

[2,6-Bis(6-methylquinolin-2-yl)pyridine- κ^3N,N',N'']dichloridoiron(II)

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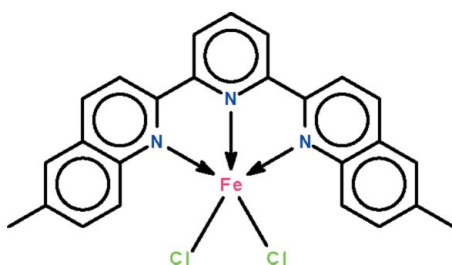
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.035; wR factor = 0.108; data-to-parameter ratio = 16.3.

In the molecule of the title compound, $[\text{FeCl}_2(\text{C}_{25}\text{H}_{19}\text{N}_3)]$, the three N atoms span the axial-equatorial-axial sites of the trigonal-bipyramidal coordination polyhedron; the geometry of the Fe^{II} atom is 32% distorted from trigonal-bipyramidal (towards square-pyramidal along the Berry pseudorotation pathway). One of the Cl atoms is disordered over two positions in a 0.938 (11):0.062 (11) ratio. Intermolecular $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonding occurs in the crystal structure.

Related literature

For the synthesis of the N -heterocyclic ligand, see: Buu-Hoi *et al.* (1965). For a related structure, see: Li *et al.* (2010).



Experimental

Crystal data

$[\text{FeCl}_2(\text{C}_{25}\text{H}_{19}\text{N}_3)]$

$M_r = 488.18$

Triclinic, $P\bar{1}$

$a = 9.6228$ (7) Å

$b = 10.2558$ (8) Å

$c = 10.7324$ (8) Å

$\alpha = 94.352$ (1)°

$\beta = 95.481$ (1)°

$\gamma = 96.121$ (1)°

$V = 1044.35$ (14) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 1.00$ mm⁻¹

$T = 100$ K

$0.30 \times 0.10 \times 0.05$ mm

Data collection

Bruker SMART APEX
diffractometer

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\text{min}} = 0.754$, $T_{\text{max}} = 0.952$

9910 measured reflections

4757 independent reflections

3954 reflections with $I > 2\sigma(I)$

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

Refinement

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

$wR(F^2) = 0.108$

$S = 1.05$

4757 reflections

292 parameters

7 restraints

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.54$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.44$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|--------|-------------|---------|------------|
| Fe1—N1 | 2.2386 (19) | Fe1—Cl1 | 2.3636 (8) |
| Fe1—N2 | 2.103 (2) | Fe1—Cl2 | 2.2748 (7) |
| Fe1—N3 | 2.2523 (19) | | |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C8—H8 \cdots Cl2 ⁱ | 0.95 | 2.70 | 3.561 (2) | 151 |
| C17—H17 \cdots Cl1 ⁱⁱ | 0.95 | 2.73 | 3.538 (4) | 144 |

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x + 1, -y + 2, -z + 1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5032).

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supplementary materials

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[2,6-Bis(6-methylquinolin-2-yl)pyridine- κ^3N,N',N'']dichloridoiron(II)

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Comment

A recent study reported the chromium(III) chloride adduct of 2,6-bis(*p*-bromophenylimino)pyridine; the *N*-heterocycle chelates to the metal atom in a terdentate manner (Li *et al.*, 2010). Bis[2'-(6-methylquinolinyl)]pyridine has a similar set of donor sites capable of binding in this manner, as demonstrated in the present iron dichloride adduct (Scheme I, Fig. 1 and Table 1). In the molecule of FeCl₂(C₂₅H₁₉N₃), the three N atoms span the axial–equatorial–axial sites of the trigonal bipyramidal coordination polyhedron; the geometry of Fe is 32% distorted from the trigonal bipyramid along the Berry pseudorotation pathway. Intermolecular C—H···Cl hydrogen bonding occurs in the crystal structure (Table 2).

Experimental

The ligand was synthesized by using a literature procedure (Buu-Hoi *et al.*, 1965).

Bis[2'-(6-methylquinolinyl)]pyridine (0.018 g, 0.05 mmol), and ferrous chloride tetrahydrate (0.02 g, 0.05 mmol) along with five drops of 1 *M* hydrochloric acid were dissolved in ethanol (10 ml). The mixture was heated in a Teflon-lined, stainless-steel Parr bomb at 363 K for 120 h. The bomb was cooled at 5 K per hour. Black crystals were isolated.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C).

One of the chlorine atoms is disordered over two positions in a 0.938 (11):0.062 (11) ratio. The Fe–Cl pair of distances were restrained to within 0.01 Å of each other; the anisotropic temperature factors of the minor component were restrained to be nearly isotropic.

Figures

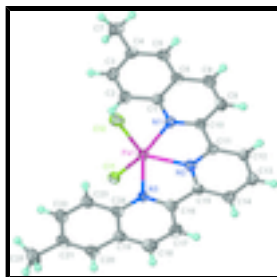


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of FeCl₂(C₂₅H₁₉N₃) at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder is not shown.

supplementary materials

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Crystal data

| | |
|---|---|
| [FeCl ₂ (C ₂₅ H ₁₉ N ₃)] | $Z = 2$ |
| $M_r = 488.18$ | $F(000) = 500$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.552 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 9.6228 (7) \text{ \AA}$ | Cell parameters from 4559 reflections |
| $b = 10.2558 (8) \text{ \AA}$ | $\theta = 2.6\text{--}28.2^\circ$ |
| $c = 10.7324 (8) \text{ \AA}$ | $\mu = 1.00 \text{ mm}^{-1}$ |
| $\alpha = 94.352 (1)^\circ$ | $T = 100 \text{ K}$ |
| $\beta = 95.481 (1)^\circ$ | Block, black |
| $\gamma = 96.121 (1)^\circ$ | $0.30 \times 0.10 \times 0.05 \text{ mm}$ |
| $V = 1044.35 (14) \text{ \AA}^3$ | |

Data collection

| | |
|---|--|
| Bruker SMART APEX diffractometer | 4757 independent reflections |
| Radiation source: fine-focus sealed tube graphite | 3954 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.024$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.1^\circ$ |
| $T_{\text{min}} = 0.754$, $T_{\text{max}} = 0.952$ | $h = -12 \rightarrow 12$ |
| 9910 measured reflections | $k = -13 \rightarrow 13$ |
| | $l = -13 \rightarrow 13$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.108$ | H-atom parameters constrained |
| $S = 1.05$ | $w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 1.0201P]$ |
| 4757 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 292 parameters | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 7 restraints | $\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$ |

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|-------------|-------------|----------------------------------|-----------|
| Fe1 | 0.34748 (3) | 0.81412 (3) | 0.22283 (3) | 0.01918 (11) | |

| | | | | | |
|------|--------------|--------------|--------------|--------------|------------|
| C11 | 0.16748 (18) | 0.73795 (9) | 0.34366 (18) | 0.0225 (4) | 0.938 (11) |
| C11' | 0.211 (5) | 0.730 (2) | 0.377 (3) | 0.047 (6) | 0.062 (11) |
| C12 | 0.27672 (8) | 0.85227 (6) | 0.02182 (6) | 0.03212 (17) | |
| N1 | 0.4298 (2) | 0.61956 (18) | 0.19090 (18) | 0.0185 (4) | |
| N2 | 0.5602 (2) | 0.84553 (19) | 0.29589 (18) | 0.0188 (4) | |
| N3 | 0.3828 (2) | 1.02324 (19) | 0.31020 (18) | 0.0185 (4) | |
| C1 | 0.3563 (2) | 0.5061 (2) | 0.1319 (2) | 0.0197 (5) | |
| C2 | 0.2114 (3) | 0.5039 (2) | 0.0943 (2) | 0.0236 (5) | |
| H2 | 0.1656 | 0.5803 | 0.1097 | 0.028* | |
| C3 | 0.1373 (3) | 0.3912 (2) | 0.0355 (2) | 0.0233 (5) | |
| H3 | 0.0398 | 0.3910 | 0.0107 | 0.028* | |
| C4 | 0.2005 (3) | 0.2747 (2) | 0.0101 (2) | 0.0214 (5) | |
| C5 | 0.3407 (3) | 0.2756 (2) | 0.0458 (2) | 0.0218 (5) | |
| H5 | 0.3846 | 0.1984 | 0.0289 | 0.026* | |
| C6 | 0.4219 (3) | 0.3901 (2) | 0.1079 (2) | 0.0201 (5) | |
| C7 | 0.1113 (3) | 0.1550 (2) | -0.0542 (2) | 0.0268 (5) | |
| H7A | 0.1709 | 0.0855 | -0.0718 | 0.040* | |
| H7B | 0.0637 | 0.1775 | -0.1333 | 0.040* | |
| H7C | 0.0411 | 0.1239 | 0.0004 | 0.040* | |
| C8 | 0.5659 (3) | 0.3955 (2) | 0.1470 (2) | 0.0251 (5) | |
| H8 | 0.6136 | 0.3202 | 0.1327 | 0.030* | |
| C9 | 0.6372 (3) | 0.5093 (3) | 0.2057 (2) | 0.0255 (5) | |
| H9 | 0.7346 | 0.5133 | 0.2328 | 0.031* | |
| C10 | 0.5660 (2) | 0.6202 (2) | 0.2256 (2) | 0.0187 (5) | |
| C11 | 0.6408 (2) | 0.7465 (2) | 0.2875 (2) | 0.0183 (5) | |
| C12 | 0.7816 (2) | 0.7639 (2) | 0.3330 (2) | 0.0209 (5) | |
| H12 | 0.8377 | 0.6935 | 0.3254 | 0.025* | |
| C13 | 0.8396 (3) | 0.8867 (2) | 0.3902 (2) | 0.0228 (5) | |
| H13 | 0.9361 | 0.9009 | 0.4218 | 0.027* | |
| C14 | 0.7556 (3) | 0.9881 (2) | 0.4005 (2) | 0.0224 (5) | |
| H14 | 0.7932 | 1.0722 | 0.4401 | 0.027* | |
| C15 | 0.6156 (2) | 0.9643 (2) | 0.3520 (2) | 0.0197 (5) | |
| C16 | 0.5151 (3) | 1.0641 (2) | 0.3564 (2) | 0.0203 (5) | |
| C17 | 0.5577 (3) | 1.1949 (3) | 0.4088 (3) | 0.0285 (6) | |
| H17 | 0.6527 | 1.2214 | 0.4412 | 0.034* | |
| C18 | 0.4599 (3) | 1.2824 (3) | 0.4121 (2) | 0.0281 (6) | |
| H18 | 0.4876 | 1.3704 | 0.4466 | 0.034* | |
| C19 | 0.3201 (3) | 1.2440 (2) | 0.3654 (2) | 0.0211 (5) | |
| C20 | 0.2134 (3) | 1.3296 (2) | 0.3667 (2) | 0.0238 (5) | |
| H20 | 0.2370 | 1.4186 | 0.3999 | 0.029* | |
| C21 | 0.0778 (3) | 1.2866 (2) | 0.3213 (2) | 0.0259 (5) | |
| C22 | 0.0444 (3) | 1.1538 (2) | 0.2698 (2) | 0.0249 (5) | |
| H22 | -0.0495 | 1.1237 | 0.2364 | 0.030* | |
| C23 | 0.1446 (3) | 1.0680 (2) | 0.2674 (2) | 0.0223 (5) | |
| H23 | 0.1192 | 0.9793 | 0.2339 | 0.027* | |
| C24 | 0.2846 (3) | 1.1113 (2) | 0.3143 (2) | 0.0199 (5) | |
| C25 | -0.0365 (3) | 1.3765 (3) | 0.3212 (3) | 0.0342 (6) | |
| H25A | 0.0059 | 1.4683 | 0.3288 | 0.051* | |
| H25B | -0.0911 | 1.3611 | 0.3923 | 0.051* | |

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H25C -0.0985 1.3583 0.2425 0.051*

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|--------------|--------------|--------------|--------------|--------------|---------------|
| Fe1 | 0.01616 (18) | 0.01714 (17) | 0.02359 (19) | 0.00279 (12) | 0.00055 (13) | -0.00168 (13) |
| Cl1 | 0.0196 (5) | 0.0208 (4) | 0.0263 (6) | -0.0002 (3) | 0.0040 (4) | -0.0017 (3) |
| Cl1' | 0.046 (10) | 0.052 (8) | 0.042 (9) | -0.001 (7) | 0.013 (8) | -0.005 (6) |
| Cl2 | 0.0433 (4) | 0.0305 (3) | 0.0245 (3) | 0.0176 (3) | 0.0004 (3) | -0.0001 (2) |
| N1 | 0.0183 (10) | 0.0174 (9) | 0.0195 (10) | 0.0013 (7) | 0.0009 (8) | 0.0012 (7) |
| N2 | 0.0170 (9) | 0.0187 (9) | 0.0206 (10) | 0.0006 (7) | 0.0048 (8) | 0.0005 (7) |
| N3 | 0.0173 (10) | 0.0197 (9) | 0.0182 (9) | 0.0014 (8) | 0.0023 (7) | 0.0001 (7) |
| C1 | 0.0218 (12) | 0.0175 (10) | 0.0200 (11) | 0.0019 (9) | 0.0032 (9) | 0.0014 (9) |
| C2 | 0.0221 (12) | 0.0200 (11) | 0.0279 (13) | 0.0032 (9) | 0.0000 (10) | -0.0021 (9) |
| C3 | 0.0191 (12) | 0.0218 (11) | 0.0279 (13) | 0.0002 (9) | 0.0005 (10) | 0.0012 (10) |
| C4 | 0.0274 (13) | 0.0191 (11) | 0.0171 (11) | 0.0009 (9) | 0.0009 (9) | 0.0014 (9) |
| C5 | 0.0275 (13) | 0.0181 (11) | 0.0203 (12) | 0.0049 (9) | 0.0032 (10) | 0.0005 (9) |
| C6 | 0.0242 (12) | 0.0185 (11) | 0.0187 (11) | 0.0037 (9) | 0.0049 (9) | 0.0040 (9) |
| C7 | 0.0339 (14) | 0.0191 (11) | 0.0255 (13) | -0.0015 (10) | 0.0006 (11) | -0.0012 (9) |
| C8 | 0.0268 (13) | 0.0236 (12) | 0.0263 (13) | 0.0073 (10) | 0.0046 (10) | 0.0019 (10) |
| C9 | 0.0214 (12) | 0.0284 (13) | 0.0276 (13) | 0.0068 (10) | 0.0025 (10) | 0.0022 (10) |
| C10 | 0.0202 (11) | 0.0186 (11) | 0.0178 (11) | 0.0028 (9) | 0.0035 (9) | 0.0032 (9) |
| C11 | 0.0186 (11) | 0.0206 (11) | 0.0154 (11) | 0.0007 (9) | 0.0036 (9) | 0.0006 (9) |
| C12 | 0.0182 (11) | 0.0263 (12) | 0.0183 (11) | 0.0036 (9) | 0.0014 (9) | 0.0016 (9) |
| C13 | 0.0177 (11) | 0.0302 (12) | 0.0202 (12) | 0.0010 (10) | 0.0029 (9) | 0.0022 (10) |
| C14 | 0.0222 (12) | 0.0228 (11) | 0.0208 (12) | -0.0021 (9) | 0.0030 (9) | -0.0016 (9) |
| C15 | 0.0210 (12) | 0.0206 (11) | 0.0181 (11) | 0.0030 (9) | 0.0046 (9) | 0.0006 (9) |
| C16 | 0.0229 (12) | 0.0201 (11) | 0.0189 (11) | 0.0026 (9) | 0.0065 (9) | 0.0023 (9) |
| C17 | 0.0276 (13) | 0.0292 (13) | 0.0274 (13) | 0.0009 (11) | 0.0000 (11) | 0.0007 (10) |
| C18 | 0.0343 (15) | 0.0222 (12) | 0.0273 (13) | -0.0010 (11) | 0.0056 (11) | 0.0012 (10) |
| C19 | 0.0256 (12) | 0.0192 (11) | 0.0187 (11) | 0.0009 (9) | 0.0054 (9) | 0.0012 (9) |
| C20 | 0.0309 (13) | 0.0181 (11) | 0.0232 (12) | 0.0039 (10) | 0.0076 (10) | 0.0003 (9) |
| C21 | 0.0285 (13) | 0.0242 (12) | 0.0281 (13) | 0.0085 (10) | 0.0100 (10) | 0.0063 (10) |
| C22 | 0.0227 (12) | 0.0248 (12) | 0.0285 (13) | 0.0036 (10) | 0.0066 (10) | 0.0056 (10) |
| C23 | 0.0241 (12) | 0.0192 (11) | 0.0242 (12) | 0.0029 (9) | 0.0053 (10) | 0.0010 (9) |
| C24 | 0.0230 (12) | 0.0187 (11) | 0.0196 (11) | 0.0039 (9) | 0.0071 (9) | 0.0028 (9) |
| C25 | 0.0296 (14) | 0.0257 (13) | 0.0502 (18) | 0.0101 (11) | 0.0092 (13) | 0.0059 (12) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-------------|---------|-----------|
| Fe1—N1 | 2.2386 (19) | C9—H9 | 0.9500 |
| Fe1—N2 | 2.103 (2) | C10—C11 | 1.488 (3) |
| Fe1—N3 | 2.2523 (19) | C11—C12 | 1.384 (3) |
| Fe1—Cl1 | 2.3636 (8) | C12—C13 | 1.393 (3) |
| Fe1—Cl1' | 2.369 (9) | C12—H12 | 0.9500 |
| Fe1—Cl2 | 2.2748 (7) | C13—C14 | 1.388 (3) |
| N1—C10 | 1.328 (3) | C13—H13 | 0.9500 |
| N1—C1 | 1.372 (3) | C14—C15 | 1.386 (3) |
| N2—C11 | 1.345 (3) | C14—H14 | 0.9500 |

| | | | |
|--------------|-------------|-------------|-------------|
| N2—C15 | 1.349 (3) | C15—C16 | 1.483 (3) |
| N3—C16 | 1.333 (3) | C16—C17 | 1.416 (3) |
| N3—C24 | 1.376 (3) | C17—C18 | 1.369 (4) |
| C1—C2 | 1.411 (3) | C17—H17 | 0.9500 |
| C1—C6 | 1.423 (3) | C18—C19 | 1.394 (4) |
| C2—C3 | 1.368 (3) | C18—H18 | 0.9500 |
| C2—H2 | 0.9500 | C19—C24 | 1.422 (3) |
| C3—C4 | 1.418 (3) | C19—C20 | 1.420 (3) |
| C3—H3 | 0.9500 | C20—C21 | 1.363 (4) |
| C4—C5 | 1.365 (3) | C20—H20 | 0.9500 |
| C4—C7 | 1.501 (3) | C21—C22 | 1.422 (4) |
| C5—C6 | 1.421 (3) | C21—C25 | 1.508 (3) |
| C5—H5 | 0.9500 | C22—C23 | 1.373 (3) |
| C6—C8 | 1.403 (3) | C22—H22 | 0.9500 |
| C7—H7A | 0.9800 | C23—C24 | 1.406 (3) |
| C7—H7B | 0.9800 | C23—H23 | 0.9500 |
| C7—H7C | 0.9800 | C25—H25A | 0.9800 |
| C8—C9 | 1.365 (4) | C25—H25B | 0.9800 |
| C8—H8 | 0.9500 | C25—H25C | 0.9800 |
| C9—C10 | 1.402 (3) | | |
| N2—Fe1—N1 | 74.57 (7) | C10—C9—H9 | 120.2 |
| N2—Fe1—N3 | 74.29 (7) | N1—C10—C9 | 122.6 (2) |
| N1—Fe1—N3 | 148.58 (7) | N1—C10—C11 | 116.18 (19) |
| N2—Fe1—C12 | 121.77 (6) | C9—C10—C11 | 121.2 (2) |
| N1—Fe1—C12 | 100.57 (5) | N2—C11—C12 | 121.4 (2) |
| N3—Fe1—C12 | 99.20 (5) | N2—C11—C10 | 114.5 (2) |
| N2—Fe1—C11 | 122.35 (8) | C12—C11—C10 | 124.1 (2) |
| N1—Fe1—C11 | 95.84 (6) | C11—C12—C13 | 118.8 (2) |
| N3—Fe1—C11 | 97.19 (6) | C11—C12—H12 | 120.6 |
| C12—Fe1—C11 | 115.88 (6) | C13—C12—H12 | 120.6 |
| N2—Fe1—C11' | 109.4 (15) | C14—C13—C12 | 119.6 (2) |
| N1—Fe1—C11' | 90.5 (9) | C14—C13—H13 | 120.2 |
| N3—Fe1—C11' | 95.9 (5) | C12—C13—H13 | 120.2 |
| C12—Fe1—C11' | 128.8 (15) | C15—C14—C13 | 118.6 (2) |
| C11—Fe1—C11' | 13.1 (14) | C15—C14—H14 | 120.7 |
| C10—N1—C1 | 118.53 (19) | C13—C14—H14 | 120.7 |
| C10—N1—Fe1 | 114.81 (15) | N2—C15—C14 | 121.5 (2) |
| C1—N1—Fe1 | 126.46 (15) | N2—C15—C16 | 114.5 (2) |
| C11—N2—C15 | 120.0 (2) | C14—C15—C16 | 124.0 (2) |
| C11—N2—Fe1 | 119.82 (16) | N3—C16—C17 | 122.2 (2) |
| C15—N2—Fe1 | 120.19 (15) | N3—C16—C15 | 116.2 (2) |
| C16—N3—C24 | 118.6 (2) | C17—C16—C15 | 121.5 (2) |
| C16—N3—Fe1 | 114.61 (15) | C18—C17—C16 | 119.0 (2) |
| C24—N3—Fe1 | 126.69 (15) | C18—C17—H17 | 120.5 |
| N1—C1—C2 | 119.2 (2) | C16—C17—H17 | 120.5 |
| N1—C1—C6 | 121.8 (2) | C17—C18—C19 | 120.9 (2) |
| C2—C1—C6 | 119.0 (2) | C17—C18—H18 | 119.6 |
| C3—C2—C1 | 119.6 (2) | C19—C18—H18 | 119.6 |
| C3—C2—H2 | 120.2 | C18—C19—C24 | 117.1 (2) |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| C1—C2—H2 | 120.2 | C18—C19—C20 | 123.7 (2) |
| C2—C3—C4 | 122.5 (2) | C24—C19—C20 | 119.2 (2) |
| C2—C3—H3 | 118.8 | C21—C20—C19 | 121.3 (2) |
| C4—C3—H3 | 118.8 | C21—C20—H20 | 119.3 |
| C5—C4—C3 | 118.5 (2) | C19—C20—H20 | 119.3 |
| C5—C4—C7 | 122.5 (2) | C20—C21—C22 | 118.7 (2) |
| C3—C4—C7 | 119.0 (2) | C20—C21—C25 | 122.0 (2) |
| C4—C5—C6 | 121.1 (2) | C22—C21—C25 | 119.3 (2) |
| C4—C5—H5 | 119.4 | C23—C22—C21 | 121.6 (2) |
| C6—C5—H5 | 119.4 | C23—C22—H22 | 119.2 |
| C8—C6—C5 | 123.2 (2) | C21—C22—H22 | 119.2 |
| C8—C6—C1 | 117.5 (2) | C22—C23—C24 | 120.1 (2) |
| C5—C6—C1 | 119.4 (2) | C22—C23—H23 | 120.0 |
| C4—C7—H7A | 109.5 | C24—C23—H23 | 120.0 |
| C4—C7—H7B | 109.5 | N3—C24—C23 | 118.8 (2) |
| H7A—C7—H7B | 109.5 | N3—C24—C19 | 122.2 (2) |
| C4—C7—H7C | 109.5 | C23—C24—C19 | 119.0 (2) |
| H7A—C7—H7C | 109.5 | C21—C25—H25A | 109.5 |
| H7B—C7—H7C | 109.5 | C21—C25—H25B | 109.5 |
| C9—C8—C6 | 119.8 (2) | H25A—C25—H25B | 109.5 |
| C9—C8—H8 | 120.1 | C21—C25—H25C | 109.5 |
| C6—C8—H8 | 120.1 | H25A—C25—H25C | 109.5 |
| C8—C9—C10 | 119.7 (2) | H25B—C25—H25C | 109.5 |
| C8—C9—H9 | 120.2 | | |
| N2—Fe1—N1—C10 | -3.07 (16) | C1—N1—C10—C11 | 179.1 (2) |
| N3—Fe1—N1—C10 | -10.9 (2) | Fe1—N1—C10—C11 | 3.8 (3) |
| Cl2—Fe1—N1—C10 | 117.20 (16) | C8—C9—C10—N1 | 0.8 (4) |
| Cl1—Fe1—N1—C10 | -125.06 (17) | C8—C9—C10—C11 | -179.1 (2) |
| Cl1'—Fe1—N1—C10 | -113.1 (13) | C15—N2—C11—C12 | -1.4 (3) |
| N2—Fe1—N1—C1 | -177.9 (2) | Fe1—N2—C11—C12 | 179.14 (17) |
| N3—Fe1—N1—C1 | 174.20 (17) | C15—N2—C11—C10 | 178.9 (2) |
| Cl2—Fe1—N1—C1 | -57.66 (19) | Fe1—N2—C11—C10 | -0.5 (3) |
| Cl1—Fe1—N1—C1 | 60.08 (19) | N1—C10—C11—N2 | -2.3 (3) |
| Cl1'—Fe1—N1—C1 | 72.0 (13) | C9—C10—C11—N2 | 177.5 (2) |
| N1—Fe1—N2—C11 | 1.86 (17) | N1—C10—C11—C12 | 178.0 (2) |
| N3—Fe1—N2—C11 | 177.61 (19) | C9—C10—C11—C12 | -2.1 (4) |
| Cl2—Fe1—N2—C11 | -91.17 (17) | N2—C11—C12—C13 | 0.9 (3) |
| Cl1—Fe1—N2—C11 | 89.00 (17) | C10—C11—C12—C13 | -179.5 (2) |
| Cl1'—Fe1—N2—C11 | 86.8 (7) | C11—C12—C13—C14 | 0.2 (4) |
| N1—Fe1—N2—C15 | -177.57 (19) | C12—C13—C14—C15 | -0.7 (4) |
| N3—Fe1—N2—C15 | -1.81 (17) | C11—N2—C15—C14 | 0.9 (3) |
| Cl2—Fe1—N2—C15 | 89.40 (18) | Fe1—N2—C15—C14 | -179.70 (18) |
| Cl1—Fe1—N2—C15 | -90.42 (18) | C11—N2—C15—C16 | -179.2 (2) |
| Cl1'—Fe1—N2—C15 | -92.6 (7) | Fe1—N2—C15—C16 | 0.2 (3) |
| N2—Fe1—N3—C16 | 3.33 (16) | C13—C14—C15—N2 | 0.2 (4) |
| N1—Fe1—N3—C16 | 11.2 (2) | C13—C14—C15—C16 | -179.7 (2) |
| Cl2—Fe1—N3—C16 | -117.23 (16) | C24—N3—C16—C17 | -0.3 (3) |
| Cl1—Fe1—N3—C16 | 124.98 (17) | Fe1—N3—C16—C17 | 176.64 (19) |
| Cl1'—Fe1—N3—C16 | 111.9 (15) | C24—N3—C16—C15 | 178.7 (2) |

| | | | |
|-----------------|--------------|-----------------|--------------|
| N2—Fe1—N3—C24 | -180.0 (2) | Fe1—N3—C16—C15 | -4.3 (3) |
| N1—Fe1—N3—C24 | -172.12 (17) | N2—C15—C16—N3 | 2.9 (3) |
| C12—Fe1—N3—C24 | 59.44 (19) | C14—C15—C16—N3 | -177.2 (2) |
| C11—Fe1—N3—C24 | -58.34 (19) | N2—C15—C16—C17 | -178.1 (2) |
| C11'—Fe1—N3—C24 | -71.4 (15) | C14—C15—C16—C17 | 1.8 (4) |
| C10—N1—C1—C2 | -180.0 (2) | N3—C16—C17—C18 | -0.1 (4) |
| Fe1—N1—C1—C2 | -5.3 (3) | C15—C16—C17—C18 | -179.0 (2) |
| C10—N1—C1—C6 | 0.4 (3) | C16—C17—C18—C19 | 0.4 (4) |
| Fe1—N1—C1—C6 | 175.07 (17) | C17—C18—C19—C24 | -0.3 (4) |
| N1—C1—C2—C3 | -179.9 (2) | C17—C18—C19—C20 | 179.6 (2) |
| C6—C1—C2—C3 | -0.3 (4) | C18—C19—C20—C21 | -179.2 (2) |
| C1—C2—C3—C4 | 0.0 (4) | C24—C19—C20—C21 | 0.7 (4) |
| C2—C3—C4—C5 | -0.1 (4) | C19—C20—C21—C22 | -1.1 (4) |
| C2—C3—C4—C7 | 179.7 (2) | C19—C20—C21—C25 | -179.6 (2) |
| C3—C4—C5—C6 | 0.5 (4) | C20—C21—C22—C23 | 1.3 (4) |
| C7—C4—C5—C6 | -179.3 (2) | C25—C21—C22—C23 | 179.9 (2) |
| C4—C5—C6—C8 | 179.6 (2) | C21—C22—C23—C24 | -1.2 (4) |
| C4—C5—C6—C1 | -0.7 (4) | C16—N3—C24—C23 | -179.1 (2) |
| N1—C1—C6—C8 | 0.0 (4) | Fe1—N3—C24—C23 | 4.4 (3) |
| C2—C1—C6—C8 | -179.7 (2) | C16—N3—C24—C19 | 0.4 (3) |
| N1—C1—C6—C5 | -179.7 (2) | Fe1—N3—C24—C19 | -176.16 (17) |
| C2—C1—C6—C5 | 0.6 (3) | C22—C23—C24—N3 | -179.8 (2) |
| C5—C6—C8—C9 | 179.7 (2) | C22—C23—C24—C19 | 0.8 (4) |
| C1—C6—C8—C9 | 0.1 (4) | C18—C19—C24—N3 | -0.1 (4) |
| C6—C8—C9—C10 | -0.4 (4) | C20—C19—C24—N3 | -180.0 (2) |
| C1—N1—C10—C9 | -0.7 (3) | C18—C19—C24—C23 | 179.4 (2) |
| Fe1—N1—C10—C9 | -176.04 (19) | C20—C19—C24—C23 | -0.5 (3) |

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> |
|------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C8—H8 \cdots C12 ⁱ | 0.95 | 2.70 | 3.561 (2) | 151 |
| C17—H17 \cdots C11 ⁱⁱ | 0.95 | 2.73 | 3.538 (4) | 144 |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y+2, -z+1$.

Fig. 1

