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Bis(4′-chloro-2,2′:6′,2″-terpyridine-κ^3N,N',N'')iron(II) dinitrate dihydrate

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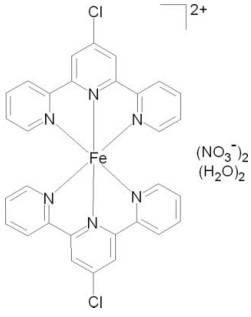
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.116; data-to-parameter ratio = 13.7.

The title complex, $[Fe(C_{15}H_{10}ClN_3)_2](NO_3)_2\cdot 2H_2O$, has a six-coordinate iron(II) center balanced by two nitrate anions. The Fe atom lies on a twofold rotation axis. The complex exhibits an octahedral coordination configuration, where the dihedral angle between the two planar tridentate ligands is 92.4 (1)°. The crystal structure involves O—H···O hydrogen bonds.

Related literature

For the related hydrochloride tetrafluoridoborate and hydrochloride hexafluorophosphate of 4′-chloro-2,2′:6′,2″-terpyridine, see: Huang & Qian (2007*a*). For the related Ru^{II}, Cu^{II}, Zn^{II}, Ni^{II} and Fe^{II} complexes of 4′-chloro-2,2′:6′,2″-terpyridine, see: Huang & Qian 2007*b*).



Experimental

Crystal data

$[Fe(C_{15}H_{10}ClN_3)_2](NO_3)_2\cdot 2H_2O$
M_r = 751.32
Monoclinic, $C2/c$
a = 18.049 (2) Å
b = 18.255 (3) Å
c = 10.0741 (14) Å
β = 102.668 (2)°
V = 3238.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 291 (2) K
0.12 × 0.10 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.922, T_{max} = 0.931
8547 measured reflections
3192 independent reflections
2120 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 0.97
3192 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N2	1.880 (3)	Fe1—N1^{i}	1.964 (2)
Fe1—N4	1.881 (3)	Fe1—N3	1.975 (2)
Fe1—N1	1.964 (2)	Fe1—N3^{i}	1.975 (2)
N2—Fe1—N4	180	N4—Fe1—N3	80.66 (6)
N2—Fe1—N1	81.04 (6)	N1—Fe1—N3	92.75 (8)
N4—Fe1—N1	98.96 (6)	N1^{i}—Fe1—N3	90.14 (8)
N1—Fe1—N1^{i}	162.09 (11)	N3—Fe1—N3^{i}	161.33 (11)
N2—Fe1—N3	99.34 (6)		

Symmetry code: (i) $-x+2, y, -z+\frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···O1^{ii}	0.93 (5)	1.96 (5)	2.859 (5)	162 (4)
O4—H4A···O3^{iii}	0.78 (3)	2.36 (3)	3.114 (4)	164 (3)
O4—H4A···O1^{iii}	0.78 (3)	2.40 (3)	3.042 (4)	140 (3)

Symmetry codes: (ii) $x, -y, z-\frac{1}{2}$; (iii) $x, y, z-1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2511).

References

Bruker (2000). *SMART* (Version 5.622), *SAINT* (Version 6.02a), *SADABS* (Version 2.03) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Huang, W. & Qian, H. F. (2007*a*). *J. Mol. Struct.* **832**, 108–106.
Huang, W. & Qian, H. F. (2007*b*). *J. Mol. Struct.* In the press. doi:10.1016/j.molstruc.2007.03.038.

supporting information

Acta Cryst. (2008). E**64**, m79 [https://doi.org/10.1107/S1600536807062836]

Bis(4′-chloro-2,2′:6′,2″-terpyridine-κ^3N,N',N'')iron(II) dinitrate dihydrate

Wei You, Xue-Yun Yang, Cheng Yao and Wei Huang

S1. Comment

We have newly reported the hydrochlorate tetrafluoroborate and hydrochlorate hexafluorophosphorate of 4′-chloro-2,2′:6′,2″-terpyridine (Huang & Qian, 2007*a*), and Ru(II), Cu(II), Zn(II), Ni(II) and Fe(II) complexes of 4′-chloro-2,2′:6′,2″-terpyridine with the metal/ligand ratios of 1:1 and 1:2. In this paper, we report the structure of a ferrous nitrate complex bearing the same 4′-chloro-2,2′:6′,2″-terpyridine ligand with the 1:2 metal/ligand ratio.

The atom-numbering scheme of the title compound (I) is shown in Fig. 1, while selected bond distances and bond angles are given in Table 1. The iron(II) center displays a six-coordinate octahedral configuration where each 4′-chloro-2,2′:6′,2′-terpyridine molecule serves as a 3 N tridentate ligand. The six Fe—N bond lengths fall with the normal ranges of 1.880 (3)—1.975 (2) Å (Huang & Qian, 2007*b*), where the two central Fe—N bond lengths are somewhat shorter than the side ones. The two terpyridine ligands are planar and the dihedral angle between them is 92.4 (1)°. In addition, O—H···O hydrogen bonds are observed between the hydrogen atoms of water molecule and the oxygen atoms of nitrate anions (Table 2).

S2. Experimental

The treatment of $Fe(NO_3)_2.6H_2O$ (0.072 g, 0.25 mmol) and 4′-chloro-2,2′:6′,2′-terpyridine (0.134 g, 0.50 mmol) in 30 cm^3 me thanol under reflux condition for 1 h produced deep red microcrystals in a yield of 83% (0.156 g). Anal. Calcd. for $FeC_{30}H_{24}N_8Cl_2O_8$: C: 47.96, H: 3.22, N: 14.91. Found: C: 47.88; H: 3.24; N: 14.98. Main FT—IR (KBr pellets, cm^{-1}): 3447 (*s*), 1663 (*s*), 1560 (*s*), 1545 (*s*), 1466 (*m*) and 1368 (*s*). Single crystals of the title complex suitable for X-ray diffraction measurement were obtained from the mixture of ethanol and water solutions at a ratio of 3:1 by slow evaporation in air at ambient temperature.

S3. Refinement

The two H atoms bonded to the water oxygen atom were located in the difference synthesis and were refined isotropically, whereas the other H atoms were placed in geometrically idealized positions (C—H = 0.93 Å) and refined as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

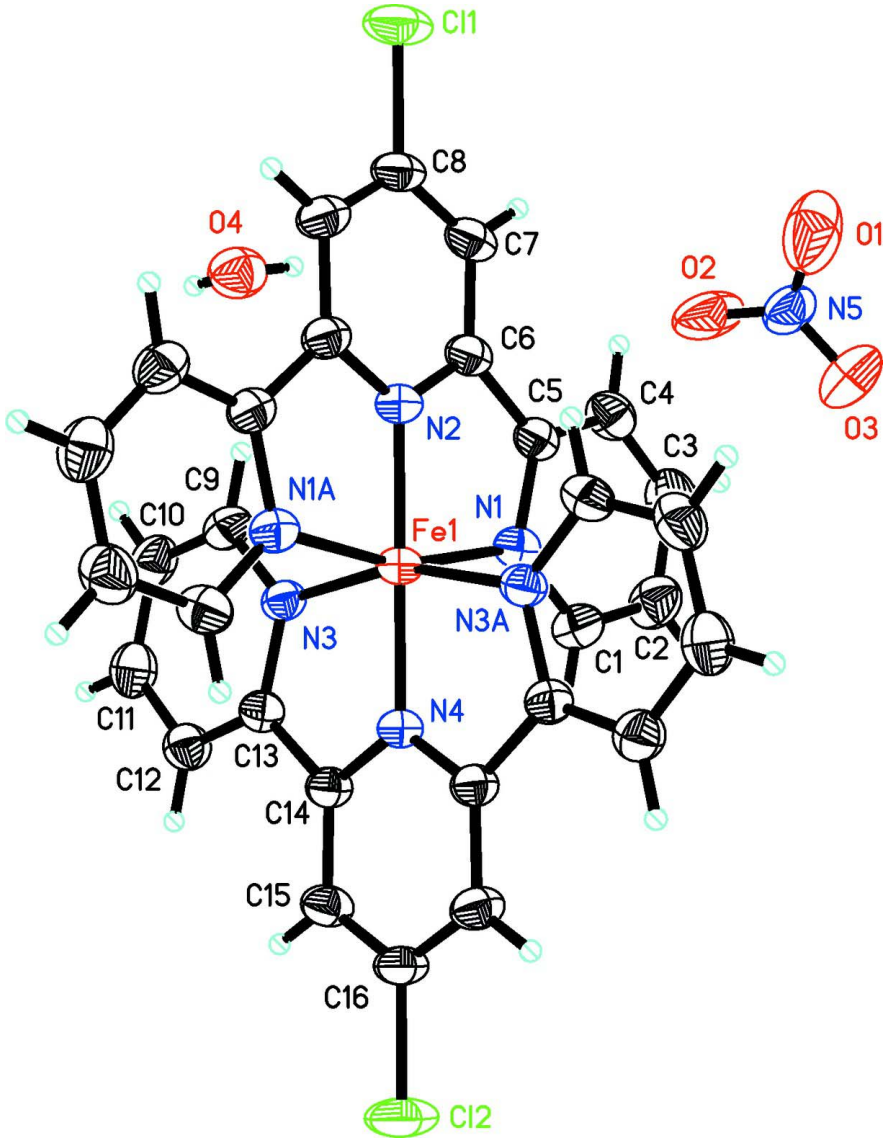

Figure 1

A drawing of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Bis(4′-chloro-2,2′:6′,2″-terpyridine-κ^3N,N',N'')iron(II) dinitrate dihydrate

Crystal data

$[Fe(C_{15}H_{10}ClN_3)_2](NO_3)_2\cdot 2H_2O$
$M_r = 751.32$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 18.049\ (2)$ Å
$b = 18.255\ (3)$ Å
$c = 10.0741\ (14)$ Å
$\beta = 102.668\ (2)°$
$V = 3238.5\ (8)$ Å^3
$Z = 4$
$F(000) = 1536$
$D_x = 1.541$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2481 reflections
$\theta = 2.3–25.0°$
$\mu = 0.70$ mm^{-1}
$T = 291$ K
Block, red
0.12 × 0.10 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.922, T_{max} = 0.931
8547 measured reflections
3192 independent reflections
2120 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.0°, θ_{min} = 1.6°
h = −21→22
k = −15→22
l = −12→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 0.97
3192 reflections
233 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Special details

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Fe1	1.0000	0.25347 (2)	0.2500	0.04349 (18)
C1	0.89844 (14)	0.28704 (15)	0.4385 (3)	0.0581 (7)
H1	0.9074	0.3364	0.4250	0.070*
C2	0.85298 (17)	0.26824 (18)	0.5259 (3)	0.0733 (9)
H2	0.8330	0.3045	0.5727	0.088*
C3	0.83702 (17)	0.19608 (19)	0.5445 (3)	0.0737 (9)
H3	0.8058	0.1829	0.6027	0.088*
C4	0.86806 (14)	0.14357 (16)	0.4753 (3)	0.0609 (7)
H4	0.8569	0.0943	0.4844	0.073*
C5	0.91552 (13)	0.16424 (13)	0.3929 (2)	0.0484 (6)
C6	0.95684 (13)	0.11401 (13)	0.3214 (2)	0.0490 (6)
C7	0.95566 (15)	0.03835 (13)	0.3241 (3)	0.0566 (7)
H7	0.9262	0.0130	0.3739	0.068*
C8	1.0000	0.00191 (19)	0.2500	0.0569 (10)
C9	0.87358 (14)	0.22036 (15)	0.0110 (3)	0.0552 (7)

H9	0.8832	0.1710	0.0305	0.066*
C10	0.81690 (16)	0.23907 (17)	−0.0981 (3)	0.0652 (8)
H10	0.7888	0.2028	−0.1514	0.078*
C11	0.80185 (16)	0.31174 (18)	−0.1283 (3)	0.0674 (8)
H11	0.7634	0.3251	−0.2019	0.081*
C12	0.84469 (14)	0.36479 (15)	−0.0478 (3)	0.0582 (7)
H12	0.8358	0.4143	−0.0666	0.070*
C13	0.90054 (12)	0.34274 (13)	0.0603 (2)	0.0459 (6)
C14	0.94967 (13)	0.39307 (13)	0.1540 (2)	0.0461 (6)
C15	0.94826 (14)	0.46877 (13)	0.1511 (3)	0.0554 (7)
H15	0.9137	0.4941	0.0849	0.066*
C16	1.0000	0.50551 (19)	0.2500	0.0563 (9)
Cl1	1.0000	−0.09281 (5)	0.2500	0.0883 (4)
Cl2	1.0000	0.60025 (6)	0.2500	0.0908 (4)
H4A	0.8087 (18)	0.0502 (17)	−0.052 (3)	0.069 (11)*
H4B	0.809 (3)	0.036 (3)	0.071 (5)	0.16 (2)*
N1	0.93034 (11)	0.23672 (10)	0.3718 (2)	0.0483 (5)
N2	1.0000	0.15050 (14)	0.2500	0.0450 (7)
N3	0.91592 (11)	0.27102 (10)	0.0912 (2)	0.0462 (5)
N4	1.0000	0.35651 (14)	0.2500	0.0421 (6)
N5	0.72096 (17)	0.05138 (16)	0.6869 (3)	0.0819 (8)
O1	0.7680 (2)	0.0029 (2)	0.7212 (3)	0.1692 (17)
O2	0.6813 (2)	0.05091 (14)	0.5761 (4)	0.1604 (16)
O3	0.71662 (18)	0.09870 (17)	0.7664 (3)	0.1287 (11)
O4	0.84123 (15)	0.04554 (14)	0.0124 (3)	0.0804 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0474 (3)	0.0321 (3)	0.0510 (3)	0.000	0.0108 (2)	0.000
C1	0.0563 (16)	0.0513 (16)	0.0695 (18)	−0.0025 (13)	0.0201 (14)	−0.0072 (14)
C2	0.075 (2)	0.075 (2)	0.078 (2)	−0.0025 (17)	0.0355 (17)	−0.0120 (17)
C3	0.0697 (19)	0.087 (2)	0.072 (2)	−0.0072 (17)	0.0318 (16)	0.0073 (18)
C4	0.0575 (16)	0.0599 (17)	0.0656 (17)	−0.0099 (14)	0.0140 (14)	0.0088 (15)
C5	0.0479 (14)	0.0449 (14)	0.0500 (14)	−0.0026 (12)	0.0059 (11)	0.0047 (12)
C6	0.0509 (14)	0.0398 (14)	0.0520 (15)	−0.0021 (12)	0.0020 (12)	0.0048 (12)
C7	0.0576 (16)	0.0429 (15)	0.0612 (17)	−0.0068 (13)	−0.0043 (13)	0.0098 (13)
C8	0.062 (2)	0.0336 (19)	0.063 (2)	0.000	−0.013 (2)	0.000
C9	0.0576 (16)	0.0470 (14)	0.0608 (17)	−0.0072 (12)	0.0128 (13)	−0.0106 (13)
C10	0.0645 (18)	0.071 (2)	0.0578 (17)	−0.0130 (16)	0.0088 (14)	−0.0152 (15)
C11	0.0615 (17)	0.082 (2)	0.0537 (17)	−0.0016 (16)	0.0021 (13)	−0.0035 (16)
C12	0.0598 (16)	0.0551 (16)	0.0566 (16)	0.0010 (13)	0.0058 (13)	0.0016 (13)
C13	0.0450 (13)	0.0427 (14)	0.0497 (14)	0.0014 (11)	0.0094 (11)	0.0014 (11)
C14	0.0468 (14)	0.0388 (13)	0.0506 (14)	0.0008 (11)	0.0060 (11)	0.0008 (11)
C15	0.0576 (16)	0.0406 (14)	0.0635 (17)	0.0034 (12)	0.0035 (13)	0.0068 (12)
C16	0.066 (2)	0.0329 (18)	0.068 (2)	0.000	0.011 (2)	0.000
Cl1	0.1096 (9)	0.0342 (5)	0.1052 (9)	0.000	−0.0113 (7)	0.000
Cl2	0.1091 (9)	0.0332 (5)	0.1170 (10)	0.000	−0.0033 (8)	0.000

N1	0.0486 (12)	0.0416 (12)	0.0539 (12)	0.0015 (9)	0.0095 (10)	0.0020 (9)
N2	0.0479 (16)	0.0347 (15)	0.0522 (17)	0.000	0.0107 (13)	0.000
N3	0.0512 (12)	0.0390 (11)	0.0501 (12)	−0.0044 (9)	0.0147 (9)	−0.0042 (9)
N4	0.0444 (15)	0.0335 (14)	0.0480 (16)	0.000	0.0096 (13)	0.000
N5	0.083 (2)	0.0703 (19)	0.082 (2)	−0.0056 (15)	−0.0041 (17)	−0.0227 (16)
O1	0.159 (3)	0.232 (5)	0.097 (2)	0.093 (3)	−0.015 (2)	−0.032 (2)
O2	0.204 (3)	0.0771 (18)	0.142 (3)	0.033 (2)	−0.089 (3)	−0.0424 (18)
O3	0.154 (3)	0.114 (2)	0.116 (2)	−0.015 (2)	0.025 (2)	−0.0545 (19)
O4	0.0779 (16)	0.0696 (15)	0.0858 (18)	0.0044 (12)	0.0011 (16)	−0.0029 (13)

Geometric parameters (Å, º)

Fe1—N2	1.880 (3)	C9—C10	1.371 (4)
Fe1—N4	1.881 (3)	C9—H9	0.9300
Fe1—N1	1.964 (2)	C10—C11	1.375 (4)
Fe1—N1^{i}	1.964 (2)	C10—H10	0.9300
Fe1—N3	1.975 (2)	C11—C12	1.385 (4)
Fe1—N3^{i}	1.975 (2)	C11—H11	0.9300
C1—N1	1.340 (3)	C12—C13	1.372 (3)
C1—C2	1.373 (4)	C12—H12	0.9300
C1—H1	0.9300	C13—N3	1.360 (3)
C2—C3	1.370 (4)	C13—C14	1.468 (3)
C2—H2	0.9300	C14—N4	1.349 (3)
C3—C4	1.374 (4)	C14—C15	1.382 (3)
C3—H3	0.9300	C15—C16	1.381 (3)
C4—C5	1.370 (3)	C15—H15	0.9300
C4—H4	0.9300	C16—C15^{i}	1.381 (3)
C5—N1	1.375 (3)	C16—Cl2	1.729 (4)
C5—C6	1.467 (3)	N2—C6^{i}	1.346 (3)
C6—N2	1.346 (3)	N4—C14^{i}	1.349 (3)
C6—C7	1.382 (3)	N5—O2	1.187 (3)
C7—C8	1.379 (3)	N5—O3	1.193 (3)
C7—H7	0.9300	N5—O1	1.223 (4)
C8—C7^{i}	1.379 (3)	O4—H4A	0.78 (3)
C8—Cl1	1.729 (4)	O4—H4B	0.93 (5)
C9—N3	1.349 (3)		

N2—Fe1—N4	180.000 (1)	N3—C9—H9	118.8
N2—Fe1—N1	81.04 (6)	C10—C9—H9	118.8
N4—Fe1—N1	98.96 (6)	C9—C10—C11	119.6 (3)
N2—Fe1—N1^{i}	81.04 (6)	C9—C10—H10	120.2
N4—Fe1—N1^{i}	98.96 (6)	C11—C10—H10	120.2
N1—Fe1—N1^{i}	162.09 (11)	C10—C11—C12	119.1 (3)
N2—Fe1—N3	99.34 (6)	C10—C11—H11	120.4
N4—Fe1—N3	80.66 (6)	C12—C11—H11	120.4
N1—Fe1—N3	92.75 (8)	C13—C12—C11	118.6 (3)
N1^{i}—Fe1—N3	90.14 (8)	C13—C12—H12	120.7
N2—Fe1—N3^{i}	99.34 (6)	C11—C12—H12	120.7

N4—Fe1—N3^{i}	80.66 (6)	N3—C13—C12	122.8 (2)
N1—Fe1—N3^{i}	90.14 (8)	N3—C13—C14	113.0 (2)
N1^{i}—Fe1—N3^{i}	92.75 (8)	C12—C13—C14	124.2 (2)
N3—Fe1—N3^{i}	161.33 (11)	N4—C14—C15	121.1 (2)
N1—C1—C2	122.2 (3)	N4—C14—C13	111.6 (2)
N1—C1—H1	118.9	C15—C14—C13	127.3 (2)
C2—C1—H1	118.9	C16—C15—C14	117.6 (2)
C3—C2—C1	120.1 (3)	C16—C15—H15	121.2
C3—C2—H2	120.0	C14—C15—H15	121.2
C1—C2—H2	120.0	C15—C16—C15^{i}	121.9 (3)
C2—C3—C4	118.7 (3)	C15—C16—Cl2	119.06 (16)
C2—C3—H3	120.6	C15^{i}—C16—Cl2	119.06 (16)
C4—C3—H3	120.6	C1—N1—C5	117.6 (2)
C5—C4—C3	119.6 (3)	C1—N1—Fe1	127.63 (18)
C5—C4—H4	120.2	C5—N1—Fe1	114.76 (16)
C3—C4—H4	120.2	C6^{i}—N2—C6	120.7 (3)
C4—C5—N1	121.8 (2)	C6^{i}—N2—Fe1	119.65 (14)
C4—C5—C6	125.3 (2)	C6—N2—Fe1	119.65 (14)
N1—C5—C6	112.9 (2)	C9—N3—C13	117.5 (2)
N2—C6—C7	121.2 (2)	C9—N3—Fe1	127.40 (18)
N2—C6—C5	111.7 (2)	C13—N3—Fe1	115.06 (15)
C7—C6—C5	127.1 (2)	C14^{i}—N4—C14	120.7 (3)
C8—C7—C6	117.3 (3)	C14^{i}—N4—Fe1	119.65 (13)
C8—C7—H7	121.4	C14—N4—Fe1	119.65 (13)
C6—C7—H7	121.4	O2—N5—O3	121.5 (4)
C7—C8—C7^{i}	122.3 (3)	O2—N5—O1	119.9 (3)
C7—C8—Cl1	118.84 (17)	O3—N5—O1	118.6 (3)
C7^{i}—C8—Cl1	118.84 (17)	H4A—O4—H4B	95 (3)
N3—C9—C10	122.3 (3)		

Symmetry code: (i) $-x+2, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*B*···O1^{ii}	0.93 (5)	1.96 (5)	2.859 (5)	162 (4)
O4—H4*A*···O3^{iii}	0.78 (3)	2.36 (3)	3.114 (4)	164 (3)
O4—H4*A*···O1^{iii}	0.78 (3)	2.40 (3)	3.042 (4)	140 (3)

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