

Bis(8-methylquinolinium) tetrachlorido-ferrate(III) chloride

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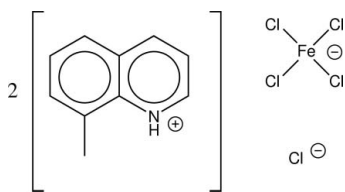
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.029; wR factor = 0.086; data-to-parameter ratio = 16.1.

The asymmetric unit of the title compound, $(\text{C}_{10}\text{H}_{10}\text{N})_2[\text{FeCl}_4]\text{Cl}$, contains two protonated 8-methylquinolinium cations, one chloride anion and one tetrachloridoferrate(III) anion. The mean Fe—Cl distance is 2.1880 (7) Å. The two 8-methylquinolinium cations and chloride anion are connected *via* N—H \cdots Cl hydrogen bonds. Furthermore, there are stacking interactions between cations. N—H \cdots Cl hydrogen bonds link two 8-methylquinolinium cations. The bromido analogue is isostructural with the title compound.

Related literature

For the bromido analogue, see: Kruszynski *et al.* (2007). For details of other tetrahalogenoferrates with quinoline and its derivatives see: Bottomley *et al.* (1984); Warnke *et al.* (2006); Wyrzykowski, Sikorski, Konitz *et al.* (2006); Wyrzykowski, Sikorski, Lis *et al.* (2006); Wyrzykowski, Warnke *et al.* (2006); and for similar tetrahalogenoferrates with aromatic amines acting as balancing cations see: Abboud *et al.* (2005); Barbaro *et al.* (1992); Chan & Baird (2004); Couce *et al.* (1995); Daran *et al.* (1979); James *et al.* (2001, 1982), Khan *et al.* (1987); Lowe *et al.* (1990, 1994); Hackert & Jacobson (1971); Podesta & Orpen (2005); Shaviv *et al.* (1992); Veidis *et al.* (1979, 1981); Zora *et al.* (1990); Zordan *et al.* (2005). For general synthetic procedures, see: Warnke *et al.* (2003).



Experimental

Crystal data

$(\text{C}_{10}\text{H}_{10}\text{N})_2[\text{FeCl}_4]\text{Cl}$
 $M_r = 521.48$
 Triclinic, $P\bar{1}$
 $a = 7.9065$ (3) Å
 $b = 10.1445$ (3) Å
 $c = 14.8518$ (6) Å
 $\alpha = 84.097$ (3)°
 $\beta = 78.200$ (3)°
 $\gamma = 84.869$ (2)°
 $V = 1157.00$ (7) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.24$ mm⁻¹
 $T = 291.0$ (3) K
 $0.38 \times 0.10 \times 0.06$ mm

Data collection

Kuma KM-4 CCD diffractometer
 Absorption correction: numerical
 (*X-RED*; Stoe & Cie, 1999)
 $T_{\min} = 0.861$, $T_{\max} = 0.919$
 11265 measured reflections
 4101 independent reflections
 3257 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.086$
 $S = 1.09$
 4101 reflections
 255 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots C199 ⁱ	0.96	2.15	3.0490 (18)	156
N11—H11N \cdots C199	0.94	2.15	3.0143 (18)	151

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

Table 2

Geometry of stacking interactions (Å, °).

$C_g(Z)$ is the centroid of the six-membered ring containing atom Z . $C_g\cdots C_g$ is the distance between ring centroids, α is the dihedral angle between planes I and J , β is the angle between the $C_g(I)-C_g(J)$ vector and the normal to plane I , γ is the angle between the $C_g(I)-C_g(J)$ vector and the normal to plane J , $C_g(I)_p$ is the perpendicular distance of $C_g(I)$ from ring J and $C_g(J)_p$ is the perpendicular distance of $C_g(J)$ from ring I .

$C_g(I)\cdots C_g(J)$	$C_g\cdots C_g$	α	β	γ	$C_g(I)_p$	$C_g(J)_p$
$C_g(\text{N1})\cdots C_g(\text{C19})$	3.8676	5.10	24.54	29.63	3.362	3.518
$C_g(\text{C9})\cdots C_g(\text{N11})$	3.8044	4.49	26.30	22.23	3.522	3.411
$C_g(\text{C9})\cdots C_g(\text{C19})$	3.7121	4.03	18.93	18.86	3.513	3.511

Data collection: *CrysAlis CCD* (UNIL IC & Kuma, 2000); cell refinement: *CrysAlis RED* (UNIL IC & Kuma, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1990b) and *ORTEP-3* (Windows Version 1.062; Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2459).

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supplementary materials

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Bis(8-methylquinolinium) tetrachloridoferrate(III) chloride

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Comment

The composition of quinolinium and methylquinolinium tetrahalogenoferrates(III) has been found to be distinctly dependent on the location of the methyl substituent in the quinoline ring and the kind of halide ligands in the coordination sphere of Fe(III) (Warnke *et al.*, 2003). When a quinolinium cation is a counter-ion, both the $[\text{FeBr}_4]^-$ and $[\text{FeCl}_4]^-$ anions form binary (1:1) salts (Wyrzykowski, Sikorski, Konitz *et al.*, 2006). With a 2-methyl substituted quinolinium cation (MeQH), resulting salts have a composition of $(2\text{MeQH})_2[\text{FeX}_4]X$ (where $X = \text{Br}$ or Cl) (Warnke *et al.*, 2006; Wyrzykowski, Sikorski, Lis *et al.*, 2006). Introduction of the 2-methylquinoline substituted at position 4 by $-\text{NH}_2$ leads again to formation of 1:1 salt (Wyrzykowski, Warnke *et al.*, 2006). The 8-methylquinoline forms $(8\text{MeQH})_2[\text{FeBr}_4]\text{Br}$ salt (Kruszynski *et al.*, 2007). Generally in compounds containing aromatic amines acting as balancing cations, replacing the FeBr_4 anions by FeCl_4 anions leads to isostructural compounds: $(2\text{MeQH})_2[\text{FeX}_4]X$ (Warnke *et al.*, 2006; Wyrzykowski, Sikorski, Lis *et al.*, 2006), $(4\text{ClpyH})_3(\text{FeX}_4)_2X$ (where 4ClpyH means 4-chloropyridinium) (Lowe *et al.*, 1990; Zordan *et al.*, 2005), but cases of non-isostructurality are also known: $(\text{pyH})_3(\text{FeX}_4)_2X$ (where pyH means pyridinium) (Shaviv *et al.*, 1992; Lowe *et al.*, 1994). For some compounds, salts with different composition are found for FeCl_4 anions: $(\text{pyH})_5(\text{FeX}_4)_2X_3$ (James *et al.*, 1982). Thus determining the composition of FeCl_4 complex containing quinoline methylated at position 8 was undertaken.

All 8-methylquinolinium cations intramolecular distances and angles in (I) (Fig. 1) can be considered normal. All atoms lie in general positions. The asymmetric unit contains two protonated 8-methylquinolinium cations, one chloride anion and one tetrachloroferrate anion. The mean Fe—Cl distance is 2.1880 (7) Å. Four Cl—Fe—Cl angles are smaller than tetrahedral and two are greater than tetrahedral one. The 8-methylquinolinium cations can be considered planar and are inclined at 4.71 (7)°. From weighted least squares planes calculated through all non-hydrogen atoms of cations the most deviate atom C10 (about 0.018 (2) Å) in one molecule and C14 (about 0.018 (2) Å) in second molecule. The two 8-methylquinolinium cations and chloride anion are connected *via* $\text{N—H}\cdots\text{Cl}\cdots\text{H—N}$ hydrogen bonds (Table 2, Fig. 2). The cations are associated *via* $\pi\cdots\pi$ stacking interactions (Table 2) to dimers, and dimers are separated by anions from each other. Thus, in the considered structure, tetrachloroferrate anions play the role of a stacking breaker. The $\text{N—H}\cdots\text{Cl}\cdots\text{H—N}$ hydrogen bonds together with $\pi\cdots\pi$ stacking interactions expands molecules to the seminfinite chain along crystallographic *b* axis (Fig. 2).

Experimental

The synthesis of the title compound was carried out using a procedure similar to that previously reported for the preparation of bis(8-methylquinolinium) tetrabromidoferrate(III) bromide (Kruszynski *et al.*, 2007). To a solution of FeCl_3 (*ca* 0.05 mol) in ethanol (96%) (25 ml), a stoichiometric quantity of a 12 mol/dm³ HCl solution and 8-methylquinoline (*ca* 0.05 mol) were added in turn. After 7 days the title compound precipitated. It was recrystallized from ethanol at ambient temperature. After 2 days yellow crystals appeared. The compound was dried over P_4O_{10} in a vacuum desiccator. Elemental analysis (calculated/found %): C 46.04/46.36, H 3.84/3.82, N 5.37/5.17, Cl 34.05/33.85, Fe 10.71/10.52.

Refinement

The carbon-bonded hydrogen atoms were placed in calculated positions and were refined as riding on adjacent carbon atom with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C-non-methyl})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$. The methyl groups were allowed to rotate about their local threefold axis (AFIX 137). The nitrogen-bonded hydrogen atoms were found from difference Fourier synthesis after eight cycles of anisotropic refinement and were refined as riding on adjacent nitrogen atom with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Figures

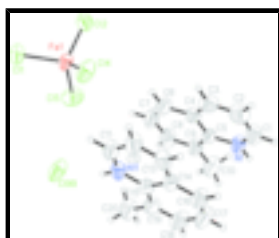


Fig. 1. Molecular structure of the title compound (I). Displacement ellipsoids are drawn at the 50% probability level.



Fig. 2. A part of the molecular packing of the title compound showing short intermolecular interactions (hydrogen bonds are indicated by dashed lines).

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Crystal data

$(\text{C}_{10}\text{H}_{10}\text{N})_2[\text{FeCl}_4]\text{Cl}$
 $M_r = 521.48$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.9065(3) \text{ \AA}$
 $b = 10.1445(3) \text{ \AA}$
 $c = 14.8518(6) \text{ \AA}$
 $\alpha = 84.097(3)^\circ$
 $\beta = 78.200(3)^\circ$
 $\gamma = 84.869(2)^\circ$
 $V = 1157.00(7) \text{ \AA}^3$

$Z = 2$

$F_{000} = 530$

$D_x = 1.497 \text{ Mg m}^{-3}$

$D_m = 1.50 \text{ Mg m}^{-3}$

D_m measured by Berman density torsion balance

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6196 reflections

$\theta = 5\text{--}20^\circ$

$\mu = 1.24 \text{ mm}^{-1}$

$T = 291.0(3) \text{ K}$

Needle, yellow

$0.38 \times 0.10 \times 0.06 \text{ mm}$

Data collection

Kuma KM-4-CCD
diffractometer

Radiation source: fine-focus sealed tube

4101 independent reflections

3257 reflections with $I > 2\sigma(I)$

Monochromator: graphite $R_{\text{int}} = 0.039$
 Detector resolution: 1048576 pixels mm⁻¹ $\theta_{\text{max}} = 25.1^\circ$
 $T = 291.0(3)$ K $\theta_{\text{min}} = 2.0^\circ$
 ω scans $h = -9 \rightarrow 8$
 Absorption correction: numerical (X-RED; Stoe & Cie, 1999) $k = -12 \rightarrow 12$
 $T_{\text{min}} = 0.861$, $T_{\text{max}} = 0.919$ $l = -16 \rightarrow 17$
 11265 measured reflections

Refinement

Refinement on F^2 Secondary atom site location: structure-invariant direct methods
 Least-squares matrix: full Hydrogen site location: mixed
 $R[F^2 > 2\sigma(F^2)] = 0.029$ H-atom parameters constrained
 $wR(F^2) = 0.086$ $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.09$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 4101 reflections $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{Å}^{-3}$
 255 parameters $\Delta\rho_{\text{min}} = -0.29 \text{ e } \text{Å}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.84101 (4)	0.23561 (3)	0.128413 (19)	0.04951 (12)
Cl1	0.82335 (8)	0.02684 (6)	0.10945 (4)	0.06251 (17)
Cl2	1.08019 (8)	0.30260 (6)	0.03856 (4)	0.06542 (18)
Cl4	0.61592 (9)	0.35139 (7)	0.08881 (5)	0.0759 (2)
Cl5	0.85692 (11)	0.25261 (9)	0.27133 (4)	0.0882 (2)
N1	0.3990 (2)	1.01488 (17)	0.24864 (11)	0.0513 (4)
H1N	0.3935	1.0690	0.2983	0.062*
C1	0.3282 (3)	1.0698 (2)	0.17910 (15)	0.0606 (6)
H1	0.2776	1.1561	0.1810	0.073*
C2	0.3285 (3)	1.0010 (3)	0.10422 (16)	0.0685 (7)

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H2	0.2783	1.0398	0.0556	0.082*
C3	0.4035 (3)	0.8752 (3)	0.10222 (15)	0.0622 (6)
H3	0.4057	0.8284	0.0513	0.075*
C4	0.4772 (3)	0.8150 (2)	0.17530 (14)	0.0523 (5)
C5	0.4737 (3)	0.8883 (2)	0.25102 (13)	0.0450 (5)
C6	0.5517 (3)	0.6842 (2)	0.17673 (19)	0.0650 (6)
H6	0.5559	0.6342	0.1271	0.078*
C7	0.6166 (3)	0.6318 (3)	0.2500 (2)	0.0772 (8)
H7	0.6645	0.5447	0.2512	0.093*
C8	0.6136 (3)	0.7065 (3)	0.32554 (19)	0.0678 (7)
H8	0.6609	0.6674	0.3751	0.081*
C9	0.5436 (3)	0.8341 (2)	0.32818 (15)	0.0554 (5)
C10	0.5423 (4)	0.9154 (3)	0.40664 (16)	0.0757 (8)
H10A	0.5891	0.8618	0.4541	0.114*
H10B	0.4254	0.9469	0.4311	0.114*
H10C	0.6113	0.9897	0.3851	0.114*
N11	0.1904 (2)	0.51280 (17)	0.30506 (11)	0.0500 (4)
H11N	0.2380	0.4449	0.3429	0.060*
C11	0.1783 (3)	0.4871 (2)	0.22141 (15)	0.0580 (6)
H11	0.2165	0.4037	0.2013	0.070*
C12	0.1095 (3)	0.5828 (2)	0.16336 (16)	0.0628 (6)
H12	0.1022	0.5649	0.1042	0.075*
C13	0.0529 (3)	0.7029 (2)	0.19396 (15)	0.0569 (6)
H13	0.0034	0.7671	0.1559	0.068*
C14	0.0673 (3)	0.7326 (2)	0.28227 (14)	0.0476 (5)
C15	0.1394 (3)	0.6330 (2)	0.33969 (13)	0.0446 (5)
C16	0.0135 (3)	0.8566 (2)	0.31588 (17)	0.0628 (6)
H16	-0.0348	0.9236	0.2793	0.075*
C17	0.0315 (4)	0.8789 (3)	0.40055 (18)	0.0784 (8)
H17	-0.0032	0.9619	0.4222	0.094*
C18	0.1020 (4)	0.7788 (3)	0.45683 (17)	0.0718 (7)
H18	0.1104	0.7966	0.5159	0.086*
C19	0.1590 (3)	0.6560 (2)	0.42852 (14)	0.0551 (6)
C20	0.2383 (4)	0.5506 (3)	0.48932 (16)	0.0733 (7)
H20A	0.2492	0.5869	0.5449	0.110*
H20B	0.3507	0.5201	0.4572	0.110*
H20C	0.1655	0.4774	0.5047	0.110*
C199	0.31343 (11)	0.23929 (7)	0.37586 (4)	0.0810 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0657 (2)	0.04101 (19)	0.04674 (18)	0.00170 (14)	-0.02298 (14)	-0.00737 (12)
Cl1	0.0779 (4)	0.0394 (3)	0.0755 (4)	-0.0032 (3)	-0.0279 (3)	-0.0049 (2)
Cl2	0.0801 (4)	0.0563 (4)	0.0618 (3)	-0.0113 (3)	-0.0194 (3)	0.0021 (3)
Cl4	0.0841 (4)	0.0607 (4)	0.0900 (4)	0.0244 (3)	-0.0386 (3)	-0.0223 (3)
Cl5	0.1168 (6)	0.1058 (6)	0.0494 (3)	-0.0085 (5)	-0.0284 (3)	-0.0168 (3)
N1	0.0686 (12)	0.0436 (10)	0.0443 (9)	0.0053 (9)	-0.0179 (8)	-0.0098 (7)

C1	0.0774 (16)	0.0508 (14)	0.0555 (13)	0.0066 (12)	-0.0240 (11)	-0.0008 (10)
C2	0.0866 (18)	0.0747 (19)	0.0501 (13)	-0.0060 (14)	-0.0285 (12)	-0.0023 (12)
C3	0.0740 (16)	0.0723 (18)	0.0451 (12)	-0.0168 (13)	-0.0116 (11)	-0.0174 (11)
C4	0.0558 (13)	0.0501 (13)	0.0516 (12)	-0.0104 (10)	-0.0034 (10)	-0.0151 (10)
C5	0.0508 (12)	0.0401 (11)	0.0438 (10)	0.0006 (9)	-0.0080 (9)	-0.0081 (8)
C6	0.0631 (15)	0.0507 (15)	0.0796 (16)	-0.0018 (12)	-0.0019 (12)	-0.0236 (12)
C7	0.0704 (17)	0.0424 (14)	0.113 (2)	0.0083 (12)	-0.0048 (15)	-0.0156 (14)
C8	0.0660 (15)	0.0542 (15)	0.0826 (17)	0.0083 (12)	-0.0233 (13)	0.0032 (13)
C9	0.0637 (14)	0.0481 (13)	0.0549 (12)	0.0059 (11)	-0.0173 (10)	-0.0043 (10)
C10	0.102 (2)	0.0740 (18)	0.0588 (14)	0.0181 (15)	-0.0408 (13)	-0.0118 (12)
N11	0.0620 (11)	0.0394 (10)	0.0502 (10)	-0.0013 (8)	-0.0195 (8)	0.0031 (7)
C11	0.0800 (16)	0.0412 (12)	0.0568 (13)	-0.0017 (11)	-0.0220 (11)	-0.0081 (10)
C12	0.0945 (18)	0.0470 (14)	0.0544 (13)	-0.0078 (12)	-0.0313 (12)	-0.0029 (10)
C13	0.0705 (15)	0.0488 (13)	0.0553 (13)	-0.0043 (11)	-0.0266 (11)	0.0064 (10)
C14	0.0494 (12)	0.0410 (12)	0.0528 (11)	-0.0007 (9)	-0.0139 (9)	0.0004 (9)
C15	0.0478 (11)	0.0398 (11)	0.0448 (11)	-0.0007 (9)	-0.0080 (8)	-0.0012 (8)
C16	0.0712 (16)	0.0481 (14)	0.0681 (15)	0.0134 (11)	-0.0199 (12)	-0.0037 (11)
C17	0.101 (2)	0.0611 (17)	0.0747 (17)	0.0241 (15)	-0.0230 (15)	-0.0260 (13)
C18	0.095 (2)	0.0710 (18)	0.0510 (13)	0.0126 (15)	-0.0188 (12)	-0.0174 (12)
C19	0.0646 (14)	0.0540 (14)	0.0460 (11)	0.0038 (11)	-0.0134 (10)	-0.0026 (10)
C20	0.103 (2)	0.0677 (17)	0.0531 (13)	0.0077 (15)	-0.0325 (13)	-0.0006 (12)
Cl99	0.1340 (6)	0.0521 (4)	0.0576 (3)	0.0333 (4)	-0.0340 (4)	-0.0127 (3)

Geometric parameters (Å, °)

Fe1—C15	2.1770 (7)	C10—H10B	0.9600
Fe1—C11	2.1853 (7)	C10—H10C	0.9600
Fe1—C14	2.1921 (7)	N11—C11	1.319 (3)
Fe1—C12	2.1977 (7)	N11—C15	1.368 (3)
N1—C1	1.324 (3)	N11—H11N	0.9444
N1—C5	1.365 (3)	C11—C12	1.381 (3)
N1—H1N	0.9560	C11—H11	0.9300
C1—C2	1.372 (3)	C12—C13	1.351 (3)
C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.358 (4)	C13—C14	1.404 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.396 (3)	C14—C16	1.401 (3)
C3—H3	0.9300	C14—C15	1.411 (3)
C4—C6	1.403 (3)	C15—C19	1.404 (3)
C4—C5	1.405 (3)	C16—C17	1.338 (3)
C5—C9	1.413 (3)	C16—H16	0.9300
C6—C7	1.338 (4)	C17—C18	1.399 (4)
C6—H6	0.9300	C17—H17	0.9300
C7—C8	1.412 (4)	C18—C19	1.364 (3)
C7—H7	0.9300	C18—H18	0.9300
C8—C9	1.362 (3)	C19—C20	1.505 (3)
C8—H8	0.9300	C20—H20A	0.9600
C9—C10	1.493 (3)	C20—H20B	0.9600
C10—H10A	0.9600	C20—H20C	0.9600

supplementary materials

C15—Fe1—C11	108.91 (3)	C9—C10—H10C	109.5
C15—Fe1—C14	112.49 (3)	H10A—C10—H10C	109.5
C11—Fe1—C14	108.72 (3)	H10B—C10—H10C	109.5
C15—Fe1—C12	108.64 (3)	C11—N11—C15	123.54 (18)
C11—Fe1—C12	108.12 (3)	C11—N11—H11N	118.3
C14—Fe1—C12	109.85 (3)	C15—N11—H11N	118.2
C1—N1—C5	122.81 (19)	N11—C11—C12	120.6 (2)
C1—N1—H1N	115.9	N11—C11—H11	119.7
C5—N1—H1N	121.3	C12—C11—H11	119.7
N1—C1—C2	120.8 (2)	C13—C12—C11	119.0 (2)
N1—C1—H1	119.6	C13—C12—H12	120.5
C2—C1—H1	119.6	C11—C12—H12	120.5
C3—C2—C1	118.9 (2)	C12—C13—C14	121.1 (2)
C3—C2—H2	120.5	C12—C13—H13	119.4
C1—C2—H2	120.5	C14—C13—H13	119.4
C2—C3—C4	121.1 (2)	C16—C14—C13	122.9 (2)
C2—C3—H3	119.4	C16—C14—C15	118.5 (2)
C4—C3—H3	119.4	C13—C14—C15	118.6 (2)
C3—C4—C6	122.7 (2)	N11—C15—C19	121.17 (18)
C3—C4—C5	118.3 (2)	N11—C15—C14	117.16 (18)
C6—C4—C5	118.9 (2)	C19—C15—C14	121.67 (19)
N1—C5—C4	117.97 (19)	C17—C16—C14	120.0 (2)
N1—C5—C9	120.19 (19)	C17—C16—H16	120.0
C4—C5—C9	121.8 (2)	C14—C16—H16	120.0
C7—C6—C4	119.5 (2)	C16—C17—C18	120.7 (2)
C7—C6—H6	120.2	C16—C17—H17	119.7
C4—C6—H6	120.2	C18—C17—H17	119.7
C6—C7—C8	121.2 (2)	C19—C18—C17	122.6 (2)
C6—C7—H7	119.4	C19—C18—H18	118.7
C8—C7—H7	119.4	C17—C18—H18	118.7
C9—C8—C7	122.1 (2)	C18—C19—C15	116.5 (2)
C9—C8—H8	118.9	C18—C19—C20	121.8 (2)
C7—C8—H8	118.9	C15—C19—C20	121.7 (2)
C8—C9—C5	116.4 (2)	C19—C20—H20A	109.5
C8—C9—C10	122.8 (2)	C19—C20—H20B	109.5
C5—C9—C10	120.7 (2)	H20A—C20—H20B	109.5
C9—C10—H10A	109.5	C19—C20—H20C	109.5
C9—C10—H10B	109.5	H20A—C20—H20C	109.5
H10A—C10—H10B	109.5	H20B—C20—H20C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots C199 ⁱ	0.96	2.15	3.0490 (18)	156
N11—H11N \cdots C199	0.94	2.15	3.0143 (18)	151

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

Geometry of stacking interactions (Å, °).

$Cg(I)\cdots Cg(J)$	$Cg\cdots Cg$	α	β	γ	$Cg(I)_p$	$Cg(J)_p$
$Cg(N1)\cdots Cg(C19)$	3.8676	5.10	24.54	29.63	3.362	3.518
$Cg(C9)\cdots Cg(N11)$	3.8044	4.49	26.30	22.23	3.522	3.411
$Cg(C9)\cdots Cg(C19)$	3.7121	4.03	18.93	18.86	3.513	3.511

$Cg(Z)$ is the centroid of the six-membered ring containing atom Z . $Cg\cdots Cg$ is the distance between ring centroids, α is the dihedral angle between planes I and J, β is the angle between the $Cg(I)-Cg(J)$ vector and the normal to plane I, γ is the angle between the $Cg(I)-Cg(J)$ vector and the normal to plane J, $Cg(I)_p$ is the perpendicular distance of $Cg(I)$ from ring J and $Cg(J)_p$ is the perpendicular distance of $Cg(J)$ from ring I.

Fig. 1

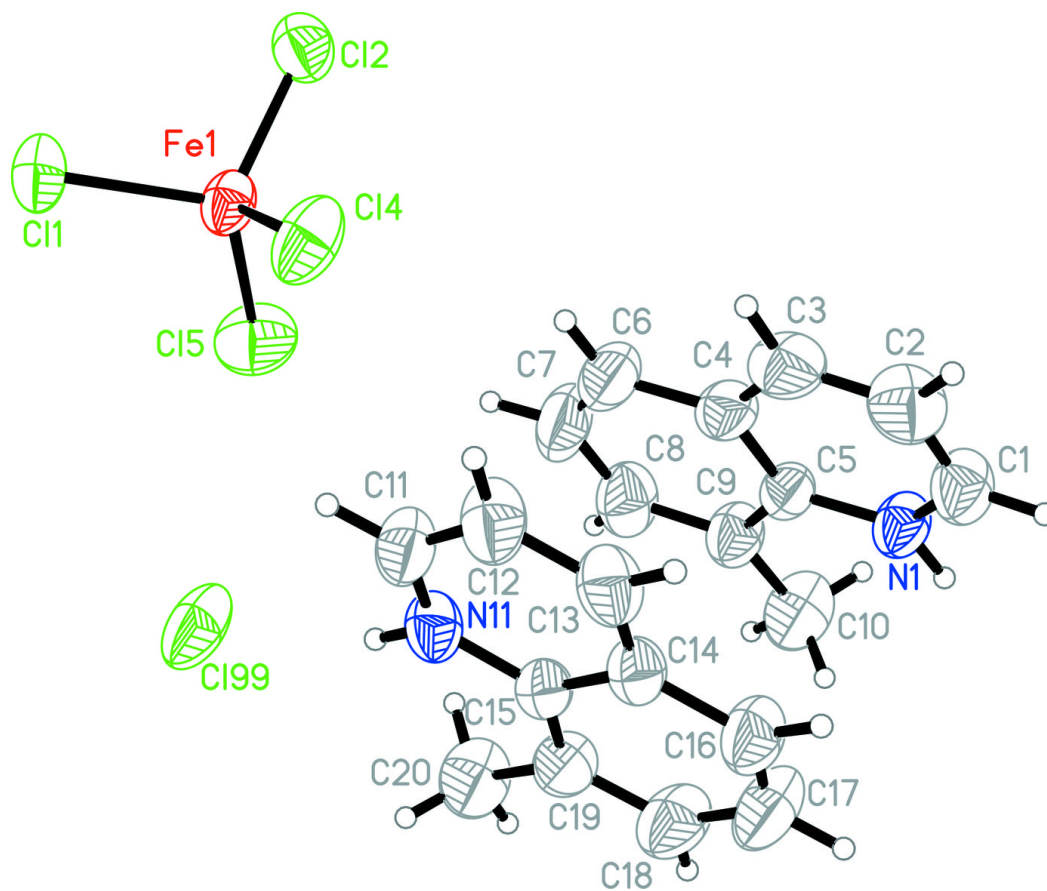


Fig. 2

