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# 2-[(*R*)-Hydroxy(6-methoxyquinolinium-4-yl)methyl]-8-vinyl-1-azoniabicyclo-[2.2.2]octane tetrachloridoferrate(III) chloride monohydrate

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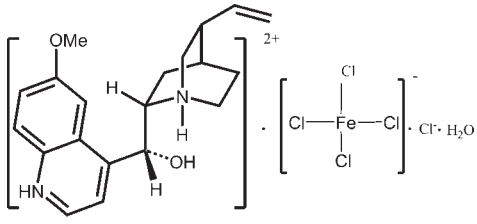
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.048; $wR$ factor = 0.118; data-to-parameter ratio = 18.4.

In the title salt, $(C_{20}H_{26}N_2O_2)[FeCl_4]Cl\cdot H_2O$, the $Fe^{III}$ atom exists in a tetrahedral coordination environment. The cation, anions and water molecules are linked by N—H···Cl, O—H···Cl and O—H···O hydrogen bonds into a layer network.

## Related literature

For ferroelectricity and SHG of chiral coordination compounds, see: Fu *et al.* (2007); Qu *et al.* (2003). For related transition-metal complexes, see: Zhao *et al.* (2003).




## Experimental

### Crystal data

$(C_{20}H_{26}N_2O_2)[FeCl_4]Cl\cdot H_2O$
$M_r$ = 577.54
Monoclinic, $P2_1$
$a$ = 6.6838 (10) Å
$b$ = 18.843 (2) Å
$c$ = 10.8716 (10) Å
$\beta$ = 104.918 (17)°
$V$ = 1323.1 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 1.10 mm$^{-1}$
$T$ = 293 K
0.30 × 0.26 × 0.22 mm

### Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min}$ = 0.82, $T_{max}$ = 0.88
12145 measured reflections
5166 independent reflections
3650 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.118
$S$ = 1.01
5166 reflections
281 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$
Absolute structure: Flack (1983), 2490 Friedel pairs
Flack parameter: 0.01 (2)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···Cl5 | 0.96 | 2.10 | 3.023 (4) | 161 |
| N2—H2$C$···Cl5$^{i}$ | 0.96 | 2.08 | 3.039 (4) | 173 |
| O2—H2$B$···O3 | 0.85 | 2.00 | 2.799 (6) | 156 |
| O3—H3$B$···Cl5$^{ii}$ | 0.85 | 2.71 | 3.070 (6) | 108 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2725).

## References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fu, D.-W., Song, Y.-M., Wang, G.-X., Ye, Q., Xiong, R.-G., Akutagawa, T., Nakamura, T., Chan, P. W. H. & Huang, S. D. (2007). *J. Am. Chem. Soc.* **129**, 5346–5347.
Qu, Z.-R., Chen, Z.-F., Zhang, J., Xiong, R.-G., Abrahams, B. F. & Xue, Z.-L. (2003). *Organometallics* **22**, 2814–2816.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhao, H., Qu, Z.-R., Ye, Q., Abrahams, B. F., Wang, Y.-P., Liu, Z. G., Xue, Z.-L., Xiong, R.-G. & You, X.-Z. (2003). *Chem. Mater.* **15**, 4166–4168.

# supporting information

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# 2-[(*R*)-Hydroxy(6-methoxyquinolinium-4-yl)methyl]-8-vinyl-1-azoniabicyclo-[2.2.2]octane tetrachloridoferrate(III) chloride monohydrate

**Li-Zhuang Chen and Mei-Na Huang**

## S1. Comment

The existence of a chiral centre in an organic ligand is very important for the construction noncentrosymmetric or chiral coordination polymers that exhibit desirable physical properties such as ferroelectricity (Fu *et al.*, 2007), Chiral quinine has a chiral centre which have shown tremendous scope in the synthesis of transition-metal complexes (Zhao *et al.*, 2003; Qu *et al.*,2003). The construction of new members of this family of ligands is an important direction in the development of modern coordination chemistry. We report here the crystal structure of the title compound

The asymmetric unit of the title compound,$C_{20}H_{26}N_2O_2.FeCl_4.Cl.H_2O$(Fig.1), consists of one protoned quinine and a tetrachloro-ironanion with the $Fe^{III}$ ion in a slightly distorted tetrahedral coordination environment, The crystal structure is stabilized by intermolecular N—H···Cl, O—H···Cl and O—H···O hydrogen bonds.The H-bonds form of1D chain viewedalong the *a*-axis (Fig.2).

## S2. Experimental

A mixture of quinine (1 mmol, 0.324 g ), $FeCl_3$(1 mmol, 0.156 g) and 10% aqueous HCl (6 ml) were mixed and dissolved in 20 ml water by heating to 353 K (0.5 h) forming a clear solution. The reaction mixture was cooled slowly to room temperature, crystals of the title compound were formed after 11 days.

## S3. Refinement

All H atoms of quinine were placed in calculated positions , with C—H = 0.93-0.98 Å O—H = 0.85 Å and N—H = 0.96 Å, and re?ned using a riding model, with $U_{iso}$(H)=1.2$U_{eq}$(C, N, O) or 1.5 $U_{eq}$(C) for methyl H atoms.H3A and H3B were located in difference fourier maps.

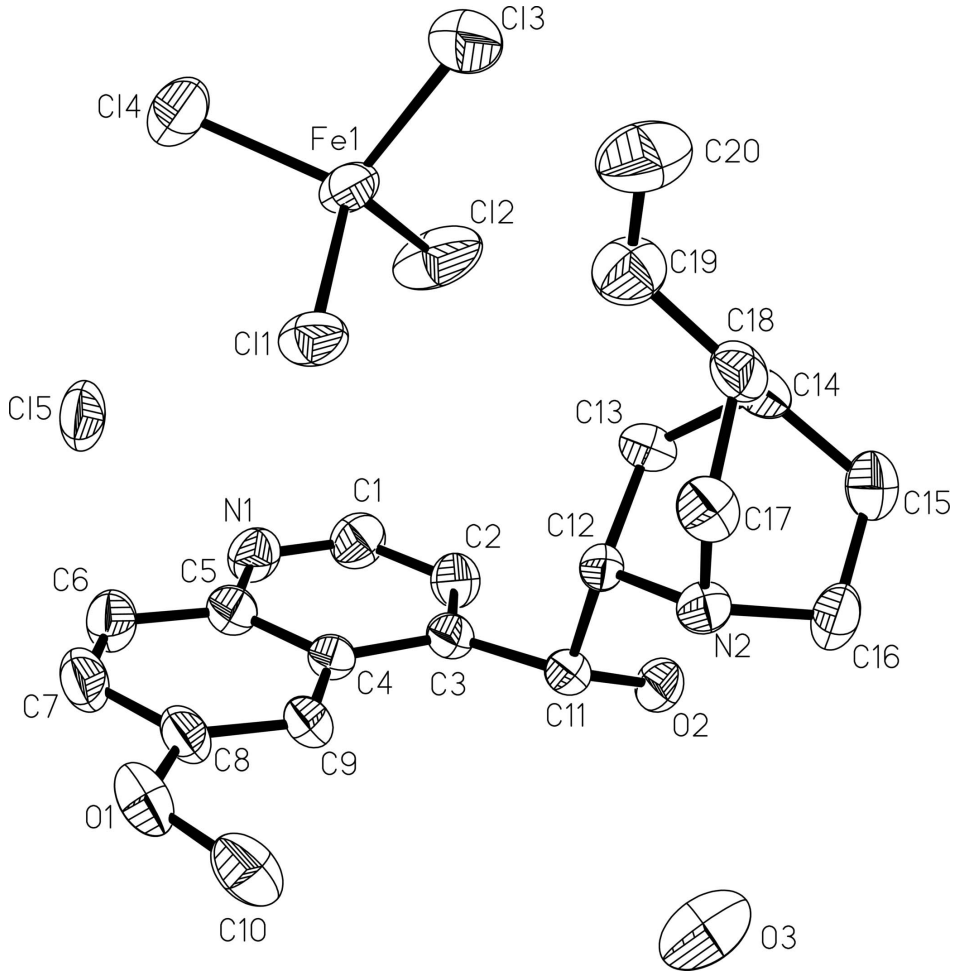


**Figure 1**

The asymmetric unit of the title compound with atom labels. Displacement ellipsoids were drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity.

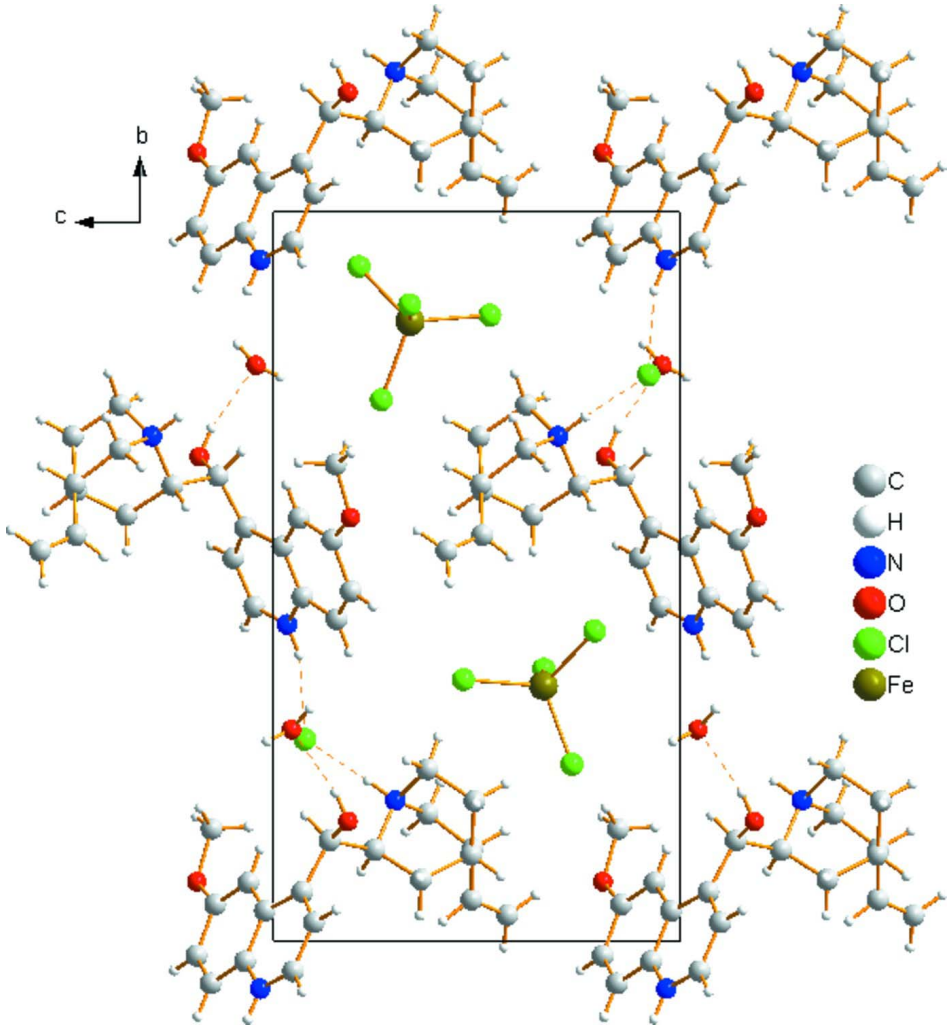


**Figure 2**

The packing viewed along the c axis. Hydrogen bonds are drawn as dashed lines

**2-[(*R*)-Hydroxy(6-methoxyquinolinium-4-yl)methyl]-8-vinyl-1- azoniabicyclo[2.2.2]octane tetrachloridoferrate(III) chloride monohydrate**

*Crystal data*

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Hall symbol: P 2yb
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$b$ = 18.843 (2) Å
$c$ = 10.8716 (10) Å
$\beta$ = 104.918 (17)°
$V$ = 1323.1 (3) Å$^3$
$Z$ = 2
$F(000)$ = 594
$D_x$ = 1.450 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3650 reflections
$\theta$ = 2.9–26.0°
$\mu$ = 1.10 mm$^{-1}$
$T$ = 293 K
Block, yellow
0.30 × 0.26 × 0.22 mm

*Data collection*

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min}$ = 0.82, $T_{max}$ = 0.88
12145 measured reflections
5166 independent reflections
3650 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 2.9°
$h$ = −8→8
$k$ = −23→23
$l$ = −13→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.118
$S$ = 1.01
5166 reflections
281 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$
Absolute structure: Flack (1983), 2490 Friedel pairs
Absolute structure parameter: 0.01 (2)

*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**. 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.1691 (8) | 0.4587 (3) | 0.0478 (5) | 0.0721 (13) |
| H1A | 0.0700 | 0.4306 | 0.0706 | 0.087* |
| C2 | 0.2105 (7) | 0.5255 (3) | 0.0993 (4) | 0.0631 (11) |
| H2A | 0.1359 | 0.5426 | 0.1544 | 0.076* |
| C3 | 0.3610 (6) | 0.5673 (2) | 0.0701 (4) | 0.0520 (10) |
| C4 | 0.4661 (6) | 0.5415 (2) | −0.0214 (4) | 0.0508 (9) |
| C5 | 0.4159 (7) | 0.4721 (3) | −0.0707 (4) | 0.0623 (11) |
| C6 | 0.5180 (8) | 0.4417 (3) | −0.1549 (4) | 0.0762 (14) |
| H6A | 0.4841 | 0.3962 | −0.1865 | 0.091* |
| C7 | 0.6682 (8) | 0.4797 (3) | −0.1902 (4) | 0.0750 (14) |
| H7A | 0.7391 | 0.4595 | −0.2447 | 0.090* |
| C8 | 0.7173 (8) | 0.5502 (3) | −0.1442 (4) | 0.0646 (12) |
| C9 | 0.6189 (7) | 0.5800 (2) | −0.0622 (3) | 0.0567 (10) |
| H9A | 0.6521 | 0.6260 | −0.0327 | 0.068* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.9150 (9) | 0.6537 (3) | −0.1527 (5) | 0.0857 (16) |
| H10A | 1.0218 | 0.6701 | −0.1901 | 0.129* |
| H10B | 0.9624 | 0.6567 | −0.0616 | 0.129* |
| H10C | 0.7938 | 0.6826 | −0.1818 | 0.129* |
| C11 | 0.4201 (6) | 0.6379 (2) | 0.1375 (3) | 0.0508 (9) |
| H11A | 0.4540 | 0.6721 | 0.0781 | 0.061* |
| C12 | 0.6126 (6) | 0.62488 (19) | 0.2487 (3) | 0.0467 (9) |
| H12A | 0.7089 | 0.5959 | 0.2156 | 0.056* |
| C13 | 0.5696 (7) | 0.5844 (2) | 0.3628 (4) | 0.0565 (10) |
| H13A | 0.4229 | 0.5861 | 0.3583 | 0.068* |
| H13B | 0.6098 | 0.5351 | 0.3601 | 0.068* |
| C14 | 0.6917 (7) | 0.6182 (3) | 0.4861 (4) | 0.0622 (11) |
| H14A | 0.6783 | 0.5893 | 0.5585 | 0.075* |
| C15 | 0.6009 (9) | 0.6922 (3) | 0.4946 (5) | 0.0815 (14) |
| H15A | 0.4575 | 0.6881 | 0.4976 | 0.098* |
| H15B | 0.6774 | 0.7157 | 0.5718 | 0.098* |
| C16 | 0.6143 (8) | 0.7356 (3) | 0.3786 (5) | 0.0770 (14) |
| H16A | 0.4763 | 0.7477 | 0.3285 | 0.092* |
| H16B | 0.6894 | 0.7793 | 0.4058 | 0.092* |
| C17 | 0.9372 (7) | 0.6747 (3) | 0.3784 (4) | 0.0672 (12) |
| H17A | 1.0108 | 0.7180 | 0.4108 | 0.081* |
| H17B | 1.0137 | 0.6506 | 0.3261 | 0.081* |
| C18 | 0.9227 (7) | 0.6269 (3) | 0.4897 (4) | 0.0667 (12) |
| H18A | 0.9909 | 0.6513 | 0.5689 | 0.080* |
| C19 | 1.0326 (9) | 0.5569 (3) | 0.4871 (6) | 0.0859 (16) |
| H19A | 0.9833 | 0.5274 | 0.4172 | 0.103* |
| C20 | 1.1894 (11) | 0.5353 (4) | 0.5747 (7) | 0.126 (3) |
| H20A | 1.2428 | 0.5634 | 0.6459 | 0.151* |
| H20B | 1.2493 | 0.4915 | 0.5667 | 0.151* |
| N1 | 0.2684 (6) | 0.4343 (2) | −0.0331 (4) | 0.0677 (10) |
| H1B | 0.2353 | 0.3875 | −0.0673 | 0.081* |
| N2 | 0.7244 (6) | 0.69263 (18) | 0.2997 (3) | 0.0574 (9) |
| H2C | 0.7373 | 0.7211 | 0.2289 | 0.069* |
| O1 | 0.8655 (5) | 0.5813 (2) | −0.1892 (3) | 0.0807 (10) |
| O2 | 0.2546 (5) | 0.66425 (17) | 0.1827 (3) | 0.0638 (8) |
| H2B | 0.2211 | 0.7051 | 0.1509 | 0.096* |
| Cl5 | 0.2012 (3) | 0.27667 (8) | −0.07702 (16) | 0.1153 (6) |
| Cl3 | 0.8401 (4) | 0.35869 (11) | 0.53267 (16) | 0.1329 (7) |
| Cl4 | 0.6722 (3) | 0.24251 (8) | 0.26529 (17) | 0.1000 (5) |
| Cl2 | 0.3307 (3) | 0.37297 (11) | 0.3350 (2) | 0.1408 (9) |
| Fe1 | 0.64976 (11) | 0.35002 (3) | 0.33590 (7) | 0.0748 (2) |
| Cl1 | 0.7554 (2) | 0.42527 (7) | 0.21006 (13) | 0.0801 (4) |
| O3 | 0.2399 (9) | 0.7902 (3) | 0.0442 (6) | 0.156 (2) |
| H3B | 0.1900 | 0.8190 | 0.0887 | 0.234* |
| H3A | 0.1429 | 0.7743 | −0.0162 | 0.234* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.073 (3) | 0.061 (3) | 0.081 (3) | −0.006 (2) | 0.017 (3) | −0.003 (3) |
| C2 | 0.062 (3) | 0.068 (3) | 0.064 (3) | 0.005 (2) | 0.026 (2) | −0.002 (2) |
| C3 | 0.056 (2) | 0.056 (2) | 0.043 (2) | 0.0083 (18) | 0.0107 (19) | 0.0022 (18) |
| C4 | 0.056 (2) | 0.052 (2) | 0.040 (2) | 0.0091 (18) | 0.0052 (19) | 0.0009 (18) |
| C5 | 0.070 (3) | 0.065 (3) | 0.048 (2) | 0.010 (2) | 0.008 (2) | 0.000 (2) |
| C6 | 0.089 (4) | 0.073 (3) | 0.065 (3) | 0.011 (3) | 0.017 (3) | −0.015 (3) |
| C7 | 0.083 (4) | 0.094 (4) | 0.045 (2) | 0.018 (3) | 0.012 (3) | −0.015 (2) |
| C8 | 0.075 (3) | 0.082 (3) | 0.038 (2) | 0.010 (2) | 0.015 (2) | −0.004 (2) |
| C9 | 0.068 (3) | 0.064 (3) | 0.038 (2) | 0.005 (2) | 0.013 (2) | −0.0013 (19) |
| C10 | 0.090 (4) | 0.110 (5) | 0.063 (3) | −0.019 (3) | 0.033 (3) | 0.002 (3) |
| C11 | 0.060 (3) | 0.051 (2) | 0.042 (2) | 0.0082 (18) | 0.0150 (19) | 0.0027 (18) |
| C12 | 0.059 (2) | 0.0405 (19) | 0.0438 (19) | 0.0040 (16) | 0.0192 (18) | −0.0006 (16) |
| C13 | 0.056 (2) | 0.065 (3) | 0.046 (2) | −0.003 (2) | 0.0091 (19) | 0.010 (2) |
| C14 | 0.063 (3) | 0.080 (3) | 0.044 (2) | 0.002 (2) | 0.014 (2) | 0.009 (2) |
| C15 | 0.095 (4) | 0.083 (4) | 0.073 (3) | 0.011 (3) | 0.034 (3) | −0.015 (3) |
| C16 | 0.094 (4) | 0.056 (3) | 0.080 (3) | 0.010 (2) | 0.020 (3) | −0.019 (2) |
| C17 | 0.065 (3) | 0.073 (3) | 0.064 (3) | −0.012 (2) | 0.017 (2) | −0.004 (2) |
| C18 | 0.063 (3) | 0.079 (3) | 0.049 (2) | 0.002 (2) | −0.003 (2) | −0.010 (2) |
| C19 | 0.068 (3) | 0.087 (4) | 0.094 (4) | 0.002 (3) | 0.006 (3) | 0.001 (3) |
| C20 | 0.107 (5) | 0.117 (5) | 0.140 (6) | 0.028 (4) | 0.006 (5) | 0.027 (5) |
| N1 | 0.076 (3) | 0.053 (2) | 0.071 (2) | −0.0045 (19) | 0.012 (2) | −0.006 (2) |
| N2 | 0.069 (2) | 0.0482 (19) | 0.057 (2) | −0.0006 (16) | 0.0193 (18) | 0.0030 (16) |
| O1 | 0.085 (2) | 0.109 (3) | 0.0543 (18) | −0.005 (2) | 0.0304 (17) | −0.0106 (19) |
| O2 | 0.0635 (18) | 0.0644 (19) | 0.0641 (17) | 0.0230 (15) | 0.0178 (15) | −0.0030 (15) |
| Cl5 | 0.1995 (19) | 0.0617 (8) | 0.1125 (12) | 0.0004 (9) | 0.0904 (13) | −0.0178 (8) |
| Cl3 | 0.201 (2) | 0.1015 (12) | 0.0886 (10) | 0.0237 (14) | 0.0246 (11) | 0.0161 (10) |
| Cl4 | 0.1138 (11) | 0.0722 (8) | 0.1292 (13) | 0.0163 (8) | 0.0588 (10) | 0.0050 (8) |
| Cl2 | 0.1171 (13) | 0.1379 (16) | 0.203 (2) | 0.0635 (11) | 0.1064 (14) | 0.0824 (15) |
| Fe1 | 0.0851 (5) | 0.0634 (4) | 0.0877 (5) | 0.0254 (4) | 0.0437 (4) | 0.0243 (4) |
| Cl1 | 0.0798 (8) | 0.0780 (8) | 0.0897 (8) | 0.0127 (6) | 0.0352 (7) | 0.0281 (7) |
| O3 | 0.163 (5) | 0.090 (3) | 0.211 (6) | 0.004 (3) | 0.040 (4) | 0.019 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.314 (6) | C13—H13B | 0.9700 |
| C1—C2 | 1.377 (7) | C14—C15 | 1.533 (7) |
| C1—H1A | 0.9300 | C14—C18 | 1.543 (7) |
| C2—C3 | 1.378 (6) | C14—H14A | 0.9800 |
| C2—H2A | 0.9300 | C15—C16 | 1.525 (7) |
| C3—C4 | 1.442 (6) | C15—H15A | 0.9700 |
| C3—C11 | 1.521 (6) | C15—H15B | 0.9700 |
| C4—C9 | 1.414 (6) | C16—N2 | 1.503 (6) |
| C4—C5 | 1.420 (6) | C16—H16A | 0.9700 |
| C5—N1 | 1.362 (6) | C16—H16B | 0.9700 |
| C5—C6 | 1.397 (6) | C17—N2 | 1.497 (6) |

| | | | |
|---|---|---|---|
| C6—C7 | 1.367 (7) | C17—C18 | 1.531 (7) |
| C6—H6A | 0.9300 | C17—H17A | 0.9700 |
| C7—C8 | 1.428 (7) | C17—H17B | 0.9700 |
| C7—H7A | 0.9300 | C18—C19 | 1.513 (7) |
| C8—O1 | 1.346 (6) | C18—H18A | 0.9800 |
| C8—C9 | 1.358 (6) | C19—C20 | 1.288 (8) |
| C9—H9A | 0.9300 | C19—H19A | 0.9300 |
| C10—O1 | 1.435 (7) | C20—H20A | 0.9300 |
| C10—H10A | 0.9600 | C20—H20B | 0.9300 |
| C10—H10B | 0.9600 | N1—H1B | 0.9599 |
| C10—H10C | 0.9600 | N2—H2C | 0.9601 |
| C11—O2 | 1.411 (5) | O2—H2B | 0.8499 |
| C11—C12 | 1.541 (5) | Cl3—Fe1 | 2.196 (2) |
| C11—H11A | 0.9800 | Cl4—Fe1 | 2.1852 (16) |
| C12—N2 | 1.511 (5) | Cl2—Fe1 | 2.1734 (17) |
| C12—C13 | 1.545 (5) | Fe1—Cl1 | 2.2085 (13) |
| C12—H12A | 0.9800 | O3—H3B | 0.8501 |
| C13—C14 | 1.517 (6) | O3—H3A | 0.8499 |
| C13—H13A | 0.9700 | | |

| | | | |
|---|---|---|---|
| N1—C1—C2 | 120.6 (5) | C15—C14—C18 | 108.1 (4) |
| N1—C1—H1A | 119.7 | C13—C14—H14A | 109.7 |
| C2—C1—H1A | 119.7 | C15—C14—H14A | 109.7 |
| C1—C2—C3 | 120.8 (4) | C18—C14—H14A | 109.7 |
| C1—C2—H2A | 119.6 | C16—C15—C14 | 109.2 (4) |
| C3—C2—H2A | 119.6 | C16—C15—H15A | 109.8 |
| C2—C3—C4 | 118.7 (4) | C14—C15—H15A | 109.8 |
| C2—C3—C11 | 120.2 (4) | C16—C15—H15B | 109.8 |
| C4—C3—C11 | 121.0 (4) | C14—C15—H15B | 109.8 |
| C9—C4—C5 | 118.3 (4) | H15A—C15—H15B | 108.3 |
| C9—C4—C3 | 124.2 (4) | N2—C16—C15 | 108.9 (4) |
| C5—C4—C3 | 117.5 (4) | N2—C16—H16A | 109.9 |
| N1—C5—C6 | 119.7 (5) | C15—C16—H16A | 109.9 |
| N1—C5—C4 | 119.2 (4) | N2—C16—H16B | 109.9 |
| C6—C5—C4 | 121.1 (5) | C15—C16—H16B | 109.9 |
| C7—C6—C5 | 119.1 (5) | H16A—C16—H16B | 108.3 |
| C7—C6—H6A | 120.4 | N2—C17—C18 | 109.9 (4) |
| C5—C6—H6A | 120.4 | N2—C17—H17A | 109.7 |
| C6—C7—C8 | 120.6 (4) | C18—C17—H17A | 109.7 |
| C6—C7—H7A | 119.7 | N2—C17—H17B | 109.7 |
| C8—C7—H7A | 119.7 | C18—C17—H17B | 109.7 |
| O1—C8—C9 | 125.8 (5) | H17A—C17—H17B | 108.2 |
| O1—C8—C7 | 113.7 (4) | C19—C18—C17 | 111.6 (4) |
| C9—C8—C7 | 120.5 (5) | C19—C18—C14 | 113.2 (4) |
| C8—C9—C4 | 120.3 (4) | C17—C18—C14 | 108.0 (4) |
| C8—C9—H9A | 119.8 | C19—C18—H18A | 107.9 |
| C4—C9—H9A | 119.8 | C17—C18—H18A | 107.9 |
| O1—C10—H10A | 109.5 | C14—C18—H18A | 107.9 |

| | | | |
|---|---|---|---|
| O1—C10—H10B | 109.5 | C20—C19—C18 | 124.7 (6) |
| H10A—C10—H10B | 109.5 | C20—C19—H19A | 117.7 |
| O1—C10—H10C | 109.5 | C18—C19—H19A | 117.7 |
| H10A—C10—H10C | 109.5 | C19—C20—H20A | 120.0 |
| H10B—C10—H10C | 109.5 | C19—C20—H20B | 120.0 |
| O2—C11—C3 | 110.2 (4) | H20A—C20—H20B | 120.0 |
| O2—C11—C12 | 110.7 (3) | C1—N1—C5 | 123.2 (4) |
| C3—C11—C12 | 107.4 (3) | C1—N1—H1B | 118.4 |
| O2—C11—H11A | 109.5 | C5—N1—H1B | 118.4 |
| C3—C11—H11A | 109.5 | C17—N2—C16 | 109.1 (3) |
| C12—C11—H11A | 109.5 | C17—N2—C12 | 109.1 (3) |
| N2—C12—C11 | 112.8 (3) | C16—N2—C12 | 113.4 (4) |
| N2—C12—C13 | 107.4 (3) | C17—N2—H2C | 108.4 |
| C11—C12—C13 | 114.7 (3) | C16—N2—H2C | 108.4 |
| N2—C12—H12A | 107.2 | C12—N2—H2C | 108.4 |
| C11—C12—H12A | 107.2 | C8—O1—C10 | 116.8 (4) |
| C13—C12—H12A | 107.2 | C11—O2—H2B | 109.0 |
| C14—C13—C12 | 109.5 (3) | Cl2—Fe1—Cl4 | 109.81 (9) |
| C14—C13—H13A | 109.8 | Cl2—Fe1—Cl3 | 108.14 (9) |
| C12—C13—H13A | 109.8 | Cl4—Fe1—Cl3 | 109.65 (7) |
| C14—C13—H13B | 109.8 | Cl2—Fe1—Cl1 | 109.82 (6) |
| C12—C13—H13B | 109.8 | Cl4—Fe1—Cl1 | 108.41 (6) |
| H13A—C13—H13B | 108.2 | Cl3—Fe1—Cl1 | 111.00 (8) |
| C13—C14—C15 | 107.7 (4) | H3B—O3—H3A | 109.5 |
| C13—C14—C18 | 112.0 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···Cl5 | 0.96 | 2.10 | 3.023 (4) | 161 |
| N2—H2*C*···Cl5$^{i}$ | 0.96 | 2.08 | 3.039 (4) | 173 |
| O2—H2*B*···O3 | 0.85 | 2.00 | 2.799 (6) | 156 |
| O3—H3*B*···Cl5$^{ii}$ | 0.85 | 2.71 | 3.070 (6) | 108 |

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