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Synthesis and crystal structure study of (*R,R*)-TMCDA ethanol derivatives doubly protonated with FeCl_4^- and Cl^- as counter-ions

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The synthesis and structural characterization of the crystal forms of (*R,R*)-TMCDA and its ethanol derivative, both doubly protonated with FeCl_4^- and Cl^- as counter-ions, are reported, namely, (*R,R*)- N^1,N^1,N^2,N^2 -tetramethylcyclohexane-1,2-bis(aminium) tetrachloridoferrate chloride, ($\text{C}_{10}\text{H}_{24}\text{N}_2$)[FeCl_4] Cl (**1a**), and (*R,R*)- N^1 -(2-hydroxyethyl)- N^1,N^2,N^2 -trimethylcyclohexane-1,2-bis(aminium) tetrachloridoferrate chloride ($\text{C}_{11}\text{H}_{26}\text{N}_2\text{O}$)[FeCl_4] Cl (**2a**). A notable feature across both synthesized compounds is the presence of $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds of moderate strength in the solid state. In the case of the ethanol derivative of (*R,R*)-TMCDA, the structure also reveals the formation of intermolecular $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

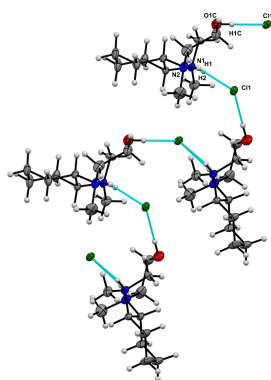
1. Chemical context

Nitrogen-containing compounds have numerous applications in coordination chemistry. Of particular interest is the molecule (*R,R*)-TMCDA (**1**), which contains two stereogenic carbon centers, enabling it to function as a bidentate, chiral ligand.

Selective deprotonation reactions play a critical role in the functionalization of certain molecules, facilitating the incorporation of new functional groups and enhancing molecular properties.

In organolithium chemistry, polyamines are the only tertiary amines susceptible to deprotonation reactions. This is due to the fact that polyamines allow the formation of a complex in which the lithiated base gets pre-coordinated. This pre-coordination brings reactive groups into close proximity, allowing selective deprotonation reactions to take place (Gessner *et al.*, 2010). This phenomenon is widely known as the complex-induced proximity effect (CIPE) (Gessner *et al.*, 2010; Whisler *et al.*, 2004; Breit, 2000; Hoveyda *et al.*, 1993; Beak & Meyers, 1986).

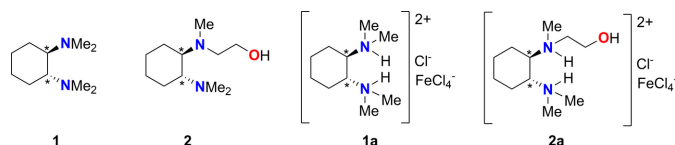
Additionally, the synthesis of new ligands with stereogenic centers presents a persistent challenge. The inherent chirality of (*R,R*)-TMCDA (**1**) coupled with its high reactivity towards lithiated bases, renders this molecule a promising precursor for the synthesis of other novel ligands. A notable example is compound **2**, which displays interesting characteristics including chirality and its function as an *N,N,O*-scorpionate ligand. In light of these characteristics, our research group has previously investigated compound **2** and related ligands (Gessner *et al.*, 2010) as well as with (*R,R*)-TMCDA (Eckert *et al.*, 2011; Strohmam & Gessner, 2008; Strohmam & Gessner,



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2007). Building upon this foundation, compounds **1a** and **2a**, incorporating the aforementioned ligands, have been successfully synthesized and crystallized in this work.



2. Structural commentary

The structure of compound **1a**, as depicted in Fig. 1, comprises a cation and two anions. The cation consists of a doubly protonated (*R,R*)-TMCD molecule, with the cyclohexane ring adopting a chair conformation. The two counter-ions are a chloride anion, that forms two N—H...Cl hydrogen bonds, and a FeCl₄⁻ anion, in which the Fe³⁺ cation is coordinated by four chlorides in a tetrahedral geometry.

The distances of interest are shown in Table 1. Hydrogen bonds between the chloride and the hydrogen atoms bonded to either N1 or N2 were observed in the crystal structure, with N1—H1...Cl1 distances of 2.133 (19) Å and N2—H2...Cl1 distances of 2.135 (17) Å and N1—H1 distances of 0.891 (19) Å and N2—H2 distances of 0.900 (18) Å. These hydrogen bonds could be classified as moderate (Steiner, 2002).

The structure of compound **2a**, illustrated in Fig. 2, consists of a cation and two anions. The cation is derived from an ethanol-substituted, doubly protonated (*R,R*)-TMCD molecule, characterized by a cyclohexane ring adopting a chair conformation. The two counter-ions include a chloride, which forms N—H...Cl hydrogen bonds, and a FeCl₄⁻ anion, wherein the Fe³⁺ cation is coordinated by four chlorides arranged in a tetrahedral geometry. The cation in this structure features three tetrahedral stereogenic centres: the two carbon atoms associated with the N-bonded cycle and the nitrogen atoms N1 or N2, which are bonded to the ethanol fragment. The nitrogen atoms have a labile configuration and both enantiomers (*R_N* and *S_N*) can occur in solid state and thus represent a further cause of the existing disorder. The more

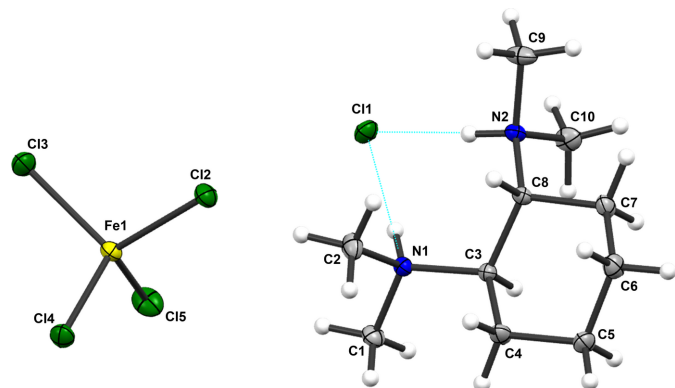


Figure 1

The molecular structure of compound **1a** with the atom labelling and displacement ellipsoids drawn at the 50% probability level.

Table 1

Hydrogen-bond geometry (Å, °) for **1a**.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...Cl1	0.891 (19)	2.133 (19)	3.0189 (11)	172.7 (19)
N2—H2...Cl1	0.900 (18)	2.135 (17)	3.0208 (10)	165 (14)

Table 2

Hydrogen-bond geometry (Å, °) for **2a**.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...Cl1	0.855 (3)	2.221 (3)	3.0603 (14)	167 (3)
N2—H2...Cl1	0.878 (3)	2.357 (3)	3.1698 (16)	154 (3)
O1C—H1C...Cl1 ⁱ	0.84	2.43	3.229 (3)	159

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$.

flexible CH₂-segments of the side chain contribute to the observed disorder of the —CH₂CH₂OH fragment as well. This is distributed at atom C2 over positions designated as *A* and *C* (CH₂ group) as well as *B* and *D* (CH₃ group) with occupancies of 0.6 and 0.4 respectively. Additionally, the C1 carbon is similarly disordered at positions *A* and *C*, also exhibiting occupancies of 0.6 and 0.4 respectively. The relevant distances are summarized in Table 2. The structural analysis reveals hydrogen bonds between the chloride anion and the hydrogen atoms attached to nitrogen atoms N1 and N2. The N2—H2...Cl1 distance is measured at 2.357 (3) Å, while the N1—H1...Cl1 distance is 2.221 (3) Å. Additionally, the N1—H1 distance is 0.855 (3) Å, and the N2—H2 distance is 0.878 (3) Å. These hydrogen bonds can be classified as moderate in strength, according to Steiner (Steiner, 2002). In contrast, no interaction is observed between the hydrogen atoms O1A—H1A or O1C—H1C and Cl1, as they are separated by a distance of 3.1245 (5) Å (H1A...Cl1) and 3.4958 (5) Å (H1C...Cl1).

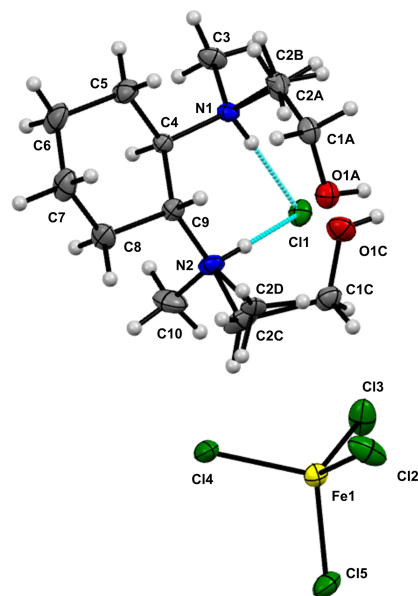


Figure 2

The molecular structure of compound **2a** with the atom labelling. The structure exhibits a disorder of the —CH₂CH₂OH fragment at two positions, with occupancies of 0.4 and 0.6 and a disorder of C2, also at two positions with occupancies of 0.4 and 0.6.

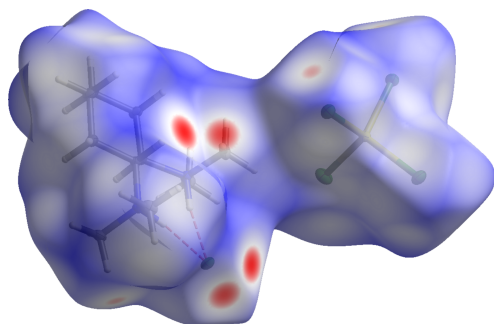


Figure 3
Three-dimensional Hirshfeld surface of **1a** mapped over d_{norm} (rescale surface property: $-0.2124 - 1.4372$).

3. Supramolecular features

To better understand the supramolecular interactions, a Hirshfeld surface analysis was performed for compound **1a**. In Fig. 3, the Hirshfeld surface generated by *CrystalExplorer21* (Spackman *et al.*, 2021) is mapped over d_{norm} (Spackman & Jayatilaka, 2009) and red dots are used to represent close contacts between the hydrogen atoms H3, H1A and H10C with the chloride anion Cl1'. The following figure (Fig. 4) shows the Hirshfeld surface with external fragments.

For further exploration of the intermolecular interactions, two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were generated as shown in Fig. 5. The $\text{H} \cdots \text{Cl}$ interaction with a contribution of 66.6% has the biggest impact on the packing in the solid state as well as the $\text{H} \cdots \text{H}$ bonds with 28.8%. $\text{Fe} \cdots \text{H}$ interactions with 0.9% and $\text{Cl} \cdots \text{Fe}$ interactions with 0.3% are less impactful in comparison.

In contrast, compound **2a** forms $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bonds. These interactions connect two different moieties of

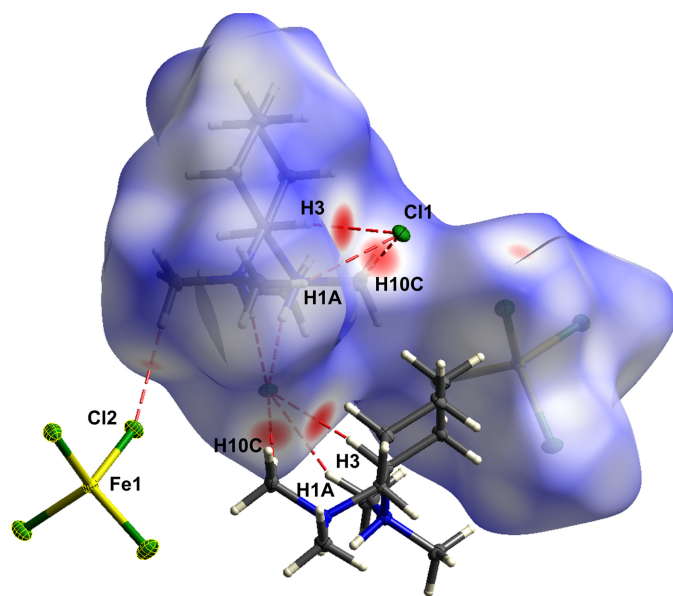


Figure 4
Three-dimensional Hirshfeld surface of **1a** mapped over d_{norm} with external fragments and atom labeling (rescale surface property: $-0.2124 - 1.4372$).

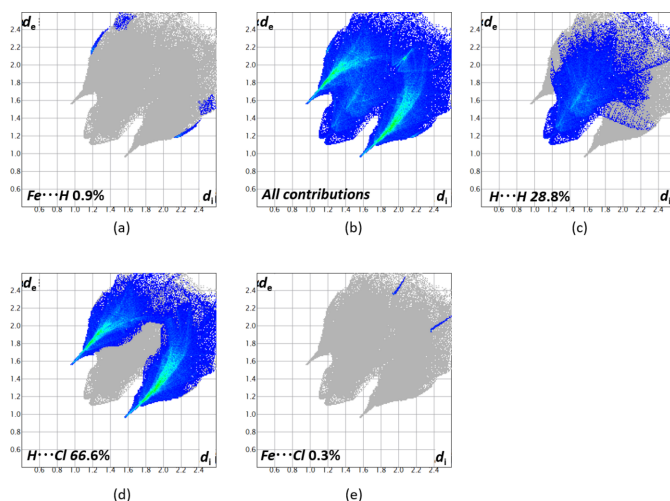


Figure 5
Two-dimensional fingerprint plots for **1a** showing (b) all interactions, and (a) and (c)–(e) delineated into contributions from other contacts (blue areas) [d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively].

compound **2a** by a chloride, resulting in a supramolecular zigzag structure in the solid state, as shown in Fig. 6. They

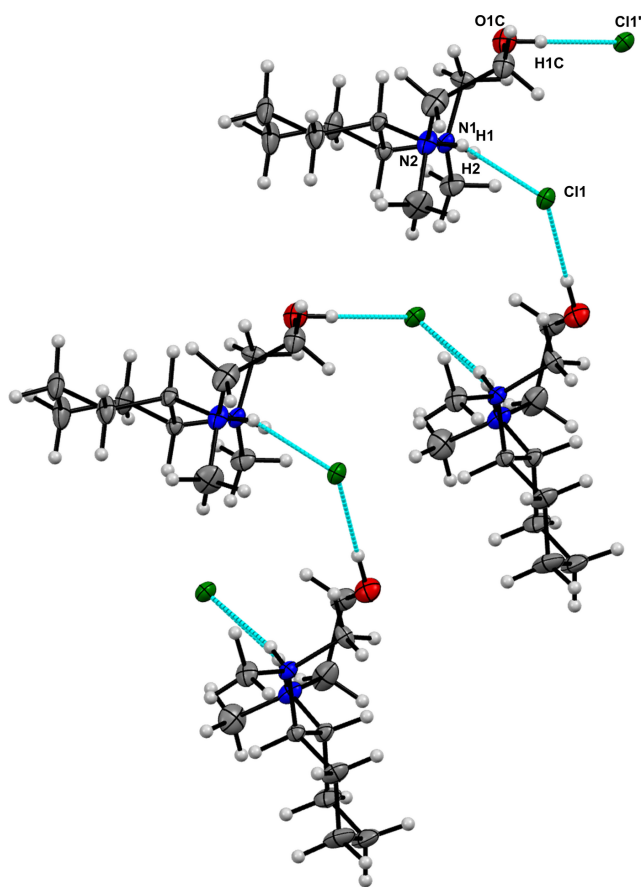


Figure 6
The supramolecular structure of compound **2a** with the formed hydrogen bonds and the atom labelling of parts of interest. The disorder with the lower occupancy (O1A, H1A) was omitted for clarity. Hydrogen bonds are depicted by dashed lines.

Table 3
Experimental details.

	1a	2a
Crystal data		
Chemical formula	(C ₁₀ H ₂₄ N ₂)[FeCl ₄]Cl	(C ₁₁ H ₂₆ N ₂ O)[FeCl ₄]Cl
<i>M_r</i>	405.41	435.44
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁
Temperature (K)	100	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.2322 (3), 9.2378 (5), 10.7384 (4)	7.6070 (5), 11.5870 (6), 21.8705 (13)
α , β , γ (°)	90, 116.797 (1), 90	90, 90, 90
<i>V</i> (Å ³)	906.02 (7)	1927.7 (2)
<i>Z</i>	2	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	1.56	1.47
Crystal size (mm)	0.50 × 0.43 × 0.38	0.8 × 0.43 × 0.18
Data collection		
Diffractometer	Bruker D8 VENTURE area detector	Bruker APEXII CCD
Absorption correction	—	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	—	0.472, 0.567
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	69005, 8763, 8442	144077, 8488, 8082
<i>R</i> _{int}	0.030	0.046
(sin θ/λ) _{max} (Å ⁻¹)	0.834	0.807
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.018, 0.037, 1.06	0.026, 0.063, 1.07
No. of reflections	8763	8488
No. of parameters	175	231
No. of restraints	1	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.23, -0.34	0.61, -0.48
Absolute structure	Flack <i>x</i> determined using 3826 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)]/[(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)	Flack <i>x</i> determined using 3373 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)]/[(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	-0.009 (3)	-0.013 (4)

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

show distances of 2.43 Å (O1C—H1C...Cl1') and the N2—H2...Cl1 distance is measured at 2.357 (3) Å, while the N1—H1...Cl1 distance is 2.221 (3) Å. In Fig. 6 the disorder with the lower occupancy is omitted for clarity, but shows the O1A—H1A...Cl1 hydrogen bond with a distance of 2.22 Å.

4. Database survey

A search of the Cambridge Structural Database (CSD version 5.43, November 2021; Groom *et al.*, 2016) for structures containing (*R,R*)-TMCD and its ethanol derivative both doubly protonated leads to three relevant and similar structures in common CUZZEC, CUZZIG (Duesler *et al.*, 1985) and POMMIO (Lian *et al.*, 2009). In these three cases, the amine substituents are -CH₂COOH fragments. Also, in all three cases there is a single anion, being either CdCl₄²⁻, PdCl₄²⁻ or PtCl₄²⁻ and none of the compounds presents a space group that matches **1a** or **2a**. Another search of structures involving non-protonated (*R,R*)-TMCD leads to several related structures, some of them being: FEJFAD, FEJFIL and FEJFOR (Eckert *et al.*, 2013), KOBDAH (Eckert *et al.*, 2014) and LECRUI (Eckert *et al.*, 2012). FEJFAD and LECRUI show the same space group as **2a** and KOBDAH with **1a**. However, all the latter structures show direct coordination to the metal unlike compounds **1a** and **2a**.

5. Synthesis and crystallization

The syntheses of compounds **1a** and **2a** were conducted according to the previously established procedure (Gessner,

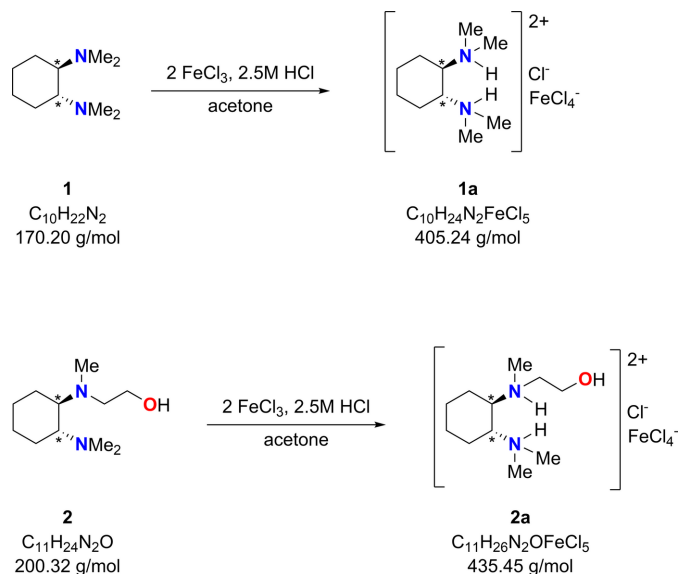


Figure 7
The synthesis of the title compounds.

2009), which involves the mixing of equimolar amounts of $\text{FeCl}_3 \cdot \text{H}_2\text{O}$ with (*R,R*)-TMCD (1) and with 2-[[(*1R,2R*)-2-(dimethylamino)cyclohexyl](methylamino)ethan-1-ol (2), respectively, to yield each compound. In both instances, the reactions were performed in a 3:2 mixture of acetone and 2.5 M of HCl at room temperature (see Fig. 7). Upon complete dissolution of the reactants, a homogeneous yellow solution was obtained, which was then allowed to stand at room temperature until complete evaporation of the solvent. After one week, yellow crystals were formed; specifically, needle-shaped crystals were obtained for compound 1a, while prism-shaped crystals were obtained for compound 2a.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms except for the protons attached to the nitrogen and oxygen atoms in both structures were positioned geometrically ($\text{C}-\text{H} = 0.95\text{--}1.00 \text{ \AA}$) and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH_2 hydrogen atoms and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for CH_3 hydrogen atoms.

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Computing details

(*R,R*)-*N*¹-(2-Hydroxyethyl)-*N*¹,*N*²,*N*²-trimethylcyclohexane-1,2-bis(aminium) tetrachloridoferrate chloride (2a)

Crystal data

(C₁₁H₂₆N₂O)[FeCl₄]Cl

$M_r = 435.44$

Orthorhombic, $P2_12_12_1$

$a = 7.6070$ (5) Å

$b = 11.5870$ (6) Å

$c = 21.8705$ (13) Å

$V = 1927.7$ (2) Å³

$Z = 4$

$F(000) = 900$

$D_x = 1.500$ Mg m⁻³

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

Cell parameters from 9581 reflections

$\theta = 2.6\text{--}34.8^\circ$

$\mu = 1.47$ mm⁻¹

$T = 100$ K

Needle, yellow

$0.8 \times 0.43 \times 0.18$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: microfocus sealed X-ray tube,

Incoatec I μ s

HELIOS mirror optics monochromator

Detector resolution: 10.4167 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.472$, $T_{\max} = 0.567$

144077 measured reflections

8488 independent reflections

8082 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 35.0^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -12 \rightarrow 12$

$k = -18 \rightarrow 18$

$l = -35 \rightarrow 35$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.063$

$S = 1.07$

8488 reflections

231 parameters

1 restraint

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2 + 0.5573P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.61$ e Å⁻³

$\Delta\rho_{\min} = -0.48$ e Å⁻³

Absolute structure: Flack x determined using

3373 quotients $[(F^-)-(F)]/[(F^+)+(F)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: -0.013 (4)

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Fe1	0.38908 (3)	0.32831 (2)	0.64131 (2)	0.02140 (5)	
Cl1	0.32469 (6)	0.71475 (4)	0.52176 (2)	0.02393 (7)	
Cl5	0.40990 (7)	0.16915 (4)	0.69535 (2)	0.03204 (10)	
Cl4	0.63253 (7)	0.34762 (4)	0.58818 (2)	0.02908 (9)	
Cl3	0.36082 (7)	0.47621 (5)	0.70394 (3)	0.03907 (12)	
Cl2	0.16007 (8)	0.31529 (4)	0.58067 (3)	0.04136 (13)	
N1	0.36655 (19)	0.80677 (11)	0.39141 (6)	0.0176 (2)	
C4	0.49489 (19)	0.73202 (12)	0.35567 (7)	0.0167 (2)	
H4	0.610719	0.734966	0.377232	0.020*	
C9	0.4413 (2)	0.60442 (13)	0.35010 (7)	0.0190 (3)	
H9	0.330578	0.600659	0.325512	0.023*	
N2	0.4066 (2)	0.54577 (12)	0.41140 (7)	0.0229 (3)	
C2A	0.1906 (10)	0.8308 (10)	0.3630 (5)	0.0204 (19)	0.6
H2AA	0.204658	0.886344	0.329535	0.031*	0.6
H2AB	0.111296	0.863005	0.393931	0.031*	0.6
H2AC	0.140939	0.758896	0.346966	0.031*	0.6
C8	0.5840 (3)	0.53894 (15)	0.31517 (9)	0.0285 (4)	
H8A	0.695235	0.542237	0.338627	0.034*	
H8B	0.549412	0.456878	0.311433	0.034*	
C5	0.5215 (3)	0.78172 (17)	0.29127 (8)	0.0283 (4)	
H5A	0.409277	0.777667	0.268459	0.034*	
H5B	0.555774	0.863928	0.294475	0.034*	
C7	0.6146 (3)	0.58882 (18)	0.25142 (9)	0.0329 (4)	
H7A	0.710524	0.546051	0.230899	0.040*	
H7B	0.506586	0.580115	0.226574	0.040*	
C10	0.5676 (3)	0.51474 (19)	0.44679 (11)	0.0380 (5)	
H10A	0.632514	0.454339	0.425011	0.057*	
H10B	0.533646	0.486423	0.487323	0.057*	
H10C	0.642387	0.583097	0.451261	0.057*	
C6	0.6630 (3)	0.71594 (19)	0.25612 (10)	0.0360 (5)	
H6A	0.675303	0.748989	0.214598	0.043*	
H6B	0.777242	0.723985	0.277372	0.043*	
C3	0.4494 (3)	0.92074 (14)	0.40779 (9)	0.0261 (3)	
H3A	0.566736	0.907371	0.424913	0.039*	
H3B	0.376186	0.960350	0.438098	0.039*	
H3C	0.459308	0.968647	0.371023	0.039*	
C2C	0.3030 (10)	0.4364 (7)	0.4077 (4)	0.0352 (15)	0.6
H2CA	0.353142	0.375708	0.434330	0.042*	0.6
H2CB	0.296099	0.407377	0.365179	0.042*	0.6

O1C	0.0357 (4)	0.5852 (2)	0.40633 (12)	0.0303 (5)	0.6
H1C	−0.026743	0.622278	0.431171	0.045*	0.6
O1A	0.0770 (5)	0.6464 (3)	0.40150 (16)	0.0268 (6)	0.4
H1A	0.015547	0.671492	0.430532	0.040*	0.4
C1C	0.0962 (5)	0.4832 (3)	0.43433 (15)	0.0273 (6)	0.6
H1CA	0.009140	0.421137	0.427170	0.033*	0.6
H1CB	0.103627	0.496020	0.479006	0.033*	0.6
C1A	0.0736 (6)	0.7274 (4)	0.3522 (2)	0.0242 (7)	0.4
H1AA	−0.047730	0.756592	0.346835	0.029*	0.4
H1AB	0.108667	0.688030	0.313934	0.029*	0.4
H2	0.351 (4)	0.594 (2)	0.4354 (13)	0.033 (7)*	
C2D	0.2584 (14)	0.4516 (10)	0.3996 (6)	0.0256 (17)	0.4
H2DA	0.230905	0.448666	0.355885	0.038*	0.4
H2DB	0.152364	0.472246	0.422608	0.038*	0.4
H2DC	0.300608	0.375886	0.413154	0.038*	0.4
H1	0.342 (4)	0.774 (2)	0.4253 (13)	0.037 (7)*	
C2B	0.1897 (17)	0.8228 (16)	0.3631 (7)	0.028 (4)	0.4
H2BA	0.125018	0.878280	0.389230	0.034*	0.4
H2BB	0.208584	0.861386	0.323279	0.034*	0.4

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02100 (10)	0.02097 (10)	0.02223 (10)	−0.00210 (8)	−0.00138 (8)	−0.00180 (8)
Cl1	0.02587 (17)	0.03036 (18)	0.01556 (13)	−0.00029 (15)	0.00178 (13)	−0.00037 (13)
Cl5	0.0315 (2)	0.0350 (2)	0.02956 (19)	−0.00769 (18)	0.00039 (16)	0.01233 (18)
Cl4	0.0340 (2)	0.02166 (16)	0.03163 (19)	0.00026 (15)	0.01043 (17)	0.00573 (14)
Cl3	0.0339 (2)	0.0387 (2)	0.0446 (3)	−0.0104 (2)	0.0089 (2)	−0.0217 (2)
Cl2	0.0391 (3)	0.0284 (2)	0.0566 (3)	0.00584 (19)	−0.0261 (2)	−0.0091 (2)
N1	0.0228 (6)	0.0148 (5)	0.0151 (5)	0.0018 (4)	0.0004 (4)	−0.0001 (4)
C4	0.0181 (6)	0.0152 (5)	0.0168 (6)	−0.0002 (4)	0.0009 (5)	−0.0016 (5)
C9	0.0223 (6)	0.0177 (6)	0.0171 (6)	−0.0033 (5)	0.0017 (5)	−0.0042 (5)
N2	0.0330 (7)	0.0138 (5)	0.0220 (6)	−0.0028 (5)	0.0049 (6)	−0.0017 (5)
C2A	0.018 (3)	0.0139 (19)	0.030 (4)	0.0001 (17)	−0.006 (2)	0.0035 (19)
C8	0.0345 (9)	0.0208 (7)	0.0303 (8)	0.0009 (6)	0.0101 (7)	−0.0093 (6)
C5	0.0386 (10)	0.0254 (8)	0.0209 (7)	0.0002 (7)	0.0093 (7)	0.0029 (6)
C7	0.0374 (10)	0.0344 (9)	0.0270 (8)	−0.0042 (8)	0.0141 (8)	−0.0121 (7)
C10	0.0487 (13)	0.0285 (9)	0.0368 (10)	0.0111 (9)	−0.0049 (9)	0.0087 (8)
C6	0.0425 (11)	0.0328 (9)	0.0326 (9)	−0.0076 (9)	0.0215 (9)	−0.0044 (7)
C3	0.0321 (9)	0.0164 (6)	0.0299 (8)	0.0006 (6)	−0.0035 (7)	−0.0057 (6)
C2C	0.053 (5)	0.024 (3)	0.029 (2)	−0.019 (3)	0.004 (3)	−0.0007 (19)
O1C	0.0374 (13)	0.0201 (10)	0.0335 (12)	0.0007 (9)	0.0022 (10)	−0.0058 (9)
O1A	0.0258 (15)	0.0288 (17)	0.0258 (15)	0.0005 (13)	0.0024 (11)	−0.0015 (13)
C1C	0.0348 (15)	0.0202 (11)	0.0269 (13)	−0.0052 (11)	0.0076 (12)	−0.0023 (10)
C1A	0.0215 (17)	0.0282 (19)	0.0229 (17)	0.0013 (14)	−0.0015 (14)	−0.0029 (15)
C2D	0.030 (4)	0.011 (2)	0.035 (4)	−0.003 (2)	0.006 (3)	−0.001 (2)
C2B	0.027 (6)	0.035 (7)	0.023 (5)	0.008 (4)	0.011 (4)	−0.002 (4)

Geometric parameters (Å, °)

Fe1—Cl5	2.1962 (5)	C7—H7A	0.9900
Fe1—Cl4	2.1976 (5)	C7—H7B	0.9900
Fe1—Cl3	2.2044 (5)	C7—C6	1.522 (3)
Fe1—Cl2	2.1946 (6)	C10—H10A	0.9800
N1—C4	1.521 (2)	C10—H10B	0.9800
N1—C2A	1.502 (7)	C10—H10C	0.9800
N1—C3	1.507 (2)	C6—H6A	0.9900
N1—H1	0.86 (3)	C6—H6B	0.9900
N1—C2B	1.492 (13)	C3—H3A	0.9800
C4—H4	1.0000	C3—H3B	0.9800
C4—C9	1.539 (2)	C3—H3C	0.9800
C4—C5	1.535 (2)	C2C—H2CA	0.9900
C9—H9	1.0000	C2C—H2CB	0.9900
C9—N2	1.526 (2)	C2C—C1C	1.763 (9)
C9—C8	1.529 (2)	O1C—H1C	0.8400
N2—C10	1.493 (3)	O1C—C1C	1.408 (4)
N2—C2C	1.494 (9)	O1A—H1A	0.8400
N2—H2	0.88 (3)	O1A—C1A	1.429 (6)
N2—C2D	1.590 (13)	C1C—H1CA	0.9900
C2A—H2AA	0.9800	C1C—H1CB	0.9900
C2A—H2AB	0.9800	C1A—H1AA	0.9900
C2A—H2AC	0.9800	C1A—H1AB	0.9900
C8—H8A	0.9900	C1A—C2B	1.435 (18)
C8—H8B	0.9900	C2D—H2DA	0.9800
C8—C7	1.527 (3)	C2D—H2DB	0.9800
C5—H5A	0.9900	C2D—H2DC	0.9800
C5—H5B	0.9900	C2B—H2BA	0.9900
C5—C6	1.526 (3)	C2B—H2BB	0.9900
Cl5—Fe1—Cl4	108.02 (2)	C8—C7—H7B	109.7
Cl5—Fe1—Cl3	108.99 (3)	H7A—C7—H7B	108.2
Cl4—Fe1—Cl3	109.37 (2)	C6—C7—C8	109.97 (16)
Cl2—Fe1—Cl5	108.95 (2)	C6—C7—H7A	109.7
Cl2—Fe1—Cl4	110.88 (3)	C6—C7—H7B	109.7
Cl2—Fe1—Cl3	110.58 (2)	N2—C10—H10A	109.5
C4—N1—H1	109 (2)	N2—C10—H10B	109.5
C2A—N1—C4	117.7 (4)	N2—C10—H10C	109.5
C2A—N1—C3	108.0 (4)	H10A—C10—H10B	109.5
C2A—N1—H1	104 (2)	H10A—C10—H10C	109.5
C3—N1—C4	110.65 (13)	H10B—C10—H10C	109.5
C3—N1—H1	106.0 (19)	C5—C6—H6A	109.6
C2B—N1—C4	115.9 (6)	C5—C6—H6B	109.6
C2B—N1—C3	111.5 (7)	C7—C6—C5	110.28 (17)
C2B—N1—H1	103 (2)	C7—C6—H6A	109.6
N1—C4—H4	107.7	C7—C6—H6B	109.6
N1—C4—C9	114.68 (12)	H6A—C6—H6B	108.1

N1—C4—C5	110.03 (13)	N1—C3—H3A	109.5
C9—C4—H4	107.7	N1—C3—H3B	109.5
C5—C4—H4	107.7	N1—C3—H3C	109.5
C5—C4—C9	108.84 (13)	H3A—C3—H3B	109.5
C4—C9—H9	107.9	H3A—C3—H3C	109.5
N2—C9—C4	113.85 (12)	H3B—C3—H3C	109.5
N2—C9—H9	107.9	N2—C2C—H2CA	111.6
N2—C9—C8	109.91 (14)	N2—C2C—H2CB	111.6
C8—C9—C4	109.14 (13)	N2—C2C—C1C	101.1 (5)
C8—C9—H9	107.9	H2CA—C2C—H2CB	109.4
C9—N2—H2	108.7 (18)	C1C—C2C—H2CA	111.6
C9—N2—C2D	106.6 (5)	C1C—C2C—H2CB	111.6
C10—N2—C9	114.90 (15)	C1C—O1C—H1C	109.5
C10—N2—C2C	104.9 (3)	C1A—O1A—H1A	109.5
C10—N2—H2	103.8 (18)	C2C—C1C—H1CA	108.8
C10—N2—C2D	120.0 (3)	C2C—C1C—H1CB	108.8
C2C—N2—C9	114.9 (4)	O1C—C1C—C2C	113.9 (3)
C2C—N2—H2	109.0 (18)	O1C—C1C—H1CA	108.8
C2D—N2—H2	101.4 (19)	O1C—C1C—H1CB	108.8
N1—C2A—H2AA	109.5	H1CA—C1C—H1CB	107.7
N1—C2A—H2AB	109.5	O1A—C1A—H1AA	109.3
N1—C2A—H2AC	109.5	O1A—C1A—H1AB	109.3
H2AA—C2A—H2AB	109.5	O1A—C1A—C2B	111.7 (7)
H2AA—C2A—H2AC	109.5	H1AA—C1A—H1AB	107.9
H2AB—C2A—H2AC	109.5	C2B—C1A—H1AA	109.3
C9—C8—H8A	109.2	C2B—C1A—H1AB	109.3
C9—C8—H8B	109.2	N2—C2D—H2DA	109.5
H8A—C8—H8B	107.9	N2—C2D—H2DB	109.5
C7—C8—C9	112.10 (16)	N2—C2D—H2DC	109.5
C7—C8—H8A	109.2	H2DA—C2D—H2DB	109.5
C7—C8—H8B	109.2	H2DA—C2D—H2DC	109.5
C4—C5—H5A	109.3	H2DB—C2D—H2DC	109.5
C4—C5—H5B	109.3	N1—C2B—H2BA	106.9
H5A—C5—H5B	108.0	N1—C2B—H2BB	106.9
C6—C5—C4	111.58 (16)	C1A—C2B—N1	121.8 (12)
C6—C5—H5A	109.3	C1A—C2B—H2BA	106.9
C6—C5—H5B	109.3	C1A—C2B—H2BB	106.9
C8—C7—H7A	109.7	H2BA—C2B—H2BB	106.7
N1—C4—C9—N2	−54.93 (18)	C2A—N1—C4—C5	53.8 (5)
N1—C4—C9—C8	−178.13 (14)	C8—C9—N2—C10	46.55 (19)
N1—C4—C5—C6	174.42 (16)	C8—C9—N2—C2C	−75.3 (4)
C4—N1—C2B—C1A	61.9 (12)	C8—C9—N2—C2D	−89.0 (4)
C4—C9—N2—C10	−76.24 (18)	C8—C7—C6—C5	−55.7 (3)
C4—C9—N2—C2C	161.9 (3)	C5—C4—C9—N2	−178.65 (14)
C4—C9—N2—C2D	148.2 (4)	C5—C4—C9—C8	58.15 (18)
C4—C9—C8—C7	−58.7 (2)	C10—N2—C2C—C1C	128.1 (3)
C4—C5—C6—C7	58.1 (2)	C3—N1—C4—C9	165.95 (13)

C9—C4—C5—C6	−59.1 (2)	C3—N1—C4—C5	−70.97 (17)
C9—N2—C2C—C1C	−104.8 (4)	C3—N1—C2B—C1A	−170.3 (8)
C9—C8—C7—C6	57.3 (2)	O1A—C1A—C2B—N1	40.0 (12)
N2—C9—C8—C7	175.82 (15)	C2B—N1—C4—C9	−65.9 (8)
N2—C2C—C1C—O1C	48.6 (5)	C2B—N1—C4—C5	57.2 (8)
C2A—N1—C4—C9	−69.3 (5)		

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>
N1—H1 $\cdots$ Cl1	0.855 (3)	2.221 (3)	3.0603 (14)	167 (3)
N2—H2 $\cdots$ Cl1	0.878 (3)	2.357 (3)	3.1698 (16)	154 (3)
O1C—H1C $\cdots$ Cl1 ⁱ	0.84	2.43	3.229 (3)	159

Symmetry code: (i) $x-1/2, -y+3/2, -z+1$.**(*R,R*)-*N*¹,*N*¹,*N*²,*N*²-Tetramethylcyclohexane-1,2-bis(aminium) tetrachloridoferrate chloride (1a)***Crystal data*(C₁₀H₂₄N₂)[FeCl₄]Cl*M_r* = 405.41Monoclinic, *P*2₁*a* = 10.2322 (3) Å*b* = 9.2378 (5) Å*c* = 10.7384 (4) Å β = 116.797 (1)°*V* = 906.02 (7) Å³*Z* = 2*F*(000) = 418*D_x* = 1.486 Mg m^{−3}Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 1350 reflections

 θ = 4.3–31.7° μ = 1.56 mm^{−1}*T* = 100 K

Prism, yellow

0.50 × 0.43 × 0.38 mm

Data collection

Bruker D8 VENTURE area detector

diffractometer

Radiation source: microfocus sealed X-ray tube,

Incoatec I μ s

HELIOS mirror optics monochromator

Detector resolution: 10.4167 pixels mm^{−1} ω and ϕ scans

69005 measured reflections

8763 independent reflections

8442 reflections with $I > 2\sigma(I)$ *R*_{int} = 0.030 $\theta_{\max}$ = 36.3°, $\theta_{\min}$ = 2.1°*h* = −13→17*k* = −15→15*l* = −17→17*Refinement*Refinement on *F*²

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)]$ = 0.018 $wR(F^2)$ = 0.037*S* = 1.06

8763 reflections

175 parameters

1 restraint

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0156P)^2 + 0.078P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max}$ = 0.001 $\Delta\rho_{\max}$ = 0.23 e Å^{−3} $\Delta\rho_{\min}$ = −0.33 e Å^{−3}Absolute structure: Flack *x* determined using 3826 quotients $[(I^-)-(I^+)]/[(I^-)+(I^+)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: −0.009 (3)

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.73543 (2)	0.37018 (2)	0.11080 (2)	0.01357 (3)
Cl2	0.76183 (3)	0.51878 (3)	0.28052 (3)	0.01944 (5)
Cl5	0.76963 (3)	0.49518 (3)	−0.04548 (3)	0.01960 (5)
Cl1	0.35677 (3)	0.17053 (3)	0.42055 (3)	0.01882 (5)
Cl3	0.52216 (3)	0.26207 (4)	0.02175 (3)	0.02597 (6)
Cl4	0.90709 (3)	0.20084 (3)	0.19738 (3)	0.02248 (5)
N1	0.28286 (10)	0.44077 (10)	0.53384 (9)	0.01356 (15)
N2	0.26193 (10)	0.43743 (10)	0.23871 (9)	0.01275 (14)
C8	0.18173 (11)	0.54564 (11)	0.28693 (10)	0.01174 (15)
H8	0.0835	0.5042	0.2652	0.014*
C9	0.17020 (12)	0.38898 (13)	0.09145 (11)	0.01754 (19)
H9A	0.0722	0.3629	0.0790	0.026*
H9B	0.2159	0.3046	0.0717	0.026*
H9C	0.1625	0.4677	0.0274	0.026*
C7	0.15769 (13)	0.68716 (12)	0.20441 (11)	0.01745 (19)
H7A	0.2536	0.7251	0.2178	0.021*
H7B	0.0983	0.6667	0.1038	0.021*
C3	0.26269 (11)	0.57574 (11)	0.44544 (10)	0.01184 (15)
H3	0.3623	0.6133	0.4670	0.014*
C6	0.08042 (13)	0.80253 (12)	0.24935 (12)	0.01837 (19)
H6A	0.0700	0.8927	0.1958	0.022*
H6B	−0.0187	0.7685	0.2299	0.022*
C1	0.40538 (13)	0.45802 (15)	0.67836 (11)	0.0231 (2)
H1A	0.4946	0.4876	0.6728	0.035*
H1B	0.4226	0.3657	0.7282	0.035*
H1C	0.3790	0.5320	0.7284	0.035*
C5	0.16937 (13)	0.83247 (12)	0.40466 (12)	0.01787 (19)
H5A	0.1201	0.9077	0.4341	0.021*
H5B	0.2677	0.8686	0.4238	0.021*
C10	0.40958 (12)	0.48351 (14)	0.25595 (12)	0.0200 (2)
H10A	0.3979	0.5602	0.1887	0.030*
H10B	0.4595	0.4005	0.2396	0.030*
H10C	0.4679	0.5200	0.3509	0.030*
C4	0.18391 (12)	0.69326 (12)	0.48655 (11)	0.01606 (18)
H4A	0.0853	0.6584	0.4681	0.019*
H4B	0.2394	0.7133	0.5877	0.019*
C2	0.14826 (12)	0.38899 (13)	0.54129 (12)	0.0194 (2)
H2A	0.1253	0.4545	0.6005	0.029*
H2B	0.1651	0.2913	0.5810	0.029*

H2C	0.0661	0.3872	0.4473	0.029*
H1	0.3094 (18)	0.366 (2)	0.4978 (18)	0.025 (4)*
H2	0.2756 (17)	0.3539 (19)	0.2862 (17)	0.022 (4)*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01513 (6)	0.01373 (6)	0.01181 (6)	−0.00141 (5)	0.00605 (5)	−0.00101 (5)
Cl2	0.02272 (11)	0.01915 (11)	0.01510 (10)	0.00063 (9)	0.00734 (9)	−0.00412 (9)
Cl5	0.02426 (12)	0.01906 (12)	0.01887 (10)	0.00237 (10)	0.01270 (9)	0.00438 (9)
Cl1	0.02039 (11)	0.01434 (10)	0.02008 (11)	0.00533 (9)	0.00767 (9)	0.00351 (9)
Cl3	0.02241 (13)	0.03108 (15)	0.02344 (13)	−0.01235 (12)	0.00946 (11)	−0.00656 (11)
Cl4	0.02828 (13)	0.01860 (12)	0.02112 (11)	0.00725 (10)	0.01163 (10)	0.00335 (9)
N1	0.0145 (4)	0.0146 (4)	0.0109 (3)	0.0009 (3)	0.0052 (3)	0.0020 (3)
N2	0.0138 (3)	0.0133 (4)	0.0110 (3)	0.0011 (3)	0.0054 (3)	−0.0002 (3)
C8	0.0124 (4)	0.0121 (4)	0.0103 (3)	0.0013 (3)	0.0047 (3)	0.0003 (3)
C9	0.0200 (4)	0.0178 (5)	0.0126 (4)	−0.0006 (4)	0.0054 (3)	−0.0040 (3)
C7	0.0249 (5)	0.0150 (5)	0.0138 (4)	0.0053 (4)	0.0099 (4)	0.0039 (3)
C3	0.0131 (4)	0.0120 (4)	0.0101 (3)	−0.0003 (3)	0.0050 (3)	0.0010 (3)
C6	0.0229 (5)	0.0152 (4)	0.0172 (4)	0.0058 (4)	0.0092 (4)	0.0032 (4)
C1	0.0212 (5)	0.0290 (6)	0.0122 (4)	−0.0021 (4)	0.0015 (4)	0.0043 (4)
C5	0.0241 (5)	0.0123 (4)	0.0190 (5)	0.0013 (4)	0.0112 (4)	−0.0005 (3)
C10	0.0150 (4)	0.0256 (5)	0.0216 (5)	−0.0018 (4)	0.0101 (4)	−0.0058 (4)
C4	0.0217 (5)	0.0143 (4)	0.0151 (4)	0.0019 (4)	0.0109 (4)	−0.0003 (3)
C2	0.0190 (4)	0.0194 (5)	0.0216 (5)	−0.0018 (4)	0.0106 (4)	0.0053 (4)

Geometric parameters ($\text{\AA}$, $^\circ$)

Fe1—Cl2	2.1989 (3)	C7—C6	1.5290 (15)
Fe1—Cl5	2.1913 (3)	C3—H3	1.0000
Fe1—Cl3	2.1891 (3)	C3—C4	1.5310 (14)
Fe1—Cl4	2.2191 (3)	C6—H6A	0.9900
N1—C3	1.5237 (13)	C6—H6B	0.9900
N1—C1	1.5004 (14)	C6—C5	1.5224 (16)
N1—C2	1.4939 (13)	C1—H1A	0.9800
N1—H1	0.891 (19)	C1—H1B	0.9800
N2—C8	1.5245 (13)	C1—H1C	0.9800
N2—C9	1.4978 (13)	C5—H5A	0.9900
N2—C10	1.4985 (14)	C5—H5B	0.9900
N2—H2	0.900 (18)	C5—C4	1.5269 (15)
C8—H8	1.0000	C10—H10A	0.9800
C8—C7	1.5358 (14)	C10—H10B	0.9800
C8—C3	1.5458 (13)	C10—H10C	0.9800
C9—H9A	0.9800	C4—H4A	0.9900
C9—H9B	0.9800	C4—H4B	0.9900
C9—H9C	0.9800	C2—H2A	0.9800
C7—H7A	0.9900	C2—H2B	0.9800
C7—H7B	0.9900	C2—H2C	0.9800

C12—Fe1—C14	108.335 (12)	C8—C3—H3	107.6
C15—Fe1—C12	107.664 (13)	C4—C3—C8	110.77 (8)
C15—Fe1—C14	108.833 (12)	C4—C3—H3	107.6
C13—Fe1—C12	111.878 (13)	C7—C6—H6A	109.8
C13—Fe1—C15	112.196 (13)	C7—C6—H6B	109.8
C13—Fe1—C14	107.837 (15)	H6A—C6—H6B	108.2
C3—N1—H1	110.2 (11)	C5—C6—C7	109.49 (9)
C1—N1—C3	111.89 (9)	C5—C6—H6A	109.8
C1—N1—H1	104.8 (11)	C5—C6—H6B	109.8
C2—N1—C3	115.23 (8)	N1—C1—H1A	109.5
C2—N1—C1	109.82 (9)	N1—C1—H1B	109.5
C2—N1—H1	104.1 (11)	N1—C1—H1C	109.5
C8—N2—H2	109.8 (10)	H1A—C1—H1B	109.5
C9—N2—C8	112.18 (8)	H1A—C1—H1C	109.5
C9—N2—C10	109.41 (8)	H1B—C1—H1C	109.5
C9—N2—H2	101.9 (11)	C6—C5—H5A	109.8
C10—N2—C8	116.03 (8)	C6—C5—H5B	109.8
C10—N2—H2	106.4 (10)	C6—C5—C4	109.44 (9)
N2—C8—H8	108.0	H5A—C5—H5B	108.2
N2—C8—C7	109.45 (8)	C4—C5—H5A	109.8
N2—C8—C3	112.87 (8)	C4—C5—H5B	109.8
C7—C8—H8	108.0	N2—C10—H10A	109.5
C7—C8—C3	110.43 (8)	N2—C10—H10B	109.5
C3—C8—H8	108.0	N2—C10—H10C	109.5
N2—C9—H9A	109.5	H10A—C10—H10B	109.5
N2—C9—H9B	109.5	H10A—C10—H10C	109.5
N2—C9—H9C	109.5	H10B—C10—H10C	109.5
H9A—C9—H9B	109.5	C3—C4—H4A	109.4
H9A—C9—H9C	109.5	C3—C4—H4B	109.4
H9B—C9—H9C	109.5	C5—C4—C3	111.00 (8)
C8—C7—H7A	109.1	C5—C4—H4A	109.4
C8—C7—H7B	109.1	C5—C4—H4B	109.4
H7A—C7—H7B	107.8	H4A—C4—H4B	108.0
C6—C7—C8	112.48 (9)	N1—C2—H2A	109.5
C6—C7—H7A	109.1	N1—C2—H2B	109.5
C6—C7—H7B	109.1	N1—C2—H2C	109.5
N1—C3—C8	113.18 (8)	H2A—C2—H2B	109.5
N1—C3—H3	107.6	H2A—C2—H2C	109.5
N1—C3—C4	109.98 (8)	H2B—C2—H2C	109.5
N1—C3—C4—C5	177.07 (8)	C7—C6—C5—C4	−59.87 (12)
N2—C8—C7—C6	−178.78 (9)	C3—C8—C7—C6	−53.93 (12)
N2—C8—C3—N1	−60.24 (11)	C6—C5—C4—C3	60.65 (12)
N2—C8—C3—C4	175.71 (8)	C1—N1—C3—C8	158.64 (9)
C8—C7—C6—C5	57.65 (13)	C1—N1—C3—C4	−76.88 (11)
C8—C3—C4—C5	−57.08 (11)	C10—N2—C8—C7	59.79 (11)
C9—N2—C8—C7	−66.97 (11)	C10—N2—C8—C3	−63.63 (11)

C9—N2—C8—C3	169.61 (9)	C2—N1—C3—C8	−74.97 (11)
C7—C8—C3—N1	176.89 (8)	C2—N1—C3—C4	49.51 (11)
C7—C8—C3—C4	52.84 (11)		

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>
N1—H1 $\cdots$ Cl1	0.891 (19)	2.133 (19)	3.0189 (11)	172.7 (19)
N2—H2 $\cdots$ Cl1	0.900 (18)	2.135 (17)	3.0208 (10)	165 (14)