Synthesis and crystal structure of hydrated \( \mu \)-oxalato-bis{bis[3-methyl-5-(pyridin-2-yl)-1H-1,2,4-triazole]iron(II)} bis(toluenesulfonate) 2.75-hydrate

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In the title compound \([\text{Fe}_2(\text{C}_2\text{O}_4)(\text{C}_8\text{H}_8\text{N}_4)_4](\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3)_2\cdot 2.75\text{H}_2\text{O}\), the two Fe\(^{II}\) ions have a highly distorted octahedral Fe\(^{II}\)N\(^4\)O\(^2\) environment formed by two bidentate triazole-based chelating ligands and a bis-bidentate oxalate bridging anion that connects the metal ions. Stabilization within the crystal structure is provided via a system of O—H· · ·O and N—H· · ·O hydrogen bonding, which determines the formation of a two-dimensional architecture along the \(a\)-axis direction.

1. Chemical context

The study of coordination compounds based on substituted 1,2,4-triazoles and 3\(d\) and 4\(d\) transition metals allows the design of supramolecular structures that can find applications in various fields such as molecular magnetism, catalysis, electrochemistry or cluster engineering (Zhang et al., 2017; Zakharchenko et al., 2019; Chen et al., 2015; Petrenko et al., 2020, 2021). The presence of the pyridine ring in such triazole systems leads to the formation of interesting isolated metal–organic frameworks that demonstrate promising magnetic properties, making them suitable for application as molecule-based magnets (Yao et al., 2015; Han et al., 2017; Li et al., 2015; Huang et al., 2015). Moreover, a combination of 3\(d^4\)–3\(d^7\) metals with N-donor bridging ligands may form coordination compounds with switchable spin states (Aromi et al., 2011; Kucheriv et al., 2021). This phenomenon is called spin crossover. Changes in the external temperature, pressure, magnetic field, light radiation or the presence of a guest alters the magnetic, electrical, mechanical and optical properties significantly in these compounds (Güttlich & Goodwin, 2004). Therefore, the synthesis and crystallographic characterization of these complexes are of current interest.

On the other hand, the ability of the oxalate anion to generate homobinuclear complexes is well known (Craig et al., 2010; Selmi et al., 2021; Karimpour et al., 2013; Paine et al., 2007). The coordination chemistry of oxalato-bridged binuclear Fe\(^{II}\) complexes with pyridyl-triazole chelating ligands is less studied. A few examples with a similar type of ligand indicate that complexes of this kind possess interesting magnetic and oxidizing properties (de Ruiter et al., 2008; Oliveira et al., 2018). In order to continue research in this field...
and in the course of our studies dedicated to the investigation of triazoles and, in particular, 3-methyl-5-(pyrid-2-yl)-2H-1,2,4-triazole (metrzpy) (Zakharchenko et al., 2017; Zakharchenko, Khomenko, Doroshchuk, Raspertova, Fesych et al., 2021; Zakharchenko, Khomenko, Doroshchuk, Raspertova, Shova et al., 2021), we report herein the synthesis and crystal structure of a new binuclear iron(II) complex with this ligand.

2. Structural commentary

The structure of the title compound is built up from dinuclear \([\text{Fe}_2(\text{metrzpy})_4(\text{C}_2\text{O}_4)]^{2+}\) complex cations, \(p\)-toluenesulfonate anions and co-crystallized water molecules in a 1:2:2.75 ratio. It crystallizes in the triclinic space group \(P\overline{1}\) with two complex molecules per unit cell. Each iron(II) ion has an \(\text{N}_4\text{O}_2\) coordination environment in a distorted octahedral geometry provided by two chelating metrzpy ligands in \(cis\) positions and a bidentate bridging oxalate anion (Fig. 1, Table 1). The reduced values of the angles subtended at the iron atom by the metrzpy and oxalate ligands are the main factors behind this distortion. The Fe—N and Fe—O bond lengths vary in the ranges 2.150 (3)—2.209 (3) Å and 2.123 (2)—2.171 (2) Å, respectively. The Fe1···Fe2 separation across the oxalate bridge of 5.576 (6) Å is in good agreement with previously reported values for other oxalate-bridged iron(II) complexes. The sets of coordinating atoms (O1/O2/N2/N6 for Fe1 and O3/O4/N10/N14 for Fe2) defining the mean equatorial planes are co-planar within 0.22 and 0.20 Å, while the displacement of the metal atom from these planes is 0.015 (1) and 0.037 (1) Å, respectively. The dihedral angle formed by each plane and the mean plane of the oxalate atoms is of 9.74 (6)° for Fe1 and 10.04 (7)° for Fe2.

3. Supramolecular features

All the species present in the structure are interconnected \(via\) a system of \(\text{O}—\text{H}···\text{O}\) and \(\text{N}—\text{H}···\text{O}\) hydrogen bonds (Table 2), which determines the formation of a two-dimensional architecture, as shown in Fig. 2. Further analysis has shown that the main crystal-structure motif consists of the parallel packing of 2D layers consolidated by the \(\pi—\pi\) stacking interactions observed between triazole and pyridine rings of

Table 1

|   | Selected bond lengths (Å) |
|---|---------------------------|
| Fe1—O1 | 2.171 (2) |
| Fe1—O2 | 2.123 (2) |
| Fe1—N1 | 2.203 (3) |
| Fe1—N2 | 2.150 (3) |
| Fe1—N5 | 2.197 (3) |
| Fe1—N6 | 2.162 (3) |
| Fe2—O3 | 2.123 (2) |
| Fe2—O4 | 2.157 (2) |
| Fe2—N9 | 2.209 (3) |
| Fe2—N10 | 2.165 (3) |
| Fe2—N13 | 2.206 (3) |
| Fe2—N14 | 2.159 (3) |

Figure 1

X-ray molecular structure of the title compound with selected atom labels and displacement ellipsoids drawn at the 50% level. Some H atoms are omitted for clarity. Key: carbon, grey; nitrogen, blue; oxygen, red; sulfur, yellow; iron, light green.

Figure 2

Two-dimensional supramolecular network viewed along the \(a\) axis.
The triazole ligand was prepared according to a synthesis described in the literature (Zakharchenko et al., 2017). Single crystals of \([\text{Fe}_2(\text{C}_2\text{O}_4)(\text{metrzpy})_4]([\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3])_2 \cdot 2.75\text{H}_2\text{O}\) were obtained by the liquid-to-liquid diffusion technique using a layering tube. The bottom was filled with \(\text{Fe}([\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3])_2 \cdot 6\text{H}_2\text{O}\) (50.6 mg, 0.1 mmol) in 2 ml of water. The middle was filled with a solution of 2 ml methanol/water (1:1) containing ascorbic acid (35.2 mg, 0.2 mmol). The top was filled with a solution of metrzpy ligand (32.0 mg, 0.2 mmol) in 2 ml of methanol. Afterwards, the tube was sealed with paraffin and light brown square-plate single crystals were formed within 3 days in relative high yield (ca 50%).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All hydrogen atoms were placed geometrically and refined as riding, with C—H = 0.96 (CH₃).
OH₂ molecule was fixed using an AFIX 3, Ueq(1) = 1.2Ueq(N). The idealized OH₂ molecule was refined with Ueq(H) = 1.2Ueq(N).

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Synthesis and crystal structure of hydrated $\mu$-oxalato-bis{bis[3-methyl-5-(pyridin-2-yl)-1H-1,2,4-triazole]iron(II)} bis(toluenesulfonate) 2.75-hydrate

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Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2021); cell refinement: CrysAlis PRO (Rigaku OD, 2021); data reduction: CrysAlis PRO (Rigaku OD, 2021); program(s) used to solve structure: SHELXT (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).

$\mu$-Oxalato-$\kappa^4$O$^1$,O$^2$:O$^\prime$:O$^\prime\prime$,O$^\prime\prime\prime$-bis{bis[3-methyl-5-(pyridin-2-yl)-1H-1,2,4-triazole-$\kappa^2$N$^4$,N$^5$]iron(II)} bis(toluenesulfonate) 2.75-hydrate

Crystal data

$\text{[Fe}_2(\text{C}_2\text{O}_4)(\text{C}_8\text{H}_8\text{N}_4)_2](\text{C}_7\text{H}_7\text{O}_3\text{S})_2\cdot 2.75\text{H}_2\text{O}$

$M_r = 1232.37$

Triclinic, $P\bar{1}$

$a = 9.9635$ (4) Å

$b = 14.4905$ (6) Å

$c = 20.1131$ (8) Å

$\alpha = 96.736$ (4)$^\circ$

$\beta = 101.490$ (4)$^\circ$

$\gamma = 95.216$ (4)$^\circ$

$V = 2806.5$ (2) Å$^3$

$Z = 2$

$F(000) = 1275$

$D_x = 1.458$ Mg m$^{-3}$

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

Cell parameters from 5996 reflections

$\theta = 2.0$–26.2$^\circ$

$\mu = 0.67$ mm$^{-1}$

$T = 293$ K

Block, clear light brown

$0.35 \times 0.2 \times 0.15$ mm

Data collection

Rigaku Oxford Diffraction Xcalibur, Eos diffractometer

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

Graphite monochromator

Detector resolution: 8.0797 pixels mm$^{-1}$

$\omega$ scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2021)

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.132$

$S = 1.06$

9886 reflections
739 parameters
0 restraints

Primary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained

$\Delta \sigma_{\text{max}} < 0.001$
$\Delta \rho_{\text{max}} = 0.59 \text{ e Å}^{-3}$
$\Delta \rho_{\text{min}} = -0.52 \text{ e Å}^{-3}$

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

|    | $x$     | $y$     | $z$     | $U_{eq}/U_{eq}$ | Occ. (<1) |
|----|---------|---------|---------|-----------------|----------|
| Fe1| 0.06627 (5) | 0.33384 (3) | 0.24663 (2) | 0.04095 (15) |
| Fe2| 0.46059 (5) | 0.63703 (3) | 0.25705 (2) | 0.04295 (15) |
| O1 | 0.1636 (2)  | 0.46283 (16) | 0.31108 (11) | 0.0477 (6)  |
| O2 | 0.2035 (3)  | 0.38999 (16) | 0.18929 (11) | 0.0485 (6)  |
| O3 | 0.3255 (3)  | 0.58058 (16) | 0.31534 (11) | 0.0505 (6)  |
| O4 | 0.3654 (3)  | 0.50805 (16) | 0.19378 (11) | 0.0487 (6)  |
| N1 | -0.1150 (3) | 0.38744 (19) | 0.18860 (13) | 0.0445 (7)  |
| N2 | -0.0855 (3) | 0.3172 (2)   | 0.30813 (13) | 0.0451 (7)  |
| N3 | -0.2364 (3) | 0.2948 (2)   | 0.36995 (14) | 0.0553 (8)  |
| H3 | -0.271736 | 0.279820 | 0.403417 | 0.066* |
| N4 | -0.3071 (3) | 0.3274 (2)   | 0.31482 (15) | 0.0540 (8)  |
| N5 | 0.2127 (3)  | 0.2521 (2)   | 0.30396 (13) | 0.0462 (7)  |
| N6 | 0.0155 (3)  | 0.19564 (19) | 0.18814 (13) | 0.0440 (7)  |
| N7 | -0.0330 (4) | 0.0583 (2)   | 0.13003 (17) | 0.0922 (14) |
| H7 | -0.071277 | 0.014005 | 0.097691 | 0.111* |
| N8 | 0.0632 (4)  | 0.1470 (2)   | 0.18557 (17) | 0.0848 (13) |
| N9 | 0.3081 (3)  | 0.7159 (2)   | 0.19942 (14) | 0.0507 (8)  |
| N10| 0.5106 (3)  | 0.77723 (19) | 0.31263 (13) | 0.0432 (7)  |
| N11| 0.5722 (3)  | 0.9187 (2)   | 0.36334 (15) | 0.0597 (9)  |
| N12| 0.615093 | 0.965062 | 0.392887 | 0.072* |
| N13| 0.4736 (4)  | 0.9271 (2)   | 0.30800 (16) | 0.0621 (9)  |
| N14| 0.6489 (3)  | 0.58921 (19) | 0.31364 (13) | 0.0455 (7)  |
| N15| 0.6070 (3)  | 0.65457 (19) | 0.19219 (13) | 0.0445 (7)  |
| N16| 0.7518 (3)  | 0.6810 (2)   | 0.12811 (14) | 0.0543 (8)  |
| C1 | 0.783958 | 0.695836 | 0.093650 | 0.065* |
| N17| 0.8289 (3)  | 0.6538 (2)   | 0.18426 (14) | 0.0529 (8)  |
| C1 | 0.2527 (4)  | 0.5063 (2)   | 0.28770 (16) | 0.0386 (8)  |
| C2 | 0.2758 (3)  | 0.4641 (2)   | 0.21719 (16) | 0.0374 (8)  |
| C3 | -0.1234 (4) | 0.4204 (3)   | 0.12807 (18) | 0.0589 (11) |
| H3A| -0.043788 | 0.429579 | 0.110984 | 0.071* |
| C4 | -0.2462 (5) | 0.4407 (3)   | 0.0907 (2)   | 0.0667 (12) |
| H4 | -0.249256 | 0.462694 | 0.048877 | 0.080* |
| C5 | -0.3627 (5) | 0.4285 (3)   | 0.1156 (2)   | 0.0702 (13) |
| H5 | -0.446072 | 0.441986 | 0.090806 | 0.084* |

$w = 1/[\sigma^2(F^2_0) + (0.0447P)^2 + 1.2466P]$ where $P = (F^2_0 + 2F^2_c)/3$
| Atom | x     | y     | z     | Ueq  | Notes |
|------|-------|-------|-------|------|-------|
| C6   | -0.3568 (4) | 0.3958 (3) | 0.17799 (19) | 0.0575 (10) |       |
| H6   | -0.435366 | 0.387506 | 0.196046 | 0.069* |       |
| C7   | -0.2312 (4) | 0.3760 (2) | 0.21249 (16) | 0.0427 (8) |       |
| C8   | -0.2112 (4) | 0.3405 (2) | 0.27900 (16) | 0.0421 (8) |       |
| C9   | -0.1053 (4) | 0.2887 (3) | 0.36643 (17) | 0.0503 (9) |       |
| C10  | -0.0023 (4) | 0.2548 (4) | 0.4192 (2) | 0.0807 (14) |       |
| H10A | 0.024759 | 0.197175 | 0.400311 | 0.121* |       |
| H10B | -0.042144 | 0.244484 | 0.457843 | 0.121* |       |
| H10C | 0.077127 | 0.300738 | 0.433744 | 0.121* |       |
| C11  | 0.3084 (4) | 0.2830 (3) | 0.36143 (18) | 0.0577 (10) |       |
| H11A | 0.326791 | 0.347058 | 0.375872 | 0.069* |       |
| C12  | 0.3799 (4) | 0.2239 (3) | 0.3996 (2) | 0.0659 (12) |       |
| H12  | 0.446270 | 0.247802 | 0.438745 | 0.079* |       |
| C13  | 0.3528 (4) | 0.1295 (3) | 0.3796 (2) | 0.0704 (13) |       |
| H13  | 0.398179 | 0.093636 | 0.405802 | 0.084* |       |
| C14  | 0.2570 (4) | 0.0956 (3) | 0.31978 (18) | 0.0625 (11) |       |
| H14  | 0.237582 | 0.031753 | 0.304686 | 0.075* |       |
| C15  | 0.1912 (4) | 0.1594 (3) | 0.28328 (17) | 0.0463 (9) |       |
| C16  | 0.0893 (4) | 0.1317 (3) | 0.21895 (17) | 0.0491 (9) |       |
| C17  | -0.0606 (4) | 0.1457 (3) | 0.13137 (19) | 0.0617 (11) |       |
| C18  | -0.1622 (5) | 0.1779 (3) | 0.0775 (2) | 0.0832 (15) |       |
| H18A | -0.235851 | 0.200596 | 0.096682 | 0.125* |       |
| H18B | -0.198935 | 0.126680 | 0.041786 | 0.125* |       |
| H18C | -0.117722 | 0.227346 | 0.058849 | 0.125* |       |
| C19  | 0.2082 (4) | 0.6836 (3) | 0.14435 (19) | 0.0642 (11) |       |
| H19  | 0.188657 | 0.619209 | 0.131749 | 0.077* |       |
| C20  | 0.1332 (4) | 0.7406 (4) | 0.1055 (2) | 0.0755 (14) |       |
| H20  | 0.063650 | 0.715303 | 0.067988 | 0.091* |       |
| C21  | 0.1627 (5) | 0.8348 (4) | 0.1230 (2) | 0.0803 (15) |       |
| H21  | 0.114664 | 0.874722 | 0.096693 | 0.096* |       |
| C22  | 0.2640 (5) | 0.8712 (3) | 0.17987 (19) | 0.0702 (13) |       |
| H22  | 0.284770 | 0.935471 | 0.192785 | 0.084* |       |
| C23  | 0.3334 (4) | 0.8092 (3) | 0.21697 (17) | 0.0501 (9) |       |
| C24  | 0.4396 (4) | 0.8401 (3) | 0.27894 (17) | 0.0485 (9) |       |
| C25  | 0.5944 (4) | 0.8301 (3) | 0.36629 (17) | 0.0487 (9) |       |
| C26  | 0.6950 (4) | 0.8004 (3) | 0.42185 (18) | 0.0677 (12) |       |
| H26A | 0.650605 | 0.751460 | 0.440960 | 0.102* |       |
| H26B | 0.729815 | 0.852738 | 0.456919 | 0.102* |       |
| H26C | 0.769006 | 0.777834 | 0.403759 | 0.102* |       |
| C27  | 0.6645 (4) | 0.5565 (3) | 0.37394 (18) | 0.0592 (11) |       |
| H27  | 0.586820 | 0.544259 | 0.392017 | 0.071* |       |
| C28  | 0.7898 (5) | 0.5401 (3) | 0.4102 (2) | 0.0684 (12) |       |
| H28  | 0.796418 | 0.518366 | 0.452334 | 0.082* |       |
| C29  | 0.9047 (5) | 0.5560 (3) | 0.3841 (2) | 0.0702 (12) |       |
| H29  | 0.990504 | 0.545042 | 0.407988 | 0.084* |       |
| C30  | 0.8916 (4) | 0.5886 (3) | 0.32143 (19) | 0.0594 (11) |       |
| H30  | 0.968095 | 0.599935 | 0.302321 | 0.071* |       |
| C31  | 0.7626 (4) | 0.6039 (2) | 0.28800 (16) | 0.0436 (8) |       |
| Atom  | U1     | U2     | U3     | U12    | U13    | U23    |
|-------|--------|--------|--------|--------|--------|--------|
| C32   | 0.7366 | 0.6382 | 0.2211 | 0.0429 |        |        |
| C33   | 0.6205 | 0.6820 | 0.1325 | 0.0515 |        |        |
| C34   | 0.5110 | 0.7077 | 0.0789 | 0.0773 |        |        |
| H34A  | 0.4863 | 0.7680 | 0.0939 | 0.116  |        |        |
| H34B  | 0.4315 | 0.6619 | 0.0707 | 0.116  |        |        |
| H34C  | 0.5441 | 0.7098 | 0.0374 | 0.116  |        |        |
| S1    | 0.1981 | 0.1515 | −0.0100| 0.0625 |        |        |
| O5    | 0.1921 | 0.2505 | −0.0094| 0.0669 |        |        |
| O6    | 0.2457 | 0.1123 | −0.0699| 0.1116 |        |        |
| O7    | 0.0732 | 0.1004 | −0.0048| 0.1409 |        |        |
| C35   | 0.3234 | 0.1378 | 0.0620 | 0.0470 |        |        |
| C36   | 0.3414 | 0.1958 | 0.1231 | 0.0534 |        |        |
| C37   | 0.4393 | 0.1816 | 0.1791 | 0.0566 |        |        |
| C38   | 0.4500 | 0.2210 | 0.2202 | 0.068  |        |        |
| C39   | 0.5008 | 0.0528 | 0.1146 | 0.0840 |        |        |
| C31   | 0.5542 | 0.0037 | 0.1110 | 0.101  |        |        |
| C40   | 0.4026 | 0.0656 | 0.0582 | 0.0755 |        |        |
| C41   | 0.3903 | 0.0249 | 0.0175 | 0.091  |        |        |
| C42   | 0.6316 | 0.0972 | 0.2360 | 0.0912 |        |        |
| C43   | 0.7188 | 0.1274 | 0.2318 | 0.137  |        |        |
| S2    | 0.6086 | 0.1239 | 0.2776 | 0.137  |        |        |
| O8    | 0.3029 | 0.8607 | 0.4975 | 0.0531 |        |        |
| O9    | 0.4317 | 0.8827 | 0.4778 | 0.0979 |        |        |
| O10   | 0.2802 | 0.9233 | 0.5546 | 0.0826 |        |        |
| C42   | 0.2837 | 0.7644 | 0.5100 | 0.0620 |        |        |
| C43   | 0.0828 | 0.9383 | 0.4305 | 0.0641 |        |        |
| H43   | 0.0921 | 0.9793 | 0.4709 | 0.077  |        |        |
| C44   | −0.0196| 0.9454 | 0.3740 | 0.0705 |        |        |
| H44   | −0.0786| 0.9911 | 0.3769 | 0.085  |        |        |
| C45   | −0.0353| 0.8857 | 0.3134 | 0.0570 |        |        |
| C46   | 0.0549 | 0.8200 | 0.3105 | 0.0561 |        |        |
| H46   | 0.0472 | 0.7798 | 0.2699 | 0.067  |        |        |
| C47   | 0.1565 | 0.8119 | 0.3661 | 0.0529 |        |        |
| H47   | 0.2162 | 0.7667 | 0.3628 | 0.063  |        |        |
| C48   | −0.1485| 0.8935 | 0.2527 | 0.0843 |        |        |
| H48A  | −0.1219| 0.8697 | 0.2111 | 0.126  |        |        |
| H48B  | −0.1632| 0.9579 | 0.2518 | 0.126  |        |        |
| H48C  | −0.2302| 0.8578 | 0.2563 | 0.126  |        |        |
| O1W   | −0.018 | 0.4596 | −0.0131| 0.159  |        |        |
| H1WA  | −0.0266| 0.5165 | −0.0007| 0.239  |        |        |
| H1WB  | 0.0650 | 0.4538 | 0.0039 | 0.239  |        |        |
| O2W   | 0.2693 | 0.4417 | 0.0460 | 0.1319 |        |        |
| H2WA  | 0.3023 | 0.4585 | 0.0884 | 0.198  |        |        |
| H2WB  | 0.2494 | 0.3826 | 0.0331 | 0.198  |        |        |
| Atomic displacement parameters ($\text{Å}^2$) |
|---|
| $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| **Fe1** | 0.0381 (3) | 0.0391 (3) | 0.0426 (3) | −0.0045 (2) | 0.0063 (2) | 0.0040 (2) |
| **Fe2** | 0.0400 (3) | 0.0413 (3) | 0.0438 (3) | −0.0073 (2) | 0.0079 (2) | 0.0018 (2) |
| **O1** | 0.0493 (16) | 0.0481 (15) | 0.0453 (13) | −0.0074 (12) | 0.0213 (12) | −0.0037 (11) |
| **O2** | 0.0551 (17) | 0.0440 (15) | 0.0427 (13) | −0.0099 (13) | 0.0163 (12) | −0.0072 (11) |
| **O3** | 0.0580 (17) | 0.0450 (15) | 0.0430 (13) | −0.0135 (13) | 0.0169 (12) | −0.0121 (12) |
| **O4** | 0.0513 (16) | 0.0495 (15) | 0.0445 (13) | −0.0114 (13) | 0.0222 (12) | −0.0044 (12) |
| **N1** | 0.052 (2) | 0.0367 (17) | 0.0414 (16) | 0.0027 (15) | 0.0042 (14) | 0.0044 (13) |
| **N2** | 0.0431 (19) | 0.0498 (19) | 0.0416 (15) | −0.0004 (15) | 0.0066 (13) | 0.0113 (14) |
| **N3** | 0.052 (2) | 0.070 (2) | 0.0449 (17) | −0.0014 (18) | 0.0152 (15) | 0.0103 (16) |
| **N4** | 0.050 (2) | 0.060 (2) | 0.0526 (18) | 0.0041 (17) | 0.0121 (16) | 0.0090 (16) |
| **N5** | 0.0345 (18) | 0.053 (2) | 0.0460 (16) | −0.0007 (15) | 0.0018 (13) | 0.0039 (15) |
| **N6** | 0.0462 (19) | 0.0392 (17) | 0.0406 (15) | −0.0001 (14) | −0.0013 (13) | 0.0033 (14) |
| **N7** | 0.129 (4) | 0.044 (2) | 0.071 (2) | 0.015 (2) | −0.045 (2) | −0.0138 (18) |
| **N8** | 0.115 (3) | 0.047 (2) | 0.070 (2) | 0.020 (2) | −0.031 (2) | −0.0039 (18) |
| **N9** | 0.0393 (19) | 0.058 (2) | 0.0488 (17) | −0.0012 (16) | 0.0014 (14) | 0.0024 (16) |
| **N10** | 0.0425 (18) | 0.0401 (17) | 0.0431 (16) | −0.0029 (14) | 0.0056 (14) | 0.0018 (14) |
| **N11** | 0.073 (3) | 0.046 (2) | 0.0486 (18) | −0.0023 (18) | −0.0015 (17) | −0.0093 (15) |
| **N12** | 0.073 (3) | 0.049 (2) | 0.0565 (19) | 0.0093 (19) | 0.0003 (18) | −0.0017 (17) |
| **N13** | 0.055 (2) | 0.0399 (17) | 0.0403 (16) | −0.0028 (15) | 0.0102 (14) | 0.0066 (14) |
| **N14** | 0.0420 (19) | 0.0462 (18) | 0.0436 (16) | −0.0027 (15) | 0.0077 (14) | 0.0076 (14) |
| **N15** | 0.053 (2) | 0.068 (2) | 0.0427 (17) | −0.0013 (18) | 0.0145 (15) | 0.0130 (16) |
| **N16** | 0.050 (2) | 0.057 (2) | 0.0520 (18) | 0.0007 (16) | 0.0143 (16) | 0.0084 (16) |
| **C1** | 0.038 (2) | 0.038 (2) | 0.0391 (18) | 0.0007 (17) | 0.0105 (16) | 0.0000 (16) |
| **C2** | 0.037 (2) | 0.036 (2) | 0.0373 (18) | 0.0023 (17) | 0.0074 (15) | −0.0002 (16) |
| **C3** | 0.074 (3) | 0.053 (3) | 0.049 (2) | 0.000 (2) | 0.010 (2) | 0.013 (2) |
| **C4** | 0.092 (4) | 0.048 (3) | 0.053 (2) | 0.008 (3) | −0.006 (2) | 0.014 (2) |
| **C5** | 0.072 (3) | 0.059 (3) | 0.069 (3) | 0.014 (3) | −0.012 (2) | 0.012 (2) |
| **C6** | 0.048 (3) | 0.053 (3) | 0.066 (2) | 0.009 (2) | −0.001 (2) | 0.004 (2) |
| **C7** | 0.046 (2) | 0.034 (2) | 0.0463 (19) | 0.0050 (17) | 0.0077 (17) | 0.0026 (16) |
| **C8** | 0.043 (2) | 0.036 (2) | 0.0445 (19) | 0.0007 (17) | 0.0064 (17) | 0.0014 (16) |
| **C9** | 0.048 (3) | 0.057 (2) | 0.043 (2) | −0.003 (2) | 0.0067 (18) | 0.0087 (18) |
| **C10** | 0.067 (3) | 0.119 (4) | 0.062 (3) | 0.008 (3) | 0.012 (2) | 0.038 (3) |
| **C11** | 0.044 (2) | 0.062 (3) | 0.057 (2) | 0.001 (2) | −0.0035 (19) | 0.000 (2) |
| **C12** | 0.046 (3) | 0.086 (3) | 0.056 (2) | 0.008 (2) | −0.0080 (19) | 0.001 (2) |
| **C13** | 0.066 (3) | 0.088 (4) | 0.057 (2) | 0.029 (3) | 0.000 (2) | 0.014 (3) |
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### Geometric parameters (Å, °)

| Bond                  | Distance  | Angle      | Value  |
|-----------------------|-----------|------------|--------|
| Fe1—O1                | 2.171 (2) | C18—H18C  | 0.9600 |
| Fe1—O2                | 2.123 (2) | C19—H19   | 0.9300 |
| Fe1—N1                | 2.203 (3) | C19—C20   | 1.372 (5) |
| Fe1—N2                | 2.150 (3) | C20—H20   | 0.9300 |
| Fe1—N5                | 2.197 (3) | C20—C21   | 1.359 (6) |
| Fe1—N6                | 2.162 (3) | C21—H21   | 0.9300 |
| Fe2—O3                | 2.123 (2) | C21—C22   | 1.381 (6) |
| Fe2—O4                | 2.157 (2) | C22—H22   | 0.9300 |
| Fe2—N9                | 2.209 (3) | C22—C23   | 1.381 (5) |
| Fe2—N10               | 2.165 (3) | C23—C24   | 1.460 (5) |
| Fe2—N13               | 2.206 (3) | C25—C26   | 1.478 (5) |
| Fe2—N14               | 2.159 (3) | C26—H26A  | 0.9600 |
| O1—C1                 | 1.241 (4) | C26—H26B  | 0.9600 |
| O2—C2                 | 1.243 (4) | C26—H26C  | 0.9600 |
| O3—C1                 | 1.245 (4) | C27—H27   | 0.9300 |
| O4—C2                 | 1.249 (4) | C27—C28   | 1.371 (5) |
| N1—C3                 | 1.349 (4) | C28—H28   | 0.9300 |
| N1—C7                 | 1.343 (4) | C28—C29   | 1.364 (6) |
| N2—C8                 | 1.363 (4) | C29—H29   | 0.9300 |
| N2—C9                 | 1.333 (4) | C29—C30   | 1.384 (5) |
| N3—H3                 | 0.8600    | C30—H30   | 0.9300 |
| N3—N4                 | 1.350 (4) | C30—C31   | 1.377 (5) |
| N3—C9                 | 1.332 (4) | C31—C32   | 1.471 (4) |
| N4—C8                 | 1.320 (4) | C33—C34   | 1.481 (5) |
| N5—C11                | 1.344 (4) | C34—H34A  | 0.9600 |
| N5—C15                | 1.344 (4) | C34—H34B  | 0.9600 |
| N6—C16                | 1.364 (4) | C34—H34C  | 0.9600 |
| N6—C17                | 1.323 (4) | S1—O5     | 1.439 (3) |
| N7—H7                 | 0.8600    | S1—O6     | 1.454 (3) |
| N7—N8                 | 1.356 (4) | S1—O7     | 1.419 (3) |
| N7—C17                | 1.318 (5) | S1—C35    | 1.758 (4) |
| N8—C16                | 1.305 (4) | C35—C36   | 1.375 (5) |
| N9—C19                | 1.338 (4) | C35—C40   | 1.371 (5) |
| N9—C23                | 1.344 (4) | C35—H36   | 0.9300 |
| N10—C24               | 1.365 (4) | C36—C37   | 1.383 (5) |
| N10—C25               | 1.334 (4) | C37—H37   | 0.9300 |
| N11—H11               | 0.8600    | C37—C38   | 1.368 (5) |
| N11—N12               | 1.355 (4) | C38—C39   | 1.368 (6) |
| N11—C25               | 1.329 (4) | C38—C41   | 1.520 (5) |
| N12—C24               | 1.309 (4) | C39—H39   | 0.9300 |
| N13—C27               | 1.339 (4) | C39—C40   | 1.384 (5) |
| N13—C31               | 1.345 (4) | C40—H40   | 0.9300 |
| N14—C32               | 1.361 (4) | C41—H41A  | 0.9600 |
| N14—C33               | 1.334 (4) | C41—H41B  | 0.9600 |
| N15—H15               | 0.8600    | C41—H41C  | 0.9600 |
| N15—N16               | 1.354 (4) | S2—O8     | 1.435 (3) |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|----------------------|--------------|
| N15—C33              | 1.331 (4)    | S2—O9                | 1.444 (3)    |
| N16—C32              | 1.312 (4)    | S2—O10               | 1.450 (3)    |
| C1—C2                | 1.546 (4)    | S2—C42               | 1.768 (4)    |
| C3—H3A               | 0.9300       | C42—C43              | 1.373 (5)    |
| C3—C4                | 1.379 (5)    | C42—C47              | 1.379 (5)    |
| C4—H4                | 0.9300       | C43—H43              | 0.9300       |
| C4—C5                | 1.357 (6)    | C43—C44              | 1.388 (5)    |
| C5—H5                | 0.9300       | C44—H44              | 0.9300       |
| C5—C6                | 1.385 (5)    | C44—C45              | 1.381 (5)    |
| C6—H6                | 0.9300       | C45—C46              | 1.372 (5)    |
| C6—C7                | 1.377 (5)    | C45—C48              | 1.512 (5)    |
| C7—C8                | 1.472 (4)    | C46—H46              | 0.9300       |
| C9—C10               | 1.484 (5)    | C46—C47              | 1.375 (5)    |
| C10—H10A             | 0.9600       | C47—H47              | 0.9300       |
| C10—H10B             | 0.9600       | C48—H48A             | 0.9600       |
| C10—H10C             | 0.9600       | C48—H48B             | 0.9600       |
| C11—H11A             | 0.9300       | C48—H48C             | 0.9600       |
| C11—C12              | 1.370 (5)    | O1W—H1WA             | 0.8500       |
| C12—H12              | 0.9300       | O1W—H1WB             | 0.8499       |
| C12—C13              | 1.366 (6)    | O2W—H2WA             | 0.8482       |
| C13—H13              | 0.9300       | O2W—H2WB             | 0.8577       |
| C13—C14              | 1.384 (5)    | O4W—H4WA             | 0.8679       |
| C14—H14              | 0.9300       | O4W—H4WB             | 0.8665       |
| C14—C15              | 1.378 (5)    | O5W—H5WA             | 0.8651       |
| C15—C16              | 1.465 (5)    | O5W—H5WB             | 0.8618       |
| C17—C18              | 1.483 (5)    | O3W—H3WA             | 0.8642       |
| C18—H18A             | 0.9600       | O3W—H3WB             | 0.8617       |
| C18—H18B             | 0.9600       |                        |              |
| O1—Fe1—N1            | 98.95 (10)   | N7—C17—C18           | 123.1 (4)    |
| O1—Fe1—N5            | 91.45 (10)   | C17—C18—H18A         | 109.5        |
| O2—Fe1—O1            | 76.88 (8)    | C17—C18—H18B         | 109.5        |
| O2—Fe1—N1            | 94.10 (10)   | C17—C18—H18C         | 109.5        |
| O2—Fe1—N2            | 163.77 (10)  | H18A—C18—H18B        | 109.5        |
| O2—Fe1—N5            | 96.26 (10)   | H18A—C18—H18C        | 109.5        |
| O2—Fe1—N6            | 97.91 (10)   | H18B—C18—H18C        | 109.5        |
| N2—Fe1—O1            | 91.45 (10)   | N9—C19—H19           | 118.3        |
| N2—Fe1—N1            | 76.39 (10)   | N9—C19—C20           | 123.4 (4)    |
| N2—Fe1—N5            | 95.26 (10)   | C20—C19—H19          | 118.3        |
| N2—Fe1—N6            | 95.76 (11)   | C19—C20—H20          | 120.7        |
| N5—Fe1—N1            | 166.75 (11)  | C21—C20—C19          | 118.6 (4)    |
| N6—Fe1—O1            | 167.01 (10)  | C21—C20—H20          | 120.7        |
| N6—Fe1—N1            | 93.25 (10)   | C20—C21—H21          | 120.0        |
| N6—Fe1—N5            | 77.17 (10)   | C20—C21—C22          | 119.9 (4)    |
| O3—Fe2—O4            | 77.01 (8)    | C22—C21—H21          | 120.0        |
| O3—Fe2—N9            | 95.46 (10)   | C21—C22—H22          | 121.0        |
| O3—Fe2—N10           | 99.08 (9)    | C21—C22—C23          | 118.0 (4)    |
| O3—Fe2—N13           | 96.09 (10)   | C23—C22—H22          | 121.0        |
O3—Fe2—N14 164.19 (10) N9—C23—C22 122.9 (4)
O4—Fe2—N9 90.81 (10) N9—C23—C24 114.7 (3)
O4—Fe2—N10 166.87 (10) C22—C23—C24 122.4 (4)
O4—Fe2—N13 100.13 (10) N10—C24—C23 120.9 (3)
O4—Fe2—N14 90.65 (9) N12—C24—N10 114.2 (3)
N10—Fe2—N9 76.99 (11) N12—C24—C23 124.9 (3)
N10—Fe2—N13 92.72 (10) N10—C25—C26 128.6 (3)
N13—Fe2—N9 165.61 (11) N11—C25—N10 108.2 (3)
N14—Fe2—N9 94.55 (10) N11—C25—C26 123.2 (3)
N14—Fe2—N10 95.07 (10) C25—C26—H26A 109.5
N14—Fe2—N13 76.15 (10) C25—C26—H26B 109.5
C1—O1—Fe1 113.9 (2) C25—C26—H26C 109.5
C2—O2—Fe1 115.3 (2) H26A—C26—H26B 109.5
C1—O3—Fe2 115.4 (2) H26A—C26—H26C 109.5
C2—O4—Fe2 114.1 (2) H26B—C26—H26C 109.5
C9—N1—Fe1 126.0 (3) N13—C27—H27 118.5
C7—N1—Fe1 115.9 (2) C27—C28—H28 120.3
C7—N1—C3 117.7 (3) C28—C27—H27 118.5
C8—N2—Fe1 113.6 (2) C29—C28—C27 119.5 (4)
C9—N2—Fe1 142.8 (2) C29—C28—H28 120.3
C9—N2—C8 103.6 (3) C29—C28—C27 119.5 (4)
N4—N3—H3 124.0 N13—C31—C30 123.1 (3)
C9—N3—H3 124.0 N13—C31—C32 123.1 (3)
C9—N3—N4 112.1 (3) C30—C31—C32 123.1 (3)
C8—N4—N3 101.7 (3) C31—C30—H30 120.8
C11—N5—Fe1 127.1 (3) C31—C30—C29 118.5 (4)
C15—N5—Fe1 115.0 (2) C31—C30—H30 120.8
C15—N5—C11 117.3 (3) N13—C31—C30 123.1 (3)
C16—N6—Fe1 111.9 (2) N13—C31—C32 113.9 (3)
C17—N6—Fe1 144.8 (2) C30—C31—C32 123.1 (3)
C17—N6—C16 103.3 (3) N14—C32—C31 119.8 (3)
N8—N7—H7 124.2 N16—C32—N14 114.6 (3)
C17—N7—H7 124.2 N16—C32—C31 125.6 (3)
C17—N7—N8 111.6 (3) N14—C33—C34 127.4 (4)
C16—N8—N7 101.8 (3) N15—C33—N14 108.0 (3)
C19—N9—Fe2 128.0 (3) N15—C33—C34 124.6 (3)
C19—N9—C23 117.1 (3) C33—C34—H34A 109.5
C23—N9—Fe2 114.2 (2) C33—C34—H34B 109.5
C24—N10—Fe2 111.8 (2) C33—C34—H34C 109.5
C25—N10—Fe2 144.1 (2) H34A—C34—H34B 109.5
C25—N10—C24 103.8 (3) H34A—C34—H34C 109.5
N12—N11—H11 124.2 H34B—C34—H34C 109.5
C25—N11—H11 124.2 O5—S1—O6 110.60 (19)
C25—N11—N12 111.6 (3) O5—S1—C35 106.93 (17)
C24—N12—N11 102.2 (3) O6—S1—C35 106.78 (19)
C27—N13—Fe2 126.7 (3) O7—S1—O5 114.3 (2)
C27—N13—C31 117.1 (3) O7—S1—O6 110.8 (3)
C31—N13—Fe2 115.8 (2) O7—S1—C35 106.96 (18)
| Bond                  | Distance (Å) | Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|----------------------|--------------|
| C32—N14—Fe2          | 113.6 (2)    | C36—C55—S1           | 121.8 (3)    | C32—N14—Fe2          | 113.6 (2)    |
| C33—N14—Fe2          | 142.5 (3)    | C40—C55—C36          | 119.4 (3)    | C33—N14—C32          | 103.7 (3)    |
| C33—N14—C32          | 103.7 (3)    | C35—C55—C36          | 118.8 (3)    | N16—N15—H15          | 124.0        |
| N16—N15—H15          | 124.0        | C35—C55—H36          | 120.0        | C33—N15—N16          | 112.0 (3)    |
| C33—N15—H15          | 124.0        | C37—C55—H36          | 120.0        | C32—N16—N15          | 101.7 (3)    |
| C32—N16—N15          | 101.7 (3)    | C36—C55—H37          | 119.3        | O1—C1—C2             | 116.8 (3)    |
| O1—C1—C2             | 116.8 (3)    | O3—C1—C2             | 116.6 (3)    | O2—C2—O4             | 126.3 (3)    |
| O2—C2—O4             | 126.3 (3)    | O2—C2—C1             | 117.0 (3)    | O4—C2—C1             | 116.7 (3)    |
| O4—C2—C1             | 116.7 (3)    | N1—C3—H3A            | 119.0        | N1—C3—H3A            | 119.0        |
| N1—C3—H3A            | 119.0        | C38—C39—C40           | 121.2 (4)    | N1—C3—C4             | 122.1 (4)    |
| C4—C3—H3A            | 119.0        | C38—C39—C40           | 121.2 (4)    | C4—C3—H3A            | 119.0        |
| C3—C4—H4             | 120.3        | C35—C40—C36          | 120.0        | C5—C4—H4             | 120.3        |
| C5—C4—H4             | 120.3        | C35—C40—C36          | 120.0        | C4—C5—H5             | 120.0        |
| C4—C5—H5             | 120.0        | C35—C40—C36          | 120.0        | C4—C5—C6             | 119.8 (4)    |
| C4—C5—C6             | 119.8 (4)    | C35—C40—C36          | 120.0        | C6—C5—H5             | 120.1        |
| C6—C5—H5             | 120.1        | C35—C40—C36          | 120.0        | C6—C5—H6             | 121.0        |
| C6—C5—H6             | 121.0        | C35—C40—C36          | 120.0        | C7—C6—C5             | 118.0 (4)    |
| C7—C6—C5             | 118.0 (4)    | C35—C40—C36          | 120.0        | O8—S2—O9             | 121.0        |
| N1—C3—H3A            | 119.0        | O8—S2—O9             | 121.0        | N1—C3—C4             | 122.1 (4)    |
| N1—C3—H3A            | 119.0        | O8—S2—O9             | 121.0        | N1—C3—C4             | 122.1 (4)    |
| N1—C7—C6             | 123.1 (3)    | C35—C40—C36          | 120.0        | C4—C5—H5             | 120.0        |
| N1—C7—C8             | 113.6 (3)    | C35—C40—C36          | 120.0        | N2—C8—C7             | 120.0 (3)    |
| C6—C7—C8             | 123.4 (3)    | C35—C40—C36          | 120.0        | N4—C8—N2             | 114.5 (3)    |
| N2—C8—C7             | 120.0 (3)    | C35—C40—C36          | 120.0        | N4—C8—C7             | 125.5 (3)    |
| N4—C8—C7             | 125.5 (3)    | C35—C40—C36          | 120.0        | N2—C9—C10            | 127.2 (3)    |
| N2—C9—C10            | 127.2 (3)    | C35—C40—C36          | 120.0        | N3—C9—N2             | 108.2 (3)    |
| N3—C9—N2             | 108.2 (3)    | C35—C40—C36          | 120.0        | C9—C10—H10A          | 109.5        |
| C9—C10—H10A          | 109.5        | C35—C40—C36          | 120.0        | C9—C10—H10B          | 109.5        |
| C9—C10—H10B          | 109.5        | C35—C40—C36          | 120.0        | H10A—C10—H10B        | 109.5        |
| H10A—C10—H10B        | 109.5        | C35—C40—C36          | 120.0        | H10A—C10—H10C        | 109.5        |
| H10B—C10—H10C        | 109.5        | C35—C40—C36          | 120.0        | C9—C10—H10C          | 109.5        |
| N5—C11—H11A          | 118.7        | C35—C40—C36          | 120.0        | C12—C11—H11A         | 118.7        |
| N5—C11—C12           | 122.7 (4)    | C35—C40—C36          | 120.0        | C11—C12—H12          | 120.3        |
| C12—C11—H11A         | 118.7        | C35—C40—C36          | 120.0        | C13—C12—C11          | 119.4 (4)    |
| C11—C12—H12          | 120.3        | C35—C40—C36          | 120.0        | C13—C12—H12          | 120.3        |
| C13—C12—C11          | 119.4 (4)    | C35—C40—C36          | 120.0        | C12—C13—H13          | 120.4        |
| C12—C13—C14          | 119.2 (4)    | C35—C40—C36          | 120.0        | C12—C13—C14          | 119.2 (4)    |
C14—C13—H13 120.4  C45—C48—H48A 109.5  
C13—C14—H14 120.9  C45—C48—H48B 109.5  
C15—C14—C13 118.1 (4)  C45—C48—H48C 109.5  
C15—C14—H14 120.9  H48A—C48—H48B 109.5  
N5—C15—C14 123.2 (3)  H48A—C48—H48C 109.5  
N5—C15—C16 114.0 (3)  C45—C48—H48D 109.5  
C14—C15—C16 122.8 (3)  H2WA—O2W—H2WB 116.3  
N6—C16—C15 121.1 (3)  H4WA—O4W—H4WB 117.9  
N8—C16—N6 114.5 (3)  H4WA—O5W—H5WB 112.9  
N8—C16—C15 124.4 (3)  H3WA—O3W—H3WB 107.7  
N6—C17—C18 128.1 (4)  
N7—C17—N6 108.8 (3)  

Fe1—O1—C1—O3 −178.1 (3)  C8—N2—C9—C10 −180.0 (4)  
Fe1—O1—C1—C2 1.9 (4)  C9—N2—C8—N4 −0.7 (4)  
Fe1—O2—C2—O4 178.1 (3)  C9—N2—C8—C7 179.9 (3)  
Fe1—O2—C2—C1 −2.0 (4)  C9—N3—N4—C8 −0.3 (4)  
Fe1—N3—C3—C4 171.0 (3)  C11—N5—C15—C14 3.3 (5)  
Fe1—N1—C7—C6 −172.4 (3)  C11—N5—C15—C16 −177.8 (3)  
Fe1—N1—C7—C8 7.7 (4)  C11—C12—C13—C14 2.2 (6)  
Fe1—N2—C8—N4 177.6 (2)  C12—C13—C14—C15 −0.9 (6)  
Fe1—N2—C8—C7 −1.8 (4)  C13—C14—C15—N5 −2.0 (6)  
Fe1—N2—C9—N3 −176.9 (3)  C13—C14—C15—C16 179.2 (4)  
Fe1—N2—C9—C10 2.6 (7)  C14—C15—C16—N6 172.9 (3)  
Fe1—N5—C11—C12 169.2 (3)  C14—C15—C16—N8 −8.9 (6)  
Fe1—N5—C15—C14 −168.8 (3)  C15—N5—C11—C12 −1.9 (5)  
Fe1—N5—C15—C16 10.1 (4)  C16—N6—C17—N7 0.1 (5)  
Fe1—N6—C16—N8 −179.6 (3)  C16—N6—C17—C18 −180.0 (4)  
Fe1—N6—C16—C15 −1.2 (4)  C17—N6—C16—N8 −0.1 (5)  
Fe1—N6—C17—N7 179.3 (3)  C17—N6—C16—C15 178.3 (4)  
Fe1—N6—C17—C18 −0.8 (8)  C17—N7—N8—C16 −0.1 (5)  
Fe2—O3—C1—O1 −177.3 (3)  C19—N9—C23—C22 −1.8 (5)  
Fe2—O3—C1—C2 2.7 (4)  C19—N9—C23—C24 177.7 (3)  
Fe2—O4—C2—O2 177.4 (3)  C19—C20—C21—C22 −1.6 (7)  
Fe2—O4—C2—C1 −2.5 (3)  C20—C21—C22—C23 0.6 (7)  
Fe2—N9—C19—C20 −168.9 (3)  C21—C22—C23—N9 1.1 (6)  
Fe2—N9—C23—C22 169.3 (3)  C21—C22—C23—C24 −178.3 (4)  
Fe2—N9—C23—C24 −11.2 (4)  C22—C23—C24—N10 −177.0 (3)  
Fe2—N10—C24—N12 −175.3 (3)  C22—C23—C24—N10 4.6 (6)  
Fe2—N10—C24—C23 6.1 (4)  C23—N9—C19—C20 0.7 (6)  
Fe2—N10—C25—N11 172.5 (3)  C24—N10—C25—N11 0.0 (4)  
Fe2—N10—C25—C26 −8.4 (7)  C24—N10—C25—C26 179.1 (4)  
Fe2—N13—C27—C28 −170.8 (3)  C25—N10—C24—N12 −0.1 (4)  
Fe2—N13—C31—C30 172.0 (3)  C25—N10—C24—C23 −178.6 (3)  
Fe2—N13—C31—C32 −7.7 (4)  C25—N11—N12—C24 −0.1 (4)  
Fe2—N14—C32—N16 −176.2 (2)  C27—N13—C31—C30 −1.4 (5)  
Fe2—N14—C32—C31 5.1 (4)  C27—N13—C31—C32 179.0 (3)  
Fe2—N14—C33—N15 174.8 (3)  C27—C28—C29—C30 0.2 (7)
Fe2—N14—C33—C34  
O1—C1—C2—O2  
O1—C1—C2—O4  
O3—C1—C2—O2  
O3—C1—C2—O4  
N1—C3—C4—C5  
N1—C7—C8—N2  
N1—C7—C8—N4  
N3—N4—C8—N2  
N3—N4—C8—C7  
N7—N8—C16—N6  
N7—N8—C16—C15  
N8—N7—C17—C18  
N9—C19—C20—C21  
N9—C23—C24—N10  
N9—C23—C24—N12  
N11—N12—C24—C23  
N12—N11—C25—N10  
N12—N11—C25—C26  
N13—C27—C28—C29  
N13—C31—C32—N14  
N13—C31—C32—N16  
N15—N16—C32—N14  
N15—N16—C32—C31  
N16—N15—C33—C34  
C3—N1—C7—C6  
C3—N1—C7—C8  
C3—C4—C5—C6  
C4—C5—C6—C7  
C5—C6—C7—N1  
C5—C6—C7—C8  
C6—C7—C8—N2  
C6—C7—C8—N4  
C7—N1—C3—C4  
C8—N2—C9—N3  

Hydrogen-bond geometry (Å, º)

| D—H···A | D—H | H···A | D···A | D—H···A |
|----------|------|-------|-------|---------|
| N3—H3···O10i | 0.86 | 1.95 | 2.766 (4) | 159 |

sup-12
| Bond                  | d (Å) | r (Å) | D (Å)   | ϕ (°) |
|-----------------------|-------|-------|---------|-------|
| N7—H7···O6<sup>i</sup> | 0.86  | 2.34  | 3.064 (5) | 142   |
| N7—H7···O7<sup>ii</sup> | 0.86  | 2.34  | 3.141 (6) | 154   |
| N11—H11···O9<sup>III</sup> | 0.86  | 1.92  | 2.769 (4) | 170   |
| N15—H15···O5<sup>IV</sup> | 0.86  | 1.99  | 2.825 (4) | 163   |
| C4—H4···O2<sup>W</sup> | 0.93  | 2.48  | 3.383 (5) | 165   |
| C11—H11···O5<sup>W</sup> | 0.93  | 2.49  | 3.206 (8) | 134   |
| C28—H28···O4<sup>W</sup> | 0.93  | 2.54  | 3.421 (6) | 159   |
| O2<sup>W</sup>—H2<sup>WA</sup>···O4 | 0.85  | 1.99  | 2.838 (4) | 172   |
| O2<sup>W</sup>—H2<sup>WB</sup>···O5 | 0.86  | 1.99  | 2.838 (4) | 172   |
| O4<sup>W</sup>—H4<sup>WA</sup>···O1 | 0.87  | 2.34  | 3.123 (5) | 150   |
| O4<sup>W</sup>—H4<sup>WB</sup>···O10 | 0.87  | 1.92  | 2.788 (5) | 174   |
| O5<sup>W</sup>—H5<sup>WA</sup>···O4<sup>W</sup> | 0.86  | 1.98  | 2.810 (11) | 159 |
| O5<sup>W</sup>—H5<sup>WB</sup>···O4<sup>W</sup> | 0.86  | 2.28  | 2.850 (10) | 123 |
| C13—H13···O8<sup>W</sup> | 0.93  | 2.57  | 3.256 (5) | 131   |
| C21—H21···O7<sup>W</sup> | 0.93  | 2.44  | 3.280 (6) | 150   |

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