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Crystal structure of ({(1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine}chloridoiron(III))-μ-oxido-[trichloridoferrate(III)] chloroform monosolvate

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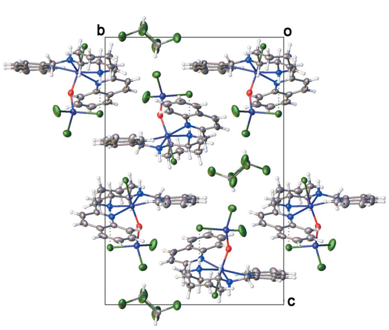
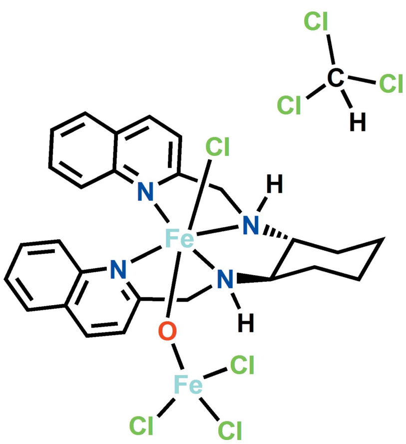
The first Fe^{III} atom in the solvated title compound, $[Fe_2Cl_4O(C_{26}H_{28}N_4)]\cdot CHCl_3$, adopts a distorted six-coordinate octahedral geometry. It is coordinated by one chloride ligand, four N atoms from the (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)-methyl]cyclohexane-1,2-diamine ligand, and a bridging oxido ligand attached to the second Fe^{III} atom, which is also bonded to three chloride ions. A very weak intramolecular N—H···Cl hydrogen bond occurs. In the crystal, the coordination complexes stack in columns, and a grouping of six such columns create channels, which are populated by disordered chloroform solvent molecules. Although the Fe—Cl bond lengths for the two metal atoms are comparable to the mean Fe—Cl bond lengths as derived from the Cambridge Structural Database, the Fe—O bond lengths are notably shorter. The solvent chloroform molecule exhibits ‘flip’ disorder of the C—H moiety in a 0.544 (3):0.456 (3) ratio. The only directional interaction noted is a weak C—H···Cl hydrogen bond.

1. Chemical context

Developing small-molecule complexes incorporating iron is an area of growing interest since the discovery of non-heme iron enzymes such as methane monooxygenase and Rieske dioxygenases to be efficient catalysts in the selective oxidation of hydrocarbons under mild reaction conditions (Company *et al.*, 2007). Studies show that highly active non-heme iron catalysts that facilitate efficient stereo-specific alkane hydroxylation using H_2O_2 as oxidant can be synthesized by employing tetradentate N_4-donor ligands such as *N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)ethane-1,2-diamine (BPMEN) or *tris*(2-pyridylmethyl)amine (TPMA) (Costas *et al.*, 2000). These catalysts have provided key insights into possible mechanisms used by enzymes to oxidize alkanes in nature (Meunier *et al.*, 2004). In addition to the application of four-coordinate iron complexes as catalysts in hydroxylation reactions, studies also show that these complexes can be utilized in epoxidation reactions of terminal and electron-deficient alkenes (Dubois *et al.*, 2003). Iron oxido-bridging complexes are reported to play an important role in oxygen transport (hemerythrin), phosphate ester hydrolysis (purple acid phosphates), or DNA synthesis (ribonucleotide reductase) (Dutta *et al.*, 1996). These oxido complexes exhibit redox and magnetic properties making them excellent candidates for future investigations into the mechanisms behind important



biological and chemical processes (Feig & Lippard, 1994). Given the significance and application of iron complexes made from tetradentate ligands, herein we report on the synthesis and crystal structure of the solvated title compound $[Fe_2(C_{26}H_{28}N_4)(Cl)(\mu\text{-}O)Fe(Cl)_3]\cdot CHCl_3$ (**1**), incorporating (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine (Fig. 1).



2. Structural commentary

There is one coordination complex and one molecule of chloroform solvent in the asymmetric unit. The coordination complex features two Fe(III) metal cations. One of the metal cations, Fe1, assumes a distorted octahedral coordination (Table 1). The tetradentate ligand, (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine, interacts with the Fe^{III} cation in the equatorial plane through the four amine groups. A chloride ion and a bridging oxido ligand, which connects the two metal cations, complete the axial coordination. The distortions from the ideal octahedral geometry occur both in the equatorial and the axial positions. The equatorial angles vary widely from 74.96 (9)°, as in the case of the N1—Fe1—N2 angle, to 133.98 (9)° for the untethered N1—Fe1—N4 angle. The axial ligands exhibit a bent conformation with a Cl1—Fe1—O1 angle of 166.06 (7)°. In contrast, the second Fe metal cation, Fe2, exhibits a near ideal tetrahedral coordination geometry composed of one O atom and three Cl atoms. As expected based on the difference in the saturation of the coordination sphere, the Fe—Cl and the Fe—O distances for Fe1 are longer than that for Fe2. The single Fe—Cl distance for Fe1 is 2.3560 (8) Å, whereas the average Fe2—Cl distance of 2.232 (9) Å is more than 0.1 Å shorter, a statistically significant variation. Similarly, the Fe—O distances for Fe1 and Fe2 are also statistically significantly different at 1.808 (2) Å and 1.756 (2) Å, respectively. The bond lengths in the title compound are comparable to the mean Fe—Cl distances from the CSD for Fe complexes in an octahedral coordination [2.33 (7) Å] and a tetrahedral coordination [2.23 (3) Å]. In contrast, the Fe—O distances for both the octahedral and tetrahedral configurations in the title compound are shorter than the mean distances from CSD [2.01 (9) and 1.87 (13) Å, respectively]. A very weak intramolecular N3—H3···Cl4 hydrogen bond (Table 2) occurs. Finally, we observe that complex (**1**) is present only as the *M* (left-handed) conformer.

Table 1
Selected bond lengths (Å).

Fe1—O1	1.808 (2)	Fe1—Cl1	2.3560 (8)
Fe1—N1	2.243 (2)	Fe2—O1	1.756 (2)
Fe1—N2	2.172 (3)	Fe2—Cl2	2.2194 (9)
Fe1—N3	2.159 (2)	Fe2—Cl3	2.2331 (10)
Fe1—N4	2.223 (2)	Fe2—Cl4	2.2432 (9)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···Cl4	0.80 (4)	2.60 (4)	3.378 (3)	167 (3)
C27*A*—H27*A*···Cl2^{i}	1.00	2.41	3.30 (2)	149

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

3. Supramolecular features

The molecules in the crystal structure are related by twofold screw axes running along the *a*-, *b*-, and *c*-axis directions. As there are no additional symmetry elements present, the resulting space group, $P2_12_12_1$, is chiral. The absolute structure was unequivocally established, as evidenced by a Hooft *y* parameter of 0.003 (6), using anomalous dispersion. Apart from a weak C—H···Cl bond from the chloroform molecule to one of the chloride ions bonded to Fe2 (Table 2), the molecules of the coordination complexes display minimal interatomic interactions. They assemble into columns that run parallel to the *a* axis. A circular arrangement of six columns of coordination complex molecules creates a channel. The

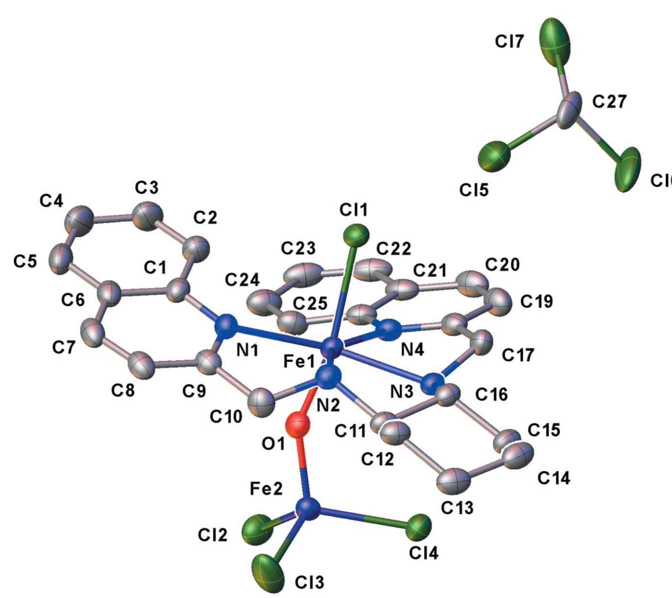


Figure 1
The molecular structure of complex (**1**), shown with 50% probability displacement ellipsoids. All H atoms and the minor-disorder components of the solvent molecule have been omitted for clarity.

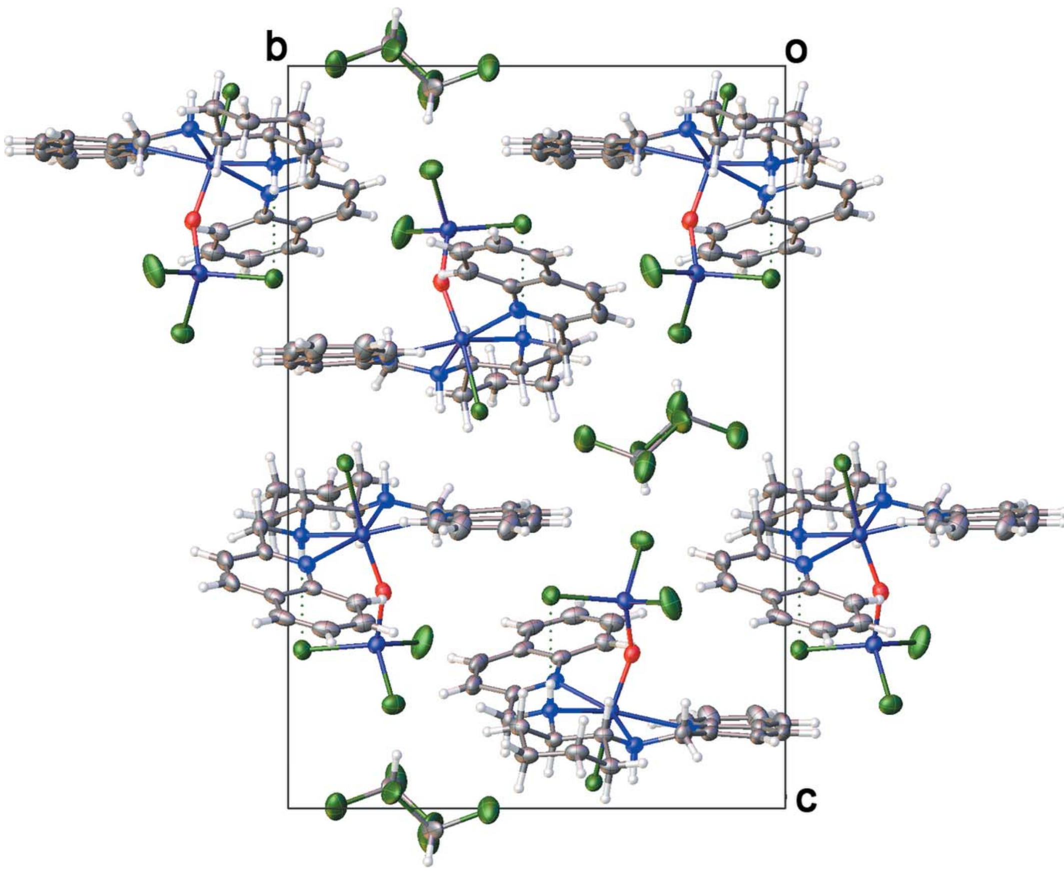


Figure 2
Packing diagram for complex (**1**), showing the columns of the coordination complex and the channels of disordered chloroform solvent molecules stacked along the *a* axis.

channel is filled with solvent chloroform molecules that exhibit extensive positional disorder. For two of the columns that frame the chloroform channels, the oxido-trichloride groups of the coordination complexes point into the channels, while the other four columns face the void with the (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine ligand. The packing is illustrated in Fig. 2.

4. Database survey

In our survey of the Cambridge Structural Database (Groom *et al.*, 2016), we found five reported structures incorporating the (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine ligand motif. Of the five, only one structure showed coordination to iron (Dengler *et al.*, 2011). In that structure, the distorted octahedral coordination of the Fe^{III} metal atom is completed by two chloride ligands in the axial positions. The two Fe—Cl distances are comparable (2.495 and 2.509 Å) and the Cl1—Fe—N angles show a narrow distribution from 92–94°, except for Cl1—Fe—N1, which is 84°.

5. Synthesis and crystallization

Synthesis of (1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine (R-QMC): In a 50 mL round-bottom flask (1*R*,2*R*)-1,2-cyclohexanediamine (0.20 g, 1.8 mmol) and 2-quinolinecarboxaldehyde (0.55 g, 3.6 mmol) were refluxed in ethanol (10 mL) for 3 h. A yellow precipitate formed that was isolated by filtration, washed twice with ethanol, and dried *in vacuo* producing the unreduced form of the ligand (QMC), (0.63 g, 89% yield). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.24 (*br*, 2 H, CH), 1.56 (*br*, 2 H, CH), 1.97 (*br*, 2 H, CH), 3.62 (*br*, 2 H, CH), 7.48 (*t*, 1 H, *J* = 8.06 Hz, CH), 7.65 (*t*, 1 H, *J* = 8.06 Hz, CH), 7.74 (*d*, 1 H, *J* = 8.06 Hz, CH), 8.03 (*d*, 1 H, *J* = 8.56 Hz, CH), 8.06 (*s*, 1 H, CH), and 8.52 (*s*, 1 H, CH). The reduced form of the ligand (R-QMC, Fig. 3) was synthesized by reacting ligand QMC (0.50 g, 1.3 mmol) with sodium borohydride (0.06 g, 1.5 mmol) in methanol at room temperature for 12 h to produce R-QMC (0.42 g, 82% yield). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.24 (*br*, 2 H, CH), 1.56 (*br*, 2 H, CH), 1.97 (*br*, 2 H, CH), 3.62 (*br*, 2 H, CH), 4.22 (*dd*, 2 H, *J* = 8.06 Hz, CH), 7.55 (*t*, 1 H, *J* = 8.06 Hz, CH), 7.61–7.73 (*m*, CH), 7.81 (*d*, 1 H, *J* = 8.56 Hz, CH), 8.06 (*d*, 1 H *J* = 8.06 Hz, CH), 8.08 (*d*, 1 H *J* = 8.06 Hz, CH).

Synthesis of ({(1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]-cyclohexane-1,2-diamine}chloridoiron(III))-*μ*-oxido-[trichloridoferrate(III)] chloroform monosolvate R-QMC (0.25 g, 0.63 mmol) was dissolved in 50/50 mixture of dichloromethane and ethanol (20 mL) in a 50 mL round-bottom flask. Iron(II) chloride (0.08 g, 0.63 mmol) was added to the flask to give a brown-colored solution. The reaction was allowed to mix for 6 h under gentle heat producing a brown-colored precipitate. The precipitate was filtered and washed twice with cold solvent then dried under vacuum for 30 minutes producing a brown powder (0.19 g, 58%). Brown prisms of (**1**) suitable for X-ray analysis were obtained by slow solvent diffusion of diethyl ether into a concentrated complex solution made in chloroform.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All hydrogen atoms, except for the amine hydrogen atom bonded to N3, were added at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The amine hydrogen bonded to N3 was allowed to refine freely. In addition to the {[(1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]-cyclohexane-1,2-diamine]chloridoiron(III)}-μ-oxido-[trichloridoferrate(III)], there is one molecule of chloroform solvent in the asymmetric unit. The solvent molecule exhibits extensive positional disorder over three positions. Initially, the disorder was modeled with chloroform molecule in an idealized geometry, where the 1,2 and the 1,3 bond lengths were constrained. As the refinement converged, the geometry constraints were lifted. The chlorine atoms Cl6 and Cl7 were modeled over two positions, with the major component contributing 54.4 (3)%. The carbon atom C27 required modeling over three positions with the major component contribution of 54.4 (3)% and the two minor components contributing 24.1 (4)% and 21.5 (4)%. The C–Cl distances for all of the disorder components were restrained to be similar. In addition, Cl6*A*–Cl7*A* and Cl7*A*–Cl5 were restrained to be similar. The absolute structure was unequivocally determined by anomalous dispersion.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Fe_2Cl_4O(C_{26}H_{28}N_4)]\cdot CHCl_3$
M_r	785.39
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	120
a, b, c (Å)	10.3489 (6), 14.3664 (8), 21.4619 (13)
V (Å^3)	3190.9 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.53
Crystal size (mm)	0.26 × 0.22 × 0.14
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.658, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	79886, 10537, 8808
R_{int}	0.065
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.736
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.082, 1.05
No. of reflections	10537
No. of parameters	413
No. of restraints	37
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.67, −0.52
Absolute structure parameter	0.000 (14)

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

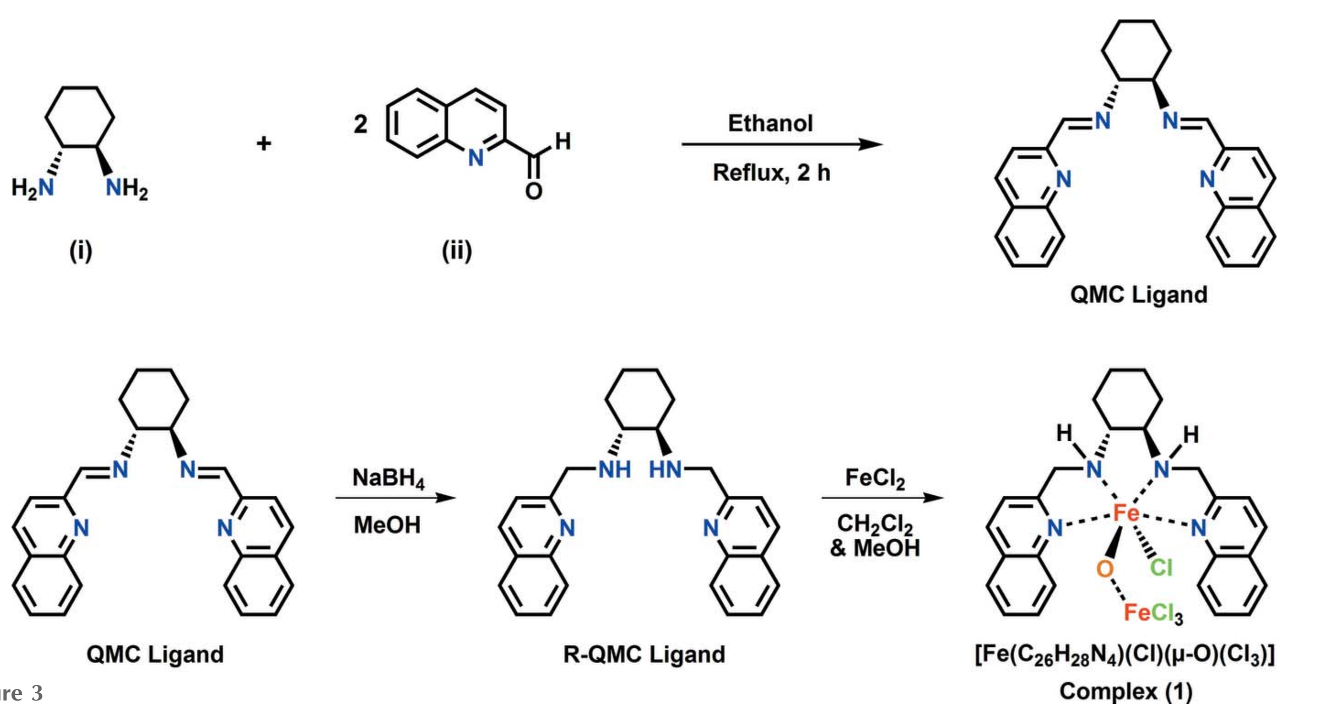


Figure 3
Synthetic scheme for complex (**1**).

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supporting information

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Crystal structure of ({(1*R*,2*R*)-*N*,*N*′-bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine}chloridoiron(III))-*μ*-oxido-[trichloridoferrate(III)] chloroform monosolvate

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Computing details

Data collection: *APEX2* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

({(1*R*,2*R*)-*N*[1],*N*[2]-Bis[(quinolin-2-yl)methyl]cyclohexane-1,2-diamine}chloridoiron(III))-*μ*-oxido-[trichloridoferrate(III)] chloroform monosolvate

Crystal data

$[Fe_2Cl_4O(C_{26}H_{28}N_4)]\cdot CHCl_3$
$M_r = 785.39$
Orthorhombic, $P2_12_12_1$
$a = 10.3489$ (6) Å
$b = 14.3664$ (8) Å
$c = 21.4619$ (13) Å
$V = 3190.9$ (3) Å^3
$Z = 4$
$F(000) = 1592$
$D_x = 1.635$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9981 reflections
$\theta = 2.7$–$30.8°$
$\mu = 1.53$ mm^{-1}
$T = 120$ K
Prism, brown
0.26 × 0.22 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.658$, $T_{max} = 0.746$
79886 measured reflections
10537 independent reflections
8808 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 31.6°$, $\theta_{min} = 2.4°$
$h = -14 \rightarrow 15$
$k = -20 \rightarrow 20$
$l = -30 \rightarrow 31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.082$
$S = 1.05$
10537 reflections
413 parameters
37 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 0.5065P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.000 (14)

Special details

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

Refinement. Refined as a 2-component inversion twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Fe1	0.24940 (4)	0.34973 (3)	0.86909 (2)	0.02294 (9)	
Fe2	0.37047 (4)	0.32209 (3)	0.72075 (2)	0.02735 (10)	
Cl1	0.15040 (7)	0.38505 (5)	0.96507 (4)	0.03021 (16)	
Cl2	0.24573 (9)	0.28798 (6)	0.63979 (4)	0.04158 (19)	
Cl3	0.54491 (9)	0.23071 (7)	0.72381 (7)	0.0617 (3)	
Cl4	0.43247 (8)	0.47130 (5)	0.71335 (4)	0.03348 (16)	
O1	0.28867 (19)	0.30834 (14)	0.79183 (10)	0.0295 (5)	
N1	0.2138 (2)	0.19951 (16)	0.89191 (12)	0.0257 (5)	
N2	0.4247 (2)	0.30306 (17)	0.91515 (12)	0.0276 (5)	
H2	0.4088	0.3066	0.9611	0.033*	
N3	0.3688 (2)	0.47300 (16)	0.86772 (13)	0.0244 (5)	
H3	0.397 (3)	0.472 (2)	0.8331 (18)	0.029*	
N4	0.1286 (2)	0.46258 (17)	0.82871 (12)	0.0259 (5)	
C1	0.0979 (3)	0.1519 (2)	0.88928 (14)	0.0279 (6)	
C2	−0.0189 (3)	0.2008 (2)	0.88477 (17)	0.0357 (7)	
H2A	−0.0188	0.2669	0.8860	0.043*	
C3	−0.1336 (3)	0.1535 (3)	0.8786 (2)	0.0474 (9)	
H3A	−0.2119	0.1875	0.8746	0.057*	
C4	−0.1369 (4)	0.0557 (3)	0.8781 (2)	0.0492 (10)	
H4	−0.2167	0.0239	0.8733	0.059*	
C5	−0.0246 (4)	0.0067 (2)	0.88457 (17)	0.0415 (9)	
H5	−0.0269	−0.0594	0.8849	0.050*	
C6	0.0945 (3)	0.0530 (2)	0.89077 (15)	0.0319 (7)	
C7	0.2130 (4)	0.0055 (2)	0.89797 (15)	0.0339 (7)	
H7	0.2143	−0.0606	0.8990	0.041*	
C8	0.3249 (3)	0.0539 (2)	0.90334 (15)	0.0318 (7)	
H8	0.4042	0.0221	0.9101	0.038*	
C9	0.3225 (3)	0.1519 (2)	0.89887 (14)	0.0273 (6)	
C10	0.4472 (3)	0.2049 (2)	0.89977 (17)	0.0320 (7)	
H10A	0.5061	0.1770	0.9310	0.038*	
H10B	0.4892	0.2004	0.8584	0.038*	
C11	0.5348 (3)	0.3655 (2)	0.90109 (15)	0.0266 (6)	
H11	0.5616	0.3551	0.8569	0.032*	
C12	0.6531 (3)	0.3515 (2)	0.94307 (15)	0.0314 (6)	

H12A	0.6286	0.3619	0.9871	0.038*	
H12B	0.6848	0.2867	0.9390	0.038*	
C13	0.7603 (3)	0.4192 (2)	0.92475 (16)	0.0367 (7)	
H13A	0.7889	0.4058	0.8817	0.044*	
H13B	0.8353	0.4106	0.9528	0.044*	
C14	0.7134 (3)	0.5200 (3)	0.92877 (16)	0.0369 (8)	
H14A	0.6956	0.5360	0.9728	0.044*	
H14B	0.7822	0.5622	0.9135	0.044*	
C15	0.5912 (3)	0.5347 (2)	0.89019 (16)	0.0316 (7)	
H15A	0.5587	0.5988	0.8970	0.038*	
H15B	0.6123	0.5281	0.8454	0.038*	
C16	0.4860 (3)	0.4654 (2)	0.90736 (14)	0.0260 (6)	
H16	0.4605	0.4762	0.9517	0.031*	
C17	0.2910 (3)	0.5562 (2)	0.87921 (16)	0.0296 (6)	
H17A	0.3375	0.6120	0.8641	0.036*	
H17B	0.2756	0.5634	0.9245	0.036*	
C18	0.1644 (3)	0.5469 (2)	0.84567 (15)	0.0288 (6)	
C19	0.0884 (3)	0.6267 (2)	0.83377 (17)	0.0374 (8)	
H19	0.1182	0.6866	0.8458	0.045*	
C20	−0.0278 (4)	0.6169 (2)	0.80486 (17)	0.0390 (8)	
H20	−0.0808	0.6699	0.7977	0.047*	
C21	−0.0696 (3)	0.5276 (2)	0.78549 (15)	0.0327 (7)	
C22	−0.1905 (3)	0.5116 (3)	0.75714 (16)	0.0400 (9)	
H22	−0.2471	0.5624	0.7494	0.048*	
C23	−0.2267 (3)	0.4237 (3)	0.74077 (16)	0.0420 (9)	
H23	−0.3098	0.4132	0.7233	0.050*	
C24	−0.1422 (3)	0.3490 (3)	0.74965 (16)	0.0398 (8)	
H24	−0.1675	0.2883	0.7370	0.048*	
C25	−0.0227 (3)	0.3622 (2)	0.77650 (16)	0.0327 (7)	
H	0.0349	0.3112	0.7811	0.039*	
C26	0.0136 (3)	0.4510 (2)	0.79695 (14)	0.0276 (6)	
Cl5	0.04556 (12)	0.59674 (7)	1.00240 (6)	0.0613 (3)	
Cl6	0.0633 (4)	0.79088 (18)	0.97904 (15)	0.0776 (11)	0.544 (3)
Cl6A	−0.0063 (4)	0.7908 (4)	0.9720 (2)	0.0927 (17)	0.456 (3)
Cl7	−0.1718 (2)	0.71764 (14)	1.04018 (13)	0.0676 (7)	0.544 (3)
Cl7A	−0.0951 (2)	0.70539 (15)	1.08266 (12)	0.0531 (7)	0.456 (3)
C27	−0.0048 (11)	0.7090 (7)	1.0301 (6)	0.046 (4)	0.544 (3)
H27	0.0360	0.7186	1.0718	0.055*	0.544 (3)
C27A	0.030 (2)	0.7114 (11)	1.0304 (9)	0.027 (5)	0.241 (4)
H27A	0.1111	0.7305	1.0522	0.032*	0.241 (4)
C27B	−0.0529 (16)	0.6944 (8)	1.0038 (7)	0.042 (5)	0.215 (4)
H27B	−0.1340	0.6761	0.9816	0.050*	0.215 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02119 (17)	0.02163 (17)	0.0260 (2)	0.00049 (15)	0.00144 (18)	0.00237 (15)
Fe2	0.0243 (2)	0.0275 (2)	0.0302 (2)	0.00048 (16)	0.00332 (18)	−0.00318 (17)

Cl1	0.0337 (4)	0.0278 (3)	0.0291 (4)	0.0003 (3)	0.0073 (3)	0.0025 (3)
Cl2	0.0377 (4)	0.0501 (5)	0.0369 (4)	−0.0118 (4)	−0.0030 (4)	−0.0031 (3)
Cl3	0.0351 (4)	0.0454 (5)	0.1045 (9)	0.0137 (4)	0.0018 (5)	−0.0252 (6)
Cl4	0.0340 (4)	0.0311 (3)	0.0354 (4)	−0.0051 (3)	0.0032 (3)	0.0020 (3)
O1	0.0321 (11)	0.0242 (10)	0.0323 (12)	0.0011 (8)	0.0013 (9)	0.0037 (8)
N1	0.0286 (12)	0.0220 (11)	0.0265 (13)	0.0005 (9)	0.0003 (10)	0.0022 (9)
N2	0.0271 (12)	0.0298 (13)	0.0260 (13)	−0.0001 (10)	−0.0009 (10)	0.0025 (10)
N3	0.0245 (12)	0.0255 (11)	0.0231 (12)	−0.0012 (9)	0.0020 (11)	0.0015 (10)
N4	0.0223 (12)	0.0284 (12)	0.0271 (13)	0.0042 (10)	0.0030 (10)	0.0029 (10)
C1	0.0326 (15)	0.0273 (14)	0.0237 (14)	−0.0049 (11)	0.0017 (12)	0.0018 (11)
C2	0.0304 (16)	0.0346 (16)	0.042 (2)	−0.0018 (12)	0.0051 (14)	−0.0025 (14)
C3	0.0321 (17)	0.048 (2)	0.062 (3)	−0.0058 (15)	0.0095 (17)	−0.0116 (19)
C4	0.0396 (19)	0.048 (2)	0.060 (3)	−0.0161 (16)	0.0080 (19)	−0.0117 (18)
C5	0.056 (2)	0.0312 (16)	0.038 (2)	−0.0146 (15)	0.0046 (17)	−0.0048 (14)
C6	0.0449 (19)	0.0266 (14)	0.0242 (15)	−0.0042 (12)	0.0031 (14)	−0.0008 (12)
C7	0.054 (2)	0.0233 (14)	0.0246 (15)	0.0014 (13)	0.0005 (14)	0.0018 (12)
C8	0.0449 (19)	0.0279 (14)	0.0225 (15)	0.0067 (12)	−0.0027 (13)	0.0009 (12)
C9	0.0332 (15)	0.0276 (13)	0.0210 (14)	0.0043 (12)	−0.0020 (12)	0.0028 (11)
C10	0.0294 (15)	0.0298 (15)	0.0368 (18)	0.0054 (12)	−0.0052 (14)	0.0021 (13)
C11	0.0205 (13)	0.0353 (15)	0.0240 (15)	−0.0015 (11)	−0.0002 (11)	−0.0001 (12)
C12	0.0264 (14)	0.0416 (16)	0.0261 (15)	0.0003 (13)	0.0003 (12)	−0.0006 (13)
C13	0.0272 (15)	0.054 (2)	0.0284 (16)	−0.0016 (15)	−0.0033 (14)	0.0001 (14)
C14	0.0324 (16)	0.053 (2)	0.0256 (16)	−0.0133 (14)	−0.0024 (13)	−0.0007 (14)
C15	0.0283 (15)	0.0386 (16)	0.0277 (16)	−0.0086 (12)	0.0020 (13)	−0.0011 (13)
C16	0.0247 (14)	0.0310 (14)	0.0223 (15)	−0.0041 (11)	0.0006 (11)	0.0011 (11)
C17	0.0318 (15)	0.0230 (13)	0.0339 (18)	−0.0018 (11)	0.0038 (13)	0.0010 (12)
C18	0.0319 (15)	0.0270 (14)	0.0275 (16)	0.0040 (11)	0.0070 (13)	0.0058 (11)
C19	0.047 (2)	0.0276 (16)	0.0380 (19)	0.0091 (13)	0.0060 (16)	0.0046 (13)
C20	0.044 (2)	0.0367 (17)	0.0366 (19)	0.0175 (15)	0.0084 (15)	0.0111 (14)
C21	0.0302 (15)	0.0430 (17)	0.0250 (16)	0.0113 (13)	0.0077 (13)	0.0096 (14)
C22	0.0293 (16)	0.065 (2)	0.0262 (17)	0.0171 (16)	0.0047 (13)	0.0148 (16)
C23	0.0234 (17)	0.074 (3)	0.0284 (17)	0.0031 (15)	−0.0004 (13)	0.0066 (17)
C24	0.0303 (16)	0.057 (2)	0.0321 (18)	−0.0008 (16)	0.0002 (14)	−0.0039 (15)
C25	0.0257 (14)	0.0415 (17)	0.0308 (17)	0.0046 (12)	0.0004 (13)	−0.0028 (14)
C26	0.0237 (14)	0.0374 (15)	0.0216 (15)	0.0060 (11)	0.0036 (11)	0.0046 (12)
Cl5	0.0708 (7)	0.0439 (5)	0.0691 (7)	−0.0069 (5)	0.0295 (6)	0.0027 (5)
Cl6	0.143 (3)	0.0404 (12)	0.0489 (14)	−0.0417 (17)	0.011 (2)	0.0081 (10)
Cl6A	0.096 (3)	0.122 (3)	0.060 (2)	0.051 (3)	0.010 (2)	0.042 (2)
Cl7	0.0658 (13)	0.0416 (10)	0.0953 (19)	0.0037 (9)	−0.0259 (13)	−0.0139 (10)
Cl7A	0.0577 (14)	0.0441 (11)	0.0573 (15)	−0.0032 (9)	0.0153 (11)	−0.0198 (10)
C27	0.059 (9)	0.043 (5)	0.036 (6)	−0.033 (5)	−0.012 (5)	0.001 (4)
C27A	0.023 (9)	0.038 (10)	0.019 (9)	−0.011 (6)	−0.010 (6)	0.000 (7)
C27B	0.025 (8)	0.027 (8)	0.073 (15)	−0.010 (6)	−0.002 (8)	−0.012 (8)

Geometric parameters (Å, º)

Fe1—O1	1.808 (2)	C12—H12B	0.9900
Fe1—N1	2.243 (2)	C12—C13	1.527 (5)

Fe1—N2	2.172 (3)	C13—H13A	0.9900
Fe1—N3	2.159 (2)	C13—H13B	0.9900
Fe1—N4	2.223 (2)	C13—C14	1.530 (5)
Fe1—Cl1	2.3560 (8)	C14—H14A	0.9900
Fe2—O1	1.756 (2)	C14—H14B	0.9900
Fe2—Cl2	2.2194 (9)	C14—C15	1.526 (4)
Fe2—Cl3	2.2331 (10)	C15—H15A	0.9900
Fe2—Cl4	2.2432 (9)	C15—H15B	0.9900
N1—C1	1.382 (4)	C15—C16	1.521 (4)
N1—C9	1.324 (4)	C16—H16	1.0000
N2—H2	1.0000	C17—H17A	0.9900
N2—C10	1.467 (4)	C17—H17B	0.9900
N2—C11	1.481 (4)	C17—C18	1.501 (4)
N3—H3	0.80 (4)	C18—C19	1.414 (4)
N3—C16	1.486 (4)	C19—H19	0.9500
N3—C17	1.462 (4)	C19—C20	1.361 (5)
N4—C18	1.318 (4)	C20—H20	0.9500
N4—C26	1.381 (4)	C20—C21	1.417 (5)
C1—C2	1.401 (4)	C21—C22	1.411 (5)
C1—C6	1.422 (4)	C21—C26	1.418 (4)
C2—H2A	0.9500	C22—H22	0.9500
C2—C3	1.374 (5)	C22—C23	1.363 (6)
C3—H3A	0.9500	C23—H23	0.9500
C3—C4	1.405 (5)	C23—C24	1.398 (5)
C4—H4	0.9500	C24—H24	0.9500
C4—C5	1.366 (6)	C24—C25	1.378 (5)
C5—H5	0.9500	C25—H	0.9500
C5—C6	1.407 (5)	C25—C26	1.401 (5)
C6—C7	1.412 (5)	Cl5—C27	1.796 (11)
C7—H7	0.9500	Cl5—C27A	1.761 (17)
C7—C8	1.355 (5)	Cl5—C27B	1.733 (13)
C8—H8	0.9500	Cl6—C27	1.755 (10)
C8—C9	1.413 (4)	Cl6A—C27A	1.735 (17)
C9—C10	1.498 (4)	Cl6A—C27B	1.618 (12)
C10—H10A	0.9900	Cl7—C27	1.746 (11)
C10—H10B	0.9900	Cl7A—C27A	1.716 (17)
C11—H11	1.0000	Cl7A—C27B	1.755 (15)
C11—C12	1.534 (4)	C27—H27	1.0000
C11—C16	1.527 (4)	C27A—H27A	1.0000
C12—H12A	0.9900	C27B—H27B	1.0000

O1—Fe1—Cl1	166.06 (7)	C13—C12—C11	110.2 (3)
O1—Fe1—N1	85.45 (9)	C13—C12—H12A	109.6
O1—Fe1—N2	97.36 (10)	C13—C12—H12B	109.6
O1—Fe1—N3	97.40 (10)	C12—C13—H13A	109.4
O1—Fe1—N4	90.50 (9)	C12—C13—H13B	109.4
N1—Fe1—Cl1	86.84 (7)	C12—C13—C14	111.0 (3)
N2—Fe1—Cl1	91.81 (7)	H13A—C13—H13B	108.0

N2—Fe1—N1	74.96 (9)	C14—C13—H13A	109.4
N2—Fe1—N4	150.72 (10)	C14—C13—H13B	109.4
N3—Fe1—Cl1	94.82 (7)	C13—C14—H14A	109.4
N3—Fe1—N1	152.34 (9)	C13—C14—H14B	109.4
N3—Fe1—N2	77.39 (9)	H14A—C14—H14B	108.0
N3—Fe1—N4	73.64 (9)	C15—C14—C13	111.3 (3)
N4—Fe1—Cl1	86.52 (7)	C15—C14—H14A	109.4
N4—Fe1—N1	133.98 (9)	C15—C14—H14B	109.4
Cl2—Fe2—Cl3	111.31 (4)	C14—C15—H15A	109.3
Cl2—Fe2—Cl4	108.78 (4)	C14—C15—H15B	109.3
Cl3—Fe2—Cl4	109.42 (4)	H15A—C15—H15B	107.9
O1—Fe2—Cl2	112.02 (8)	C16—C15—C14	111.8 (3)
O1—Fe2—Cl3	107.35 (8)	C16—C15—H15A	109.3
O1—Fe2—Cl4	107.87 (7)	C16—C15—H15B	109.3
Fe2—O1—Fe1	150.26 (13)	N3—C16—C11	106.8 (2)
C1—N1—Fe1	127.54 (19)	N3—C16—C15	113.4 (2)
C9—N1—Fe1	112.45 (19)	N3—C16—H16	108.5
C9—N1—C1	119.1 (3)	C11—C16—H16	108.5
Fe1—N2—H2	107.2	C15—C16—C11	111.0 (3)
C10—N2—Fe1	109.10 (19)	C15—C16—H16	108.5
C10—N2—H2	107.2	N3—C17—H17A	109.9
C10—N2—C11	114.5 (2)	N3—C17—H17B	109.9
C11—N2—Fe1	111.27 (18)	N3—C17—C18	109.1 (2)
C11—N2—H2	107.2	H17A—C17—H17B	108.3
Fe1—N3—H3	102 (3)	C18—C17—H17A	109.9
C16—N3—Fe1	113.52 (17)	C18—C17—H17B	109.9
C16—N3—H3	104 (3)	N4—C18—C17	117.4 (3)
C17—N3—Fe1	110.66 (17)	N4—C18—C19	122.7 (3)
C17—N3—H3	112 (3)	C19—C18—C17	120.0 (3)
C17—N3—C16	114.4 (2)	C18—C19—H19	120.3
C18—N4—Fe1	113.9 (2)	C20—C19—C18	119.3 (3)
C18—N4—C26	119.3 (3)	C20—C19—H19	120.3
C26—N4—Fe1	126.1 (2)	C19—C20—H20	120.1
N1—C1—C2	120.3 (3)	C19—C20—C21	119.8 (3)
N1—C1—C6	121.0 (3)	C21—C20—H20	120.1
C2—C1—C6	118.7 (3)	C20—C21—C26	117.8 (3)
C1—C2—H2A	119.8	C22—C21—C20	123.0 (3)
C3—C2—C1	120.3 (3)	C22—C21—C26	119.2 (3)
C3—C2—H2A	119.8	C21—C22—H22	119.8
C2—C3—H3A	119.5	C23—C22—C21	120.4 (3)
C2—C3—C4	121.0 (4)	C23—C22—H22	119.8
C4—C3—H3A	119.5	C22—C23—H23	119.9
C3—C4—H4	120.2	C22—C23—C24	120.3 (3)
C5—C4—C3	119.6 (3)	C24—C23—H23	119.9
C5—C4—H4	120.2	C23—C24—H24	119.6
C4—C5—H5	119.6	C25—C24—C23	120.9 (4)
C4—C5—C6	120.8 (3)	C25—C24—H24	119.6
C6—C5—H5	119.6	C24—C25—H	120.1

C5—C6—C1	119.5 (3)	C24—C25—C26	119.8 (3)
C5—C6—C7	122.9 (3)	C26—C25—H	120.1
C7—C6—C1	117.7 (3)	N4—C26—C21	121.0 (3)
C6—C7—H7	119.9	N4—C26—C25	119.7 (3)
C8—C7—C6	120.2 (3)	C25—C26—C21	119.3 (3)
C8—C7—H7	119.9	C27B—Cl5—C27A	35.2 (10)
C7—C8—H8	120.3	C27B—Cl6A—C27A	36.6 (11)
C7—C8—C9	119.4 (3)	C27A—Cl7A—C27B	35.5 (11)
C9—C8—H8	120.3	Cl5—C27—H27	107.3
N1—C9—C8	122.5 (3)	Cl6—C27—Cl5	106.2 (6)
N1—C9—C10	118.1 (3)	Cl6—C27—H27	107.3
C8—C9—C10	119.4 (3)	Cl7—C27—Cl5	113.1 (7)
N2—C10—C9	110.7 (3)	Cl7—C27—Cl6	115.3 (7)
N2—C10—H10A	109.5	Cl7—C27—H27	107.3
N2—C10—H10B	109.5	Cl5—C27A—H27A	109.9
C9—C10—H10A	109.5	Cl6A—C27A—Cl5	112.9 (11)
C9—C10—H10B	109.5	Cl6A—C27A—H27A	109.9
H10A—C10—H10B	108.1	Cl7A—C27A—Cl5	104.2 (9)
N2—C11—H11	108.4	Cl7A—C27A—Cl6A	110.0 (11)
N2—C11—C12	114.5 (2)	Cl7A—C27A—H27A	109.9
N2—C11—C16	107.3 (2)	Cl5—C27B—Cl7A	103.7 (8)
C12—C11—H11	108.4	Cl5—C27B—H27B	105.9
C16—C11—H11	108.4	Cl6A—C27B—Cl5	120.7 (8)
C16—C11—C12	109.6 (3)	Cl6A—C27B—Cl7A	113.8 (8)
C11—C12—H12A	109.6	Cl6A—C27B—H27B	105.9
C11—C12—H12B	109.6	Cl7A—C27B—H27B	105.9
H12A—C12—H12B	108.1		

Fe1—N1—C1—C2	−14.7 (4)	C9—N1—C1—C2	177.1 (3)
Fe1—N1—C1—C6	165.0 (2)	C9—N1—C1—C6	−3.2 (4)
Fe1—N1—C9—C8	−169.5 (2)	C10—N2—C11—C12	−69.3 (4)
Fe1—N1—C9—C10	8.4 (3)	C10—N2—C11—C16	168.8 (3)
Fe1—N2—C10—C9	−39.2 (3)	C11—N2—C10—C9	−164.6 (3)
Fe1—N2—C11—C12	166.5 (2)	C11—C12—C13—C14	57.7 (4)
Fe1—N2—C11—C16	44.6 (3)	C12—C11—C16—N3	−177.7 (2)
Fe1—N3—C16—C11	37.6 (3)	C12—C11—C16—C15	58.2 (3)
Fe1—N3—C16—C15	160.1 (2)	C12—C13—C14—C15	−54.6 (4)
Fe1—N3—C17—C18	−39.6 (3)	C13—C14—C15—C16	53.5 (4)
Fe1—N4—C18—C17	10.4 (3)	C14—C15—C16—N3	−175.9 (3)
Fe1—N4—C18—C19	−169.2 (2)	C14—C15—C16—C11	−55.7 (4)
Fe1—N4—C26—C21	165.7 (2)	C16—N3—C17—C18	−169.4 (2)
Fe1—N4—C26—C25	−13.3 (4)	C16—C11—C12—C13	−59.2 (3)
Cl1—Fe1—O1—Fe2	149.00 (17)	C17—N3—C16—C11	165.9 (2)
Cl2—Fe2—O1—Fe1	−136.6 (2)	C17—N3—C16—C15	−71.5 (3)
Cl3—Fe2—O1—Fe1	100.9 (2)	C17—C18—C19—C20	−178.2 (3)
Cl4—Fe2—O1—Fe1	−16.9 (3)	C18—N4—C26—C21	−3.6 (4)
N1—Fe1—O1—Fe2	−154.4 (3)	C18—N4—C26—C25	177.4 (3)
N1—C1—C2—C3	176.3 (3)	C18—C19—C20—C21	−1.8 (5)

N1—C1—C6—C5	−176.5 (3)	C19—C20—C21—C22	177.8 (3)
N1—C1—C6—C7	2.9 (5)	C19—C20—C21—C26	−0.4 (5)
N1—C9—C10—N2	20.6 (4)	C20—C21—C22—C23	−178.5 (3)
N2—Fe1—O1—Fe2	−80.2 (3)	C20—C21—C26—N4	3.2 (4)
N2—C11—C12—C13	−179.8 (3)	C20—C21—C26—C25	−177.8 (3)
N2—C11—C16—N3	−52.8 (3)	C21—C22—C23—C24	−2.6 (5)
N2—C11—C16—C15	−176.9 (2)	C22—C21—C26—N4	−175.2 (3)
N3—Fe1—O1—Fe2	−2.1 (3)	C22—C21—C26—C25	3.8 (4)
N3—C17—C18—N4	19.0 (4)	C22—C23—C24—C25	1.8 (5)
N3—C17—C18—C19	−161.3 (3)	C23—C24—C25—C26	1.9 (5)
N4—Fe1—O1—Fe2	71.5 (3)	C24—C25—C26—N4	174.4 (3)
N4—C18—C19—C20	1.4 (5)	C24—C25—C26—C21	−4.6 (5)
C1—N1—C9—C8	0.4 (5)	C26—N4—C18—C17	−179.1 (3)
C1—N1—C9—C10	178.3 (3)	C26—N4—C18—C19	1.3 (4)
C1—C2—C3—C4	1.5 (6)	C26—C21—C22—C23	−0.2 (5)
C1—C6—C7—C8	0.2 (5)	C27A—Cl5—C27B—Cl6A	64.6 (14)
C2—C1—C6—C5	3.2 (5)	C27A—Cl5—C27B—Cl7A	−64.3 (12)
C2—C1—C6—C7	−177.4 (3)	C27A—Cl6A—C27B—Cl5	−62.5 (14)
C2—C3—C4—C5	0.8 (6)	C27A—Cl6A—C27B—Cl7A	61.7 (12)
C3—C4—C5—C6	−1.0 (6)	C27A—Cl7A—C27B—Cl5	66.9 (12)
C4—C5—C6—C1	−1.0 (5)	C27A—Cl7A—C27B—Cl6A	−66.1 (12)
C4—C5—C6—C7	179.6 (4)	C27B—Cl5—C27A—Cl6A	−51.8 (12)
C5—C6—C7—C8	179.6 (3)	C27B—Cl5—C27A—Cl7A	67.5 (14)
C6—C1—C2—C3	−3.4 (5)	C27B—Cl6A—C27A—Cl5	54.6 (12)
C6—C7—C8—C9	−2.8 (5)	C27B—Cl6A—C27A—Cl7A	−61.3 (14)
C7—C8—C9—N1	2.6 (5)	C27B—Cl7A—C27A—Cl5	−65.1 (12)
C7—C8—C9—C10	−175.2 (3)	C27B—Cl7A—C27A—Cl6A	56.1 (12)
C8—C9—C10—N2	−161.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···Cl4	0.80 (4)	2.60 (4)	3.378 (3)	167 (3)
C27*A*—H27*A*···Cl2^{i}	1.00	2.41	3.30 (2)	149

Symmetry code: (i) −*x*+1/2, −*y*+1, *z*+1/2.