)      | 159.5 (5)               | x,y,z               |
| O4-H...N6               | 0.84 (1)    | 2.86 (1)              | 2.06 (2)      | 158.0 (5)               | x,y,z               |
| C1-H...O4               | 0.95 (1)    | 3.13 (1)              | 2.22 (1)      | 160.6 (5)               | 1.5-x,1/2+y,1/2+z   |
| C18-H...O3              | 0.95 (1)    | 3.19 (1)              | 2.27 (1)      | 162.6 (5)               | 1.5-x,-1/2+y,1/2+z  |
| C35-H...C30             | 0.95 (1)    | 3.23 (2)              | 2.62 (2)      | 121.0 (5)               | 2-x,1-y,-1/2+z      |
| C3-H...N5               | 0.95 (1)    | 3.30 (1)              | 2.46 (2)      | 148.4 (5)               | 1.5-x,-1/2+y,1/2+z  |
| C7-H...O4               | 0.95 (1)    | 3.31 (1)              | 2.46 (1)      | 148.4 (5)               | x,y,z               |
| C22-H...N11             | 0.95 (1)    | 3.32 (1)              | 2.39 (1)      | 165.6 (5)               | 1.5-x,1/2+y,1/2+z   |
| C20-H...N11             | 0.95 (1)    | 3.39 (1)              | 2.55 (2)      | 147.6 (5)               | 1.5-x,1/2+y,1/2+z   |
| C5-H...N5               | 0.95 (1)    | 3.44 (1)              | 2.53 (2)      | 160.7 (5)               | 1.5-x,-1/2+y,1/2+z  |
| C17-H...O4              | 0.95 (1)    | 3.45 (2)              | 2.52 (2)      | 159.1 (5)               | 1-x,1-y,-1/2+z      |
| C34-H...C20             | 0.95 (1)    | 3.53 (2)              | 2.63 (1)      | 159.1 (5)               | 1.5-x,-1/2+y,-1/2+z |
| C25-H...O3              | 0.95 (1)    | 3.54 (1)              | 2.69 (1)      | 149.6 (5)               | x,y,z               |
| C18-H...C36             | 0.95 (1)    | 3.65 (2)              | 2.88 (1)      | 138.0 (5)               | 1.5-x,-1/2+y,1/2+z  |
| C2-H...C31              | 0.95 (1)    | 3.64 (2)              | 2.84 (1)      | 143.0 (5)               | x,y,1+z             |
| C2-H...C32              | 0.95 (1)    | 3.66 (2)              | 2.89 (2)      | 139.0 (5)               | x,y,1+z             |
| C2-H...C30              | 0.95 (1)    | 3.73 (1)              | 2.86 (1)      | 154.4 (5)               | x,y,1+z             |