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Bis[*N*-(2-pyridylcarbonyl)pyridine-2-carboximidato]iron(III) perchlorate methanol solvate

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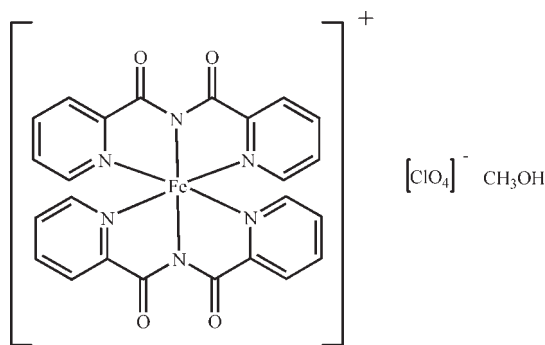
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Key indicators: single-crystal X-ray study; $T = 143$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 11.7.

In the title complex, $[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_3\text{O}_2)_2]\text{ClO}_4 \cdot \text{CH}_3\text{OH}$, the iron(III) ion is surrounded by two tridentate *N*-(2-pyridylcarbonyl)pyridine-2-carboximidate (bpca) ligands and exhibits a distorted octahedral coordination by six bpca N atoms. A classical $\text{O}-\text{H} \cdots \text{O}$ hydrogen bond exists between the methanol solvent molecule and the perchlorate anion. Magnetic susceptibility measurements indicated the complex to be in the low-spin state in the temperature range 5–400 K.

Related literature

For the structure and magnetic properties of methanol-free $[\text{Fe}(\text{bpca})_2]\text{ClO}_4$ and related compounds, see: Wocadlo *et al.* (1993).



Experimental

Crystal data

 $[\text{Fe}(\text{C}_{12}\text{H}_8\text{N}_3\text{O}_2)_2]\text{ClO}_4 \cdot \text{CH}_4\text{O}$ $M_r = 639.77$

Triclinic, $P\bar{1}$
 $a = 8.799$ (3) Å
 $b = 11.603$ (4) Å
 $c = 14.356$ (6) Å
 $\alpha = 109.507$ (4)°
 $\beta = 103.394$ (3)°
 $\gamma = 100.091$ (3)°

$V = 1292.0$ (8) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.76$ mm⁻¹
 $T = 143$ K
 $0.32 \times 0.26 \times 0.23$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (*SHELXTL*; Sheldrick, 2008)
 $T_{\min} = 0.790$, $T_{\max} = 0.840$

8499 measured reflections
 4421 independent reflections
 4177 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.107$
 $S = 1.03$
 4421 reflections

379 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|--------|-----------|--------|-----------|
| Fe1—N2 | 1.900 (2) | Fe1—N4 | 1.976 (2) |
| Fe1—N5 | 1.922 (2) | Fe1—N1 | 1.977 (2) |
| Fe1—N6 | 1.974 (2) | Fe1—N3 | 1.977 (2) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$ | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|--|--------------|---------------------|--------------|-----------------------|
| $\text{O1W}-\text{H1W} \cdots \text{O13}^{\text{i}}$ | 1.03 | 1.92 | 2.916 (3) | 160 |

Symmetry code: (i) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2207).

References

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supplementary materials

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Bis[*N*-(2-pyridylcarbonyl)pyridine-2-carboximidato]iron(III) perchlorate methanol solvate

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Comment

Our recent work indicated the N-donor tridentate ligand is suitable for the synthesis of spin-crossover materials. The *N*-2-pyridinylcarbonyl-2-pyridinecarboximidate (bpca) ligand has a typical rigid tridentate donor and was well studied to construct transition metal complexes including Fe(II), Fe(III), Co(II), Ni(II) and Cu(II) (Wocadlo *et al.* 1993 and references cited therein). One of the examples is reported by Wocadlo and coworkers, which interestingly showed the spin state can be tuned by the different counterion and solvent. It was claimed that Fe(III) complex [Fe(bpca)Cl₂(H₂O)] (CH₃)₂CO and [Fe(bpca)₂](NO₃) 1.67 H₂O adopt high spin state and the low-spin one in all the range of measured temperatures, respectively, while the [Fe(bpca)₂](ClO₄) evidence the spin-crossover behaviour. Here, we reported the crystal structure of complex [Fe(bpca)₆](ClO₄) CH₃OH. (Fig. 1). The coordination environments of Fe(III) ions are completed by two bpca ligands with average Fe—N bond length of being 1.954 Å (Table 1). A classical hydrogen bond O—H...O exists between methanol and chlorate anion with D...A distance being 2.916 (3) Å (Table 2). The temperature-dependent magnetic susceptibility was measured down to 5 K. The data in the form of molar magnetic moment multiply temperature is nearly constant and equal to about 0.45 emu K mol⁻¹, consistent with low spin state of Fe(III) (*s* = 1/2).

Experimental

A methanolic solution (25 ml) containing the bpca ligand (0.2 mmol, 0.046 g) was added dropwise to Fe(ClO₄)₂·6 H₂O (0.1 mmol, 0.036 g). After stirring for 15 minutes, the dark solution was filtered. Red block-shaped crystals suitable for single-crystal X-ray diffraction were obtained by evaporating the resulting filtration in air for several days (yield: 56.2%). Anal calc (%). for C₂₅ H₂₀ Cl Fe N₆ O₉: H 3.15 C 46.95 N 13.15. Found: H 3.12, C 46.87, N 13.54.

Refinement

C-bound H atoms were placed geometrically and allowed to ride during refinement with C—H = 0.93–0.96 Å with *U*_{iso}(H) = 1.2 *U*_{eq}(C). The hydroxy H atom of the methanol solvent molecule was located in a difference Fourier map and refined as riding with the parent atom with *U*_{iso}(H) = 1.5 *U*_{eq}(O).

Figures

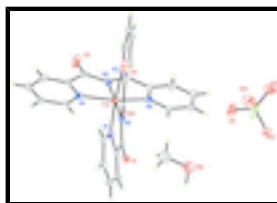


Fig. 1. The molecular structure of the title compound, the thermal ellipsoids were drawn at 50% probability level.

Bis[*N*-(2-pyridylcarbonyl)pyridine-2-carboximidato]iron(III) perchlorate methanol solvate

Crystal data

| | |
|--|---|
| [Fe(C ₁₂ H ₈ N ₃ O ₂) ₂]ClO ₄ ·CH ₄ O | <i>Z</i> = 2 |
| <i>M_r</i> = 639.77 | <i>F</i> ₀₀₀ = 654 |
| Triclinic, <i>P</i> $\bar{1}$ | <i>D_x</i> = 1.645 Mg m ⁻³ |
| Hall symbol: -P 1 | Mo <i>K</i> α radiation, λ = 0.71070 Å |
| <i>a</i> = 8.799 (3) Å | Cell parameters from 5465 reflections |
| <i>b</i> = 11.603 (4) Å | θ = 3.0–27.8° |
| <i>c</i> = 14.356 (6) Å | μ = 0.76 mm ⁻¹ |
| α = 109.507 (4)° | <i>T</i> = 143 K |
| β = 103.394 (3)° | Block, red |
| γ = 100.091 (3)° | 0.32 × 0.26 × 0.23 mm |
| <i>V</i> = 1292.0 (8) Å ³ | |

Data collection

| | |
|--|---|
| Bruker SMART APEX CCD area-detector diffractometer | 4421 independent reflections |
| Radiation source: fine-focus sealed tube | 4177 reflections with <i>I</i> > 2σ(<i>I</i>) |
| Monochromator: graphite | <i>R</i> _{int} = 0.040 |
| <i>T</i> = 143 K | θ _{max} = 25.0° |
| φ and ω scans | θ _{min} = 3.2° |
| Absorption correction: multi-scan (SHELXTL; Sheldrick, 2008) | <i>h</i> = -8→10 |
| <i>T</i> _{min} = 0.790, <i>T</i> _{max} = 0.840 | <i>k</i> = -13→13 |
| 8499 measured reflections | <i>l</i> = -17→16 |

Refinement

| | |
|--|--|
| Refinement on <i>F</i> ² | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | H-atom parameters constrained |
| $wR(F^2) = 0.107$ | $w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 1.8601P]$ |
| <i>S</i> = 1.03 | where $P = (F_o^2 + 2F_c^2)/3$ |
| 4421 reflections | (Δ/σ) _{max} < 0.001 |
| 379 parameters | Δρ _{max} = 1.48 e Å ⁻³ |
| Primary atom site location: structure-invariant direct methods | Δρ _{min} = -0.48 e Å ⁻³ |
| | Extinction correction: none |

Special details

Experimental. The magnetic measurements were performed on Quantum Design SQUID, MPMS-5S magnetometer.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|--------------|---------------|----------------------------------|
| Fe1 | 0.58016 (4) | 0.81056 (3) | 0.23045 (2) | 0.01007 (12) |
| O1 | 0.2790 (2) | 0.51858 (16) | -0.02782 (13) | 0.0148 (4) |
| N2 | 0.5013 (2) | 0.68770 (19) | 0.09138 (15) | 0.0112 (4) |
| O2 | 0.5569 (2) | 0.63892 (17) | -0.06690 (13) | 0.0168 (4) |
| N1 | 0.3602 (2) | 0.73605 (19) | 0.23053 (15) | 0.0121 (4) |
| N6 | 0.6689 (2) | 0.70815 (19) | 0.30342 (16) | 0.0129 (4) |
| N3 | 0.7802 (2) | 0.84703 (19) | 0.19255 (16) | 0.0127 (4) |
| N5 | 0.6579 (2) | 0.93790 (19) | 0.36992 (15) | 0.0136 (4) |
| N4 | 0.5149 (2) | 0.95113 (19) | 0.19857 (15) | 0.0124 (4) |
| C5 | 0.2725 (3) | 0.6311 (2) | 0.14314 (18) | 0.0118 (5) |
| O4 | 0.7774 (3) | 0.97129 (19) | 0.54311 (14) | 0.0338 (5) |
| O3 | 0.6870 (3) | 1.15149 (18) | 0.46500 (15) | 0.0326 (5) |
| C7 | 0.5952 (3) | 0.6937 (2) | 0.02702 (18) | 0.0121 (5) |
| C6 | 0.3479 (3) | 0.6029 (2) | 0.05672 (18) | 0.0119 (5) |
| C9 | 0.8828 (3) | 0.7988 (2) | 0.0474 (2) | 0.0175 (5) |
| H9A | 0.8652 | 0.7542 | -0.0230 | 0.021* |
| C4 | 0.1249 (3) | 0.5577 (2) | 0.13335 (19) | 0.0165 (5) |
| H4A | 0.0705 | 0.4844 | 0.0739 | 0.020* |
| C15 | 0.4363 (3) | 1.1664 (3) | 0.1802 (2) | 0.0202 (6) |
| H15A | 0.4089 | 1.2379 | 0.1738 | 0.024* |
| C13 | 0.4368 (3) | 0.9487 (2) | 0.10510 (19) | 0.0141 (5) |
| H13A | 0.4096 | 0.8740 | 0.0464 | 0.017* |
| C8 | 0.7602 (3) | 0.7837 (2) | 0.09035 (19) | 0.0133 (5) |
| C11 | 1.0553 (3) | 0.9449 (3) | 0.2156 (2) | 0.0211 (6) |
| H11A | 1.1562 | 0.9995 | 0.2597 | 0.025* |
| C10 | 1.0327 (3) | 0.8812 (3) | 0.1110 (2) | 0.0226 (6) |
| H10A | 1.1174 | 0.8938 | 0.0838 | 0.027* |
| C24 | 0.6699 (3) | 0.5865 (2) | 0.2595 (2) | 0.0145 (5) |
| H24A | 0.6253 | 0.5434 | 0.1878 | 0.017* |
| C14 | 0.3960 (3) | 1.0545 (3) | 0.0939 (2) | 0.0178 (5) |
| H14A | 0.3417 | 1.0501 | 0.0285 | 0.021* |
| C22 | 0.8026 (4) | 0.5867 (3) | 0.4249 (2) | 0.0273 (6) |
| H22A | 0.8482 | 0.5458 | 0.4653 | 0.033* |
| C17 | 0.5555 (3) | 1.0616 (2) | 0.28249 (19) | 0.0160 (5) |
| C18 | 0.6414 (3) | 1.0583 (2) | 0.3844 (2) | 0.0182 (5) |

supplementary materials

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|------|-------------|--------------|--------------|--------------|
| C3 | 0.0577 (3) | 0.5945 (3) | 0.2137 (2) | 0.0189 (5) |
| H3A | -0.0422 | 0.5463 | 0.2089 | 0.023* |
| C20 | 0.7334 (3) | 0.7695 (2) | 0.40816 (19) | 0.0177 (5) |
| C21 | 0.8008 (4) | 0.7121 (3) | 0.4710 (2) | 0.0257 (6) |
| H21A | 0.8441 | 0.7564 | 0.5427 | 0.031* |
| C1 | 0.2943 (3) | 0.7719 (2) | 0.30753 (19) | 0.0157 (5) |
| H1A | 0.3519 | 0.8443 | 0.3672 | 0.019* |
| C12 | 0.9269 (3) | 0.9267 (2) | 0.25399 (19) | 0.0156 (5) |
| H12A | 0.9423 | 0.9708 | 0.3242 | 0.019* |
| C23 | 0.7360 (3) | 0.5236 (3) | 0.3187 (2) | 0.0205 (6) |
| H23A | 0.7352 | 0.4393 | 0.2868 | 0.025* |
| C19 | 0.7269 (3) | 0.9048 (2) | 0.4504 (2) | 0.0188 (5) |
| C16 | 0.5182 (3) | 1.1700 (3) | 0.2764 (2) | 0.0208 (6) |
| H16A | 0.5476 | 1.2442 | 0.3358 | 0.025* |
| C2 | 0.1426 (3) | 0.7039 (3) | 0.3005 (2) | 0.0193 (6) |
| H2A | 0.0985 | 0.7320 | 0.3542 | 0.023* |
| C11 | 0.91561 (7) | 0.22764 (6) | 0.18640 (5) | 0.02052 (17) |
| O14 | 0.8085 (2) | 0.10315 (19) | 0.15308 (18) | 0.0319 (5) |
| O13 | 1.0750 (2) | 0.2169 (2) | 0.18132 (17) | 0.0346 (5) |
| O12 | 0.9291 (3) | 0.3020 (2) | 0.29168 (18) | 0.0458 (6) |
| O11 | 0.8522 (3) | 0.2870 (2) | 0.1189 (2) | 0.0512 (7) |
| C1W | 0.3474 (5) | 0.5122 (4) | 0.3889 (3) | 0.0481 (9) |
| H1WA | 0.2545 | 0.5255 | 0.4108 | 0.072* |
| H1WB | 0.3419 | 0.5313 | 0.3283 | 0.072* |
| H1WC | 0.4448 | 0.5668 | 0.4438 | 0.072* |
| O1W | 0.3486 (3) | 0.3837 (2) | 0.36481 (19) | 0.0415 (6) |
| H1W | 0.2370 | 0.3263 | 0.3121 | 0.050* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|-------------|-------------|--------------|--------------|--------------|
| Fe1 | 0.01026 (19) | 0.0101 (2) | 0.0073 (2) | 0.00144 (14) | 0.00143 (14) | 0.00187 (15) |
| O1 | 0.0143 (8) | 0.0139 (9) | 0.0091 (9) | 0.0001 (7) | 0.0000 (7) | 0.0002 (8) |
| N2 | 0.0109 (10) | 0.0113 (10) | 0.0095 (10) | 0.0014 (8) | 0.0027 (8) | 0.0027 (8) |
| O2 | 0.0202 (9) | 0.0179 (9) | 0.0092 (9) | 0.0018 (7) | 0.0047 (7) | 0.0033 (7) |
| N1 | 0.0133 (10) | 0.0139 (10) | 0.0091 (10) | 0.0041 (8) | 0.0033 (8) | 0.0045 (9) |
| N6 | 0.0126 (10) | 0.0131 (10) | 0.0121 (10) | 0.0022 (8) | 0.0044 (8) | 0.0042 (9) |
| N3 | 0.0124 (10) | 0.0123 (10) | 0.0122 (10) | 0.0022 (8) | 0.0014 (8) | 0.0056 (8) |
| N5 | 0.0160 (10) | 0.0115 (10) | 0.0091 (10) | 0.0025 (8) | 0.0011 (8) | 0.0015 (8) |
| N4 | 0.0097 (9) | 0.0148 (10) | 0.0126 (10) | 0.0012 (8) | 0.0046 (8) | 0.0054 (9) |
| C5 | 0.0137 (11) | 0.0116 (11) | 0.0095 (11) | 0.0045 (9) | 0.0029 (9) | 0.0033 (10) |
| O4 | 0.0575 (15) | 0.0241 (11) | 0.0087 (10) | 0.0165 (10) | -0.0048 (9) | 0.0003 (9) |
| O3 | 0.0580 (14) | 0.0145 (10) | 0.0134 (10) | 0.0100 (9) | -0.0006 (9) | -0.0009 (9) |
| C7 | 0.0141 (12) | 0.0115 (12) | 0.0127 (13) | 0.0056 (9) | 0.0053 (10) | 0.0053 (10) |
| C6 | 0.0125 (11) | 0.0122 (12) | 0.0113 (12) | 0.0041 (9) | 0.0020 (10) | 0.0057 (11) |
| C9 | 0.0177 (12) | 0.0204 (13) | 0.0164 (13) | 0.0053 (10) | 0.0068 (10) | 0.0086 (11) |
| C4 | 0.0143 (12) | 0.0179 (13) | 0.0117 (12) | 0.0012 (10) | 0.0010 (10) | 0.0028 (10) |
| C15 | 0.0205 (13) | 0.0209 (14) | 0.0277 (15) | 0.0101 (11) | 0.0104 (11) | 0.0157 (12) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| C13 | 0.0098 (11) | 0.0188 (13) | 0.0123 (12) | 0.0019 (9) | 0.0026 (9) | 0.0058 (10) |
| C8 | 0.0140 (12) | 0.0139 (12) | 0.0132 (12) | 0.0049 (10) | 0.0035 (10) | 0.0067 (10) |
| C11 | 0.0116 (12) | 0.0226 (14) | 0.0256 (15) | 0.0007 (10) | 0.0006 (11) | 0.0108 (12) |
| C10 | 0.0143 (12) | 0.0304 (15) | 0.0271 (15) | 0.0053 (11) | 0.0080 (11) | 0.0156 (13) |
| C24 | 0.0145 (11) | 0.0134 (12) | 0.0163 (12) | 0.0045 (9) | 0.0079 (10) | 0.0043 (10) |
| C14 | 0.0136 (12) | 0.0257 (14) | 0.0194 (13) | 0.0061 (10) | 0.0066 (10) | 0.0140 (12) |
| C22 | 0.0356 (16) | 0.0287 (16) | 0.0272 (16) | 0.0166 (13) | 0.0088 (13) | 0.0192 (13) |
| C17 | 0.0147 (12) | 0.0156 (12) | 0.0147 (13) | 0.0010 (10) | 0.0031 (10) | 0.0048 (11) |
| C18 | 0.0219 (13) | 0.0141 (13) | 0.0150 (13) | 0.0038 (10) | 0.0032 (10) | 0.0036 (11) |
| C3 | 0.0127 (12) | 0.0250 (14) | 0.0169 (13) | 0.0005 (10) | 0.0048 (10) | 0.0079 (11) |
| C20 | 0.0204 (13) | 0.0175 (13) | 0.0132 (13) | 0.0045 (10) | 0.0032 (10) | 0.0054 (11) |
| C21 | 0.0362 (16) | 0.0251 (15) | 0.0149 (13) | 0.0113 (12) | 0.0030 (12) | 0.0087 (12) |
| C1 | 0.0178 (12) | 0.0176 (13) | 0.0112 (12) | 0.0057 (10) | 0.0048 (10) | 0.0045 (10) |
| C12 | 0.0152 (12) | 0.0156 (12) | 0.0131 (12) | 0.0023 (10) | 0.0002 (10) | 0.0060 (10) |
| C23 | 0.0229 (13) | 0.0172 (13) | 0.0268 (15) | 0.0077 (11) | 0.0144 (12) | 0.0098 (12) |
| C19 | 0.0218 (13) | 0.0180 (13) | 0.0120 (13) | 0.0058 (11) | 0.0004 (10) | 0.0036 (11) |
| C16 | 0.0260 (14) | 0.0148 (13) | 0.0210 (14) | 0.0067 (11) | 0.0078 (11) | 0.0055 (11) |
| C2 | 0.0194 (13) | 0.0269 (15) | 0.0153 (13) | 0.0084 (11) | 0.0093 (11) | 0.0091 (12) |
| C11 | 0.0209 (3) | 0.0169 (3) | 0.0216 (3) | 0.0046 (3) | 0.0049 (3) | 0.0063 (3) |
| O14 | 0.0243 (10) | 0.0215 (11) | 0.0485 (13) | 0.0023 (8) | 0.0087 (10) | 0.0160 (10) |
| O13 | 0.0217 (10) | 0.0423 (13) | 0.0359 (12) | 0.0036 (9) | 0.0139 (9) | 0.0097 (11) |
| O12 | 0.0401 (13) | 0.0555 (16) | 0.0267 (12) | 0.0222 (12) | 0.0075 (10) | -0.0049 (11) |
| O11 | 0.0555 (16) | 0.0345 (13) | 0.0552 (16) | 0.0034 (12) | -0.0092 (13) | 0.0299 (12) |
| C1W | 0.049 (2) | 0.049 (2) | 0.055 (2) | 0.0203 (18) | 0.0172 (18) | 0.0263 (19) |
| O1W | 0.0393 (13) | 0.0343 (13) | 0.0425 (14) | 0.0149 (10) | 0.0025 (11) | 0.0092 (11) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|----------|-----------|
| Fe1—N2 | 1.900 (2) | C13—H13A | 0.9300 |
| Fe1—N5 | 1.922 (2) | C11—C10 | 1.381 (4) |
| Fe1—N6 | 1.974 (2) | C11—C12 | 1.381 (4) |
| Fe1—N4 | 1.976 (2) | C11—H11A | 0.9300 |
| Fe1—N1 | 1.977 (2) | C10—H10A | 0.9300 |
| Fe1—N3 | 1.977 (2) | C24—C23 | 1.388 (4) |
| O1—C6 | 1.206 (3) | C24—H24A | 0.9300 |
| N2—C7 | 1.384 (3) | C14—H14A | 0.9300 |
| N2—C6 | 1.391 (3) | C22—C23 | 1.377 (4) |
| O2—C7 | 1.216 (3) | C22—C21 | 1.387 (4) |
| N1—C1 | 1.344 (3) | C22—H22A | 0.9300 |
| N1—C5 | 1.362 (3) | C17—C16 | 1.379 (4) |
| N6—C24 | 1.343 (3) | C17—C18 | 1.500 (4) |
| N6—C20 | 1.356 (3) | C3—C2 | 1.380 (4) |
| N3—C12 | 1.345 (3) | C3—H3A | 0.9300 |
| N3—C8 | 1.356 (3) | C20—C21 | 1.379 (4) |
| N5—C19 | 1.379 (3) | C20—C19 | 1.500 (4) |
| N5—C18 | 1.381 (3) | C21—H21A | 0.9300 |
| N4—C13 | 1.347 (3) | C1—C2 | 1.390 (4) |
| N4—C17 | 1.353 (3) | C1—H1A | 0.9300 |
| C5—C4 | 1.371 (3) | C12—H12A | 0.9300 |

supplementary materials

| | | | |
|------------|-------------|--------------|-----------|
| C5—C6 | 1.507 (3) | C23—H23A | 0.9300 |
| O4—C19 | 1.219 (3) | C16—H16A | 0.9300 |
| O3—C18 | 1.211 (3) | C2—H2A | 0.9300 |
| C7—C8 | 1.499 (3) | C11—O12 | 1.433 (2) |
| C9—C8 | 1.374 (4) | C11—O14 | 1.434 (2) |
| C9—C10 | 1.380 (4) | C11—O11 | 1.434 (2) |
| C9—H9A | 0.9300 | C11—O13 | 1.446 (2) |
| C4—C3 | 1.392 (4) | C1W—O1W | 1.416 (4) |
| C4—H4A | 0.9300 | C1W—H1WA | 0.9600 |
| C15—C14 | 1.381 (4) | C1W—H1WB | 0.9600 |
| C15—C16 | 1.384 (4) | C1W—H1WC | 0.9600 |
| C15—H15A | 0.9300 | O1W—H1W | 1.0342 |
| C13—C14 | 1.385 (4) | | |
| N2—Fe1—N5 | 178.55 (8) | C12—C11—H11A | 120.3 |
| N2—Fe1—N6 | 100.02 (9) | C9—C10—C11 | 119.2 (2) |
| N5—Fe1—N6 | 81.43 (9) | C9—C10—H10A | 120.4 |
| N2—Fe1—N4 | 96.55 (9) | C11—C10—H10A | 120.4 |
| N5—Fe1—N4 | 82.01 (9) | N6—C24—C23 | 121.5 (2) |
| N6—Fe1—N4 | 163.43 (9) | N6—C24—H24A | 119.2 |
| N2—Fe1—N1 | 82.14 (8) | C23—C24—H24A | 119.2 |
| N5—Fe1—N1 | 97.85 (9) | C15—C14—C13 | 119.8 (2) |
| N6—Fe1—N1 | 89.91 (8) | C15—C14—H14A | 120.1 |
| N4—Fe1—N1 | 92.51 (8) | C13—C14—H14A | 120.1 |
| N2—Fe1—N3 | 82.31 (8) | C23—C22—C21 | 119.2 (3) |
| N5—Fe1—N3 | 97.73 (9) | C23—C22—H22A | 120.4 |
| N6—Fe1—N3 | 91.22 (8) | C21—C22—H22A | 120.4 |
| N4—Fe1—N3 | 90.84 (8) | N4—C17—C16 | 122.9 (2) |
| N1—Fe1—N3 | 164.37 (9) | N4—C17—C18 | 115.5 (2) |
| C7—N2—C6 | 123.1 (2) | C16—C17—C18 | 121.6 (2) |
| C7—N2—Fe1 | 117.82 (16) | O3—C18—N5 | 127.9 (2) |
| C6—N2—Fe1 | 118.93 (16) | O3—C18—C17 | 122.0 (2) |
| C1—N1—C5 | 118.1 (2) | N5—C18—C17 | 110.1 (2) |
| C1—N1—Fe1 | 127.89 (17) | C2—C3—C4 | 118.6 (2) |
| C5—N1—Fe1 | 113.91 (16) | C2—C3—H3A | 120.7 |
| C24—N6—C20 | 118.5 (2) | C4—C3—H3A | 120.7 |
| C24—N6—Fe1 | 126.44 (17) | N6—C20—C21 | 122.6 (2) |
| C20—N6—Fe1 | 115.01 (17) | N6—C20—C19 | 114.9 (2) |
| C12—N3—C8 | 118.2 (2) | C21—C20—C19 | 122.6 (2) |
| C12—N3—Fe1 | 128.32 (17) | C20—C21—C22 | 118.5 (3) |
| C8—N3—Fe1 | 113.43 (16) | C20—C21—H21A | 120.7 |
| C19—N5—C18 | 123.3 (2) | C22—C21—H21A | 120.7 |
| C19—N5—Fe1 | 118.66 (17) | N1—C1—C2 | 121.8 (2) |
| C18—N5—Fe1 | 118.07 (16) | N1—C1—H1A | 119.1 |
| C13—N4—C17 | 117.9 (2) | C2—C1—H1A | 119.1 |
| C13—N4—Fe1 | 127.82 (17) | N3—C12—C11 | 121.8 (2) |
| C17—N4—Fe1 | 114.28 (16) | N3—C12—H12A | 119.1 |
| N1—C5—C4 | 122.6 (2) | C11—C12—H12A | 119.1 |
| N1—C5—C6 | 115.1 (2) | C22—C23—C24 | 119.7 (3) |
| C4—C5—C6 | 122.3 (2) | C22—C23—H23A | 120.2 |

| | | | |
|--------------|-----------|---------------|-------------|
| O2—C7—N2 | 128.2 (2) | C24—C23—H23A | 120.2 |
| O2—C7—C8 | 122.1 (2) | O4—C19—N5 | 127.6 (2) |
| N2—C7—C8 | 109.7 (2) | O4—C19—C20 | 122.4 (2) |
| O1—C6—N2 | 128.2 (2) | N5—C19—C20 | 110.0 (2) |
| O1—C6—C5 | 122.5 (2) | C17—C16—C15 | 118.8 (2) |
| N2—C6—C5 | 109.3 (2) | C17—C16—H16A | 120.6 |
| C8—C9—C10 | 118.7 (2) | C15—C16—H16A | 120.6 |
| C8—C9—H9A | 120.6 | C3—C2—C1 | 119.7 (2) |
| C10—C9—H9A | 120.6 | C3—C2—H2A | 120.2 |
| C5—C4—C3 | 119.1 (2) | C1—C2—H2A | 120.2 |
| C5—C4—H4A | 120.5 | O12—C11—O14 | 109.89 (15) |
| C3—C4—H4A | 120.5 | O12—C11—O11 | 110.31 (17) |
| C14—C15—C16 | 118.6 (2) | O14—C11—O11 | 108.86 (14) |
| C14—C15—H15A | 120.7 | O12—C11—O13 | 108.75 (13) |
| C16—C15—H15A | 120.7 | O14—C11—O13 | 109.36 (13) |
| N4—C13—C14 | 121.8 (2) | O11—C11—O13 | 109.66 (16) |
| N4—C13—H13A | 119.1 | O1W—C1W—H1WA | 109.5 |
| C14—C13—H13A | 119.1 | O1W—C1W—H1WB | 109.5 |
| N3—C8—C9 | 122.6 (2) | H1WA—C1W—H1WB | 109.5 |
| N3—C8—C7 | 115.4 (2) | O1W—C1W—H1WC | 109.5 |
| C9—C8—C7 | 121.9 (2) | H1WA—C1W—H1WC | 109.5 |
| C10—C11—C12 | 119.5 (2) | H1WB—C1W—H1WC | 109.5 |
| C10—C11—H11A | 120.3 | C1W—O1W—H1W | 108.2 |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O1W—H1W \cdots O13 ⁱ | 1.03 | 1.92 | 2.916 (3) | 160 |

Symmetry codes: (i) $x-1, y, z$.

Fig. 1

