

Quinolinium tetrachloroferrate(III)

Dariusz Wyrzykowski,^a Artur Sikorski,^{a*} Antoni Konitz^{a,b} and Zygmunta Warnke^a^aUniversity of Gdańsk, Faculty of Chemistry, J. Sobieskiego 18, 80-952 Gdańsk, Poland, and ^bGdańsk University of Technology, Department of Inorganic Chemistry, G. Narutowicza 11/12, 80-952 Gdańsk, Poland

Correspondence e-mail: art@chem.univ.gda.pl

Key indicators

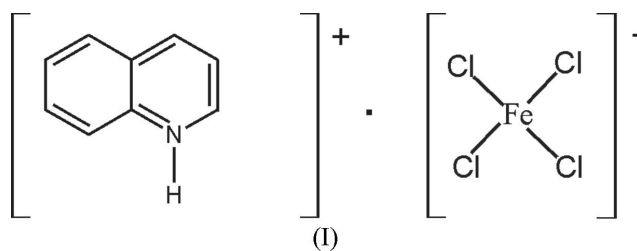
Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
Disorder in main residue
 R factor = 0.040
 wR factor = 0.101
Data-to-parameter ratio = 15.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $(\text{C}_9\text{H}_8\text{N})[\text{FeCl}_4]$, the tetrachloroferrate(III) anion lies on a mirror plane, whereas the quinolinium cation has inversion symmetry as the result of orientational disorder. In the crystal structure, $\text{C}-\text{H}\cdots\text{Cl}$ interactions between the ions form zigzag ribbons along the b axis. These ribbons are linked *via* $\text{Fe}-\text{Cl}\cdots\pi$ interactions, forming a three-dimensional network.

Received 23 October 2006
Accepted 20 November 2006

Comment

Tetrahalogenoferrate(III) ions have attracted the interest of many research teams (Butcher *et al.*, 1990; Bottomley *et al.*, 1984). Owing to the presence of high-spin d^5 Fe^{III} atoms in these compounds, they are likely to become attractive new magnetic materials (Zora *et al.*, 1990; Bentrup *et al.*, 1999). Our interest in compounds containing tetrahalogenoferrate(III) has been sparked by studies on their interaction with non-aqueous solvents (Warnke *et al.*, 2001, 2003) and, more recently, on the thermal stability of compounds incorporating cations of aliphatic and aromatic nitrogen-containing organic bases (Wyrzykowski *et al.*, 2005; Wyrzykowski & Pattek-Janczyk *et al.*, 2006). In this connection, we have recently reported the structure of bis(2-methylquinolinium) tetrachloroferrate(III) chloride (Wyrzykowski & Sikorski *et al.*, 2006). In this paper, we report the crystal structure of the title compound, (I).



The asymmetric unit of (I) consists of half each of a quinolinium cation and a tetrachloroferrate(III) anion (Fig. 1). The $\text{Fe}1/\text{Cl}2/\text{Cl}3$ atoms of the tetrachloroferrate(III) anion lie on a mirror plane. The quinolinium cation has an inversion center at the mid-point of the $\text{C}3-\text{C}3^{\text{ii}}$ bond [symmetry code: (ii) $1-x, 1-y, -z$] as a result of orientational disorder; it is assumed that atoms $\text{C}2$ and $\text{N}2$ share the same position. The geometry of the FeCl_4^- ion is similar to those observed in other compounds (Zora *et al.*, 1990; Barbaro *et al.*, 1992). However, the $\text{C}3-\text{C}4$ bond is slightly shorter than in other quinoline complexes. This may be due to further orientational disorder such that the N atom is located at the $\text{C}4$ or $\text{C}4^{\text{ii}}$ positions.

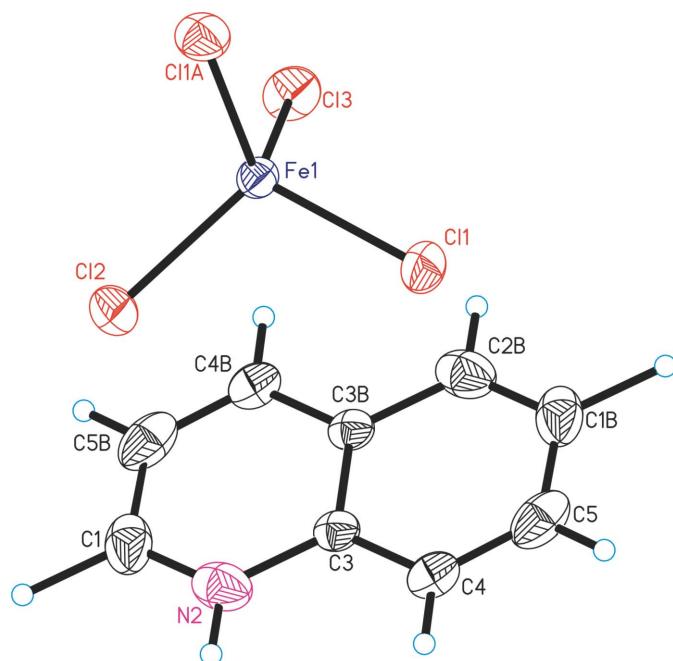


Figure 1
The structure of (I), showing the atom-labelling scheme and 25% probability displacement ellipsoids. [Symmetry codes: (A) $x, \frac{3}{2} - y, z$; (B) $1 - x, 1 - y, -z$.]

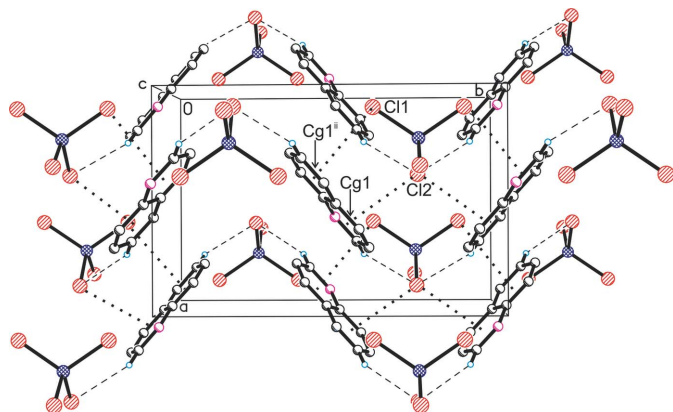


Figure 2
The crystal structure of (I), viewed along the c axis. $C-H \cdots Cl$ interactions are represented by dashed lines and $Fe-Cl \cdots \pi$ interactions by dotted lines. H atoms not involved in these interactions have been omitted. [Symmetry code: (ii) $1 - x, 1 - y, -z$.]

In the crystal structure, ions are linked *via* $C-H \cdots Cl$ interactions (Table 1), forming zigzag ribbons along the b axis (Fig. 2). These ribbons are linked through one of the Cl atoms of the tetrachloroferrate(III) anion, namely Cl2, *via* $Fe-Cl \cdots \pi$ interactions (Table 2) to create a three-dimensional structure.

Experimental

The synthesis of compound (I) was carried out using a procedure similar to that used for other tetrachloroferrates(III) (Warnke *et al.*, 2003). To a solution of $FeCl_3$ (*ca* 0.05 mol) in 96% ethanol (25 ml), a

stoichiometric amount of a 12 M HCl solution was added followed by quinoline (*ca* 0.05 mol). Compound (I) crystallized directly from this mixture as yellow–orange crystals after two weeks in a refrigerator and was dried over P_4O_{10} in a vacuum desiccator (yield *ca* 75%). Elemental analysis (calculated/found %): C 32.46/32.76, H 2.44/2.44, N 4.27/4.17, Cl 43.32/43.55.

Crystal data

$(C_9H_8N)[FeCl_4]$
 $M_r = 327.81$
Orthorhombic, $Pnma$
 $a = 9.211$ (2) Å
 $b = 14.162$ (3) Å
 $c = 9.772$ (2) Å
 $V = 1274.7$ (5) Å³

$Z = 4$
 $D_x = 1.708$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 1.99$ mm⁻¹
 $T = 295$ (2) K
Prism, yellow–orange
 $0.5 \times 0.3 \times 0.2$ mm

Data collection

Kuma KM-4 diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{min} = 0.499$, $T_{max} = 0.672$
1489 measured reflections
1174 independent reflections

640 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.076$
 $\theta_{max} = 25.0^\circ$
3 standard reflections
every 200 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.101$
 $S = 1.00$
1174 reflections
74 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.35$ e Å⁻³
 $\Delta\rho_{min} = -0.36$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0095 (15)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$Cl1-H1 \cdots Cl2^i$	0.93	2.87	3.697 (6)	148

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

Table 2

$Fe-Cl \cdots \pi$ interactions (Å, °) in (I).

Y	X	$C_g(I)$	$X \cdots C_g$	$Y-X \cdots C_g$
Fe1	Cl1	C_g1^{ii}	3.688 (2)	96.2 (1)
Fe1	Cl2	C_g1	3.600 (2)	98.4 (1)

Symmetry codes: (ii) $1-x, 1-y, -z$. C_g1 is the centroid of the ring $C1/N2/C3/C3^{ii}/C4^{ii}/C5^{ii}$.

All H atoms were positioned geometrically and refined using a riding model, with $C-H = 0.93$ Å and $N-H = 0.86$ Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$. Atoms N2 and C2 (and attached H atoms) were refined assuming the same coordinates and displacement parameters, with occupancy factors of 0.5 for each atom.

Data collection: *KM-4 Software* (Oxford Diffraction, 1989); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Johnson, 1976); software used to

prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

This research was supported by the Polish State Committee for Scientific Research under grant DS/8230-4-0088-6 and the European Social Fund project No. ZPORR/2.22/II/2.6/ARP/U/2/05.

References

- Barbaro, P., Bianchini, C., Fochi, M., Masi, D. & Mealli, C. (1992). *Acta Cryst. C* **48**, 625–627.
- Bentrup, U., Feist, M. & Kemnitz, E. (1999). *Prog. Solid State Chem.* **27**, 75–129.
- Bottomley, G. A., Carter, A. M., Engelhardt, L. M., Lincoln, F. J., Patrick, J. M. & White, A. H. (1984). *Aust. J. Chem.* **37**, 871–877.
- Butcher, K. D., Didziulis, S. V., Brait, B. & Solomon, E. I. (1990). *J. Am. Chem. Soc.* **112**, 2231–2242.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst. A* **24**, 351–359.
- Oxford Diffraction (1989). *KM-4 Software User's Guide*. Version 3.1. Oxford Diffraction, Wrocław, Poland.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Warnke, Z., Wawrzyniak, G., Wyrzykowski, D. & Kosmowski, J. (2001). *Pol. J. Chem.* **75**, 759–763.
- Warnke, Z., Wyrzykowski, D. & Wawrzyniak, G. (2003). *Pol. J. Chem.* **77**, 1121–1129.
- Wyrzykowski, D., Maniecki, T., Pattek-Janczyk, A., Stanek, J. & Warnke, Z. (2005). *Thermochim. Acta*, **435**, 92–98.
- Wyrzykowski, D., Pattek-Janczyk, A., Maniecki, T., Zaremba, K. & Warnke, Z. (2006). *Thermochim. Acta*, **443**, 72–77.
- Wyrzykowski, D., Sikorski, A., Lis, T., Konitz, A. & Warnke, Z. (2006). *Acta Cryst. E* **62**, m1737–m1739.
- Zora, J. A., Seddon, K. R., Hitchcock, P. B., Lowe, C. B., Shum, D. P. & Carlin, R. L. (1990). *Inorg. Chem.* **29**, 3302–3308.