Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Tetrabutylammonium tetrachloroferrate(III)

Michael T. Hay[a] and Steven J. Geib[b]*

[a]Penn State Beaver, 100 University Drive, Monaca, PA 15061, USA, and [b]Department of Chemistry, University of Pittsburgh, PA 15260, USA

Correspondence e-mail: geib@pitt.edu

Received 24 November 2004
Accepted 1 December 2004
Online 24 December 2004

In the title compound, $[(C_4H_9)_4N][FeCl_4]$, the central Fe^{III} atom in the anion is tetrahedrally coordinated by four Cl atoms, while the cation exists as a central N atom bonded to four *n*-butyl groups, also in a tetrahedral arrangement. Both the cation and anion lie on crystallographic twofold axes.

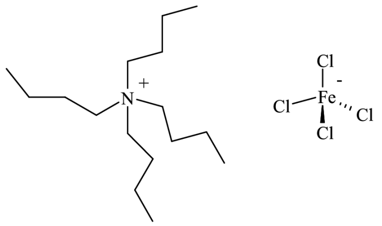
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.173
Data-to-parameter ratio = 27.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Iron-containing compounds are ubiquitous throughout the field of coordination chemistry. Recently, a variety of iron-containing silsesquioxane compounds have been added to the list of iron coordination complexes (Liu *et al.*, 2000; Lorenz *et al.*, 2000; Hay *et al.*, 2003; Shapley *et al.*, 2003). Some of the published iron silsesquioxane compounds are synthesized from tetrabutylammonium tetrachlorferrate(III), $[(C_4H_9)_4N]$-$[FeCl_4]$ (Hay *et al.*, 2003; Shapley *et al.*, 2003). For reasons of completeness, the structural characterization of this useful iron(III) starting material is reported here.



(I)

In the title compound (I), (Fig. 1) both cation and anion reside on crystallographic twofold axes. The Fe^{III} atom in the anion, $[FeCl_4]^-$, is four-coordinate, with a tetrahedral

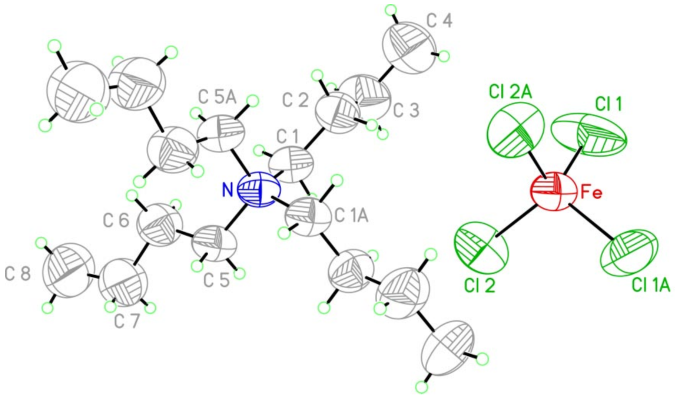


Figure 1
The component ions of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The suffix A corresponds to symmetry code (i) in Table 1.

arrangement of the four chlorides. The Cl—Fe—Cl bond angles are 107.35 (5)–112.19 (10)°, while the Fe—Cl bond lengths are in the range 2.1808 (9)–2.1835 (12) Å. The tetrabutylammonium cation, $(C_4H_9)_4N^+$, also has a tetrahedral arrangement around the central N atom, with C—N—C bond angles in the range 108.46 (12)–111.6 (3)° and independent N—C bond lengths of 1.507 (3) and 1.524 (3) Å. These values are consistent with the structure previously reported for tetraethylammonium tetrachloroferrate(III) (Evans *et al.*, 1990).

Experimental

A solution of $FeCl_3 \cdot 6H_2O$ (7.4 mmol, 2.0 g) was dissolved in 9 *M* aqueous HCl (100.0 ml). To this yellow solution, one equivalent of $(C_{16}H_{36}N)Cl \cdot H_2O$ (7.4 mmol, 2.2 g) was added with stirring. Immediately, a yellow precipitate formed. The yellow precipitate was collected by filtration, and rinsed with diethyl ether. The yellow precipitate was then recrystallized from a mixture of dichloromethane/diethyl ether at 243 K to afford yellow crystals. The yellow crystals were recrystallized a second time from dichloromethane/diethyl ether before they were analyzed.

Crystal data

$(C_{16}H_{36}N)[FeCl_4]$
$M_r = 440.11$
Orthorhombic, *Pnna*
$a = 18.4522$ (9) Å
$b = 11.5264$ (6) Å
$c = 11.4079$ (6) Å
$V = 2426.3$ (2) Å^3
$Z = 4$
$D_x = 1.205$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 999 reflections
$\theta = 5$–15°
$\mu = 1.06$ mm^{-1}
$T = 295$ (2) K
Prism, yellow
0.37 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.695$, $T_{\max} = 0.832$
22 329 measured reflections
2784 independent reflections
1684 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\max} = 27.5°$
$h = -23 \rightarrow 23$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.173$
$S = 1.33$
2784 reflections
103 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.078P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe—Cl2	2.1808 (9)	Fe—Cl1	2.1835 (12)
Cl2^i—Fe—Cl2	111.00 (6)	C1^i—N—C1	111.6 (3)
Cl2^i—Fe—Cl1	109.49 (4)	C1—N—C5^i	108.46 (12)
Cl2—Fe—Cl1	107.35 (5)	C1—N—C5	108.51 (13)
Cl1—Fe—Cl1^i	112.19 (10)	C5^i—N—C5	111.3 (3)

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

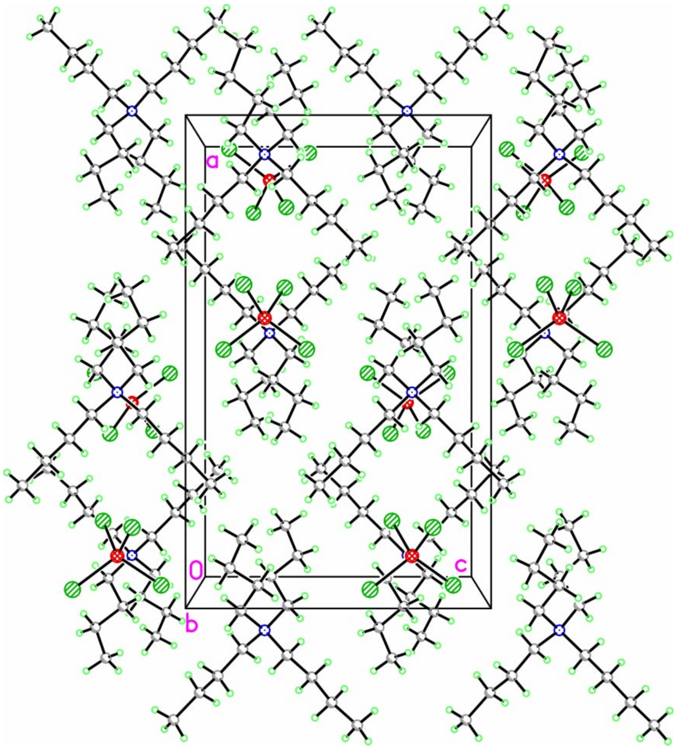


Figure 2
Packing diagram of (I), viewed along the *b* axis.

All H atoms were placed in calculated positions (C—H = 0.96 and 0.97 Å) and refined with riding-model constraints, with $U_{\rm iso}$(H) set to $1.2U_{\rm eq}$(C) [$1.5U_{\rm eq}$(C) for methyl groups].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank the Commonwealth College of the Pennsylvania State University for financial support.

References

Bruker (1997). *SMART* (Version 5.629) and *SAINT* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Evans, D. J., Hills, A., Hughes, D. L. & Leigh, G. J. (1990). *Acta Cryst.* C**46**, 1818–1821.
Hay, M. T., Hainaut, B. J. & Geib, S. J. (2003). *Inorg. Chem. Commun.* **6**, 431–434.
Liu, F., John, K. D., Scott, B. L., Baker, T. R., Ott, K. C. & Tumas, W. (2000). *Angew. Chem. Int. Ed.* **39**, 3127–3130.
Lorenz, V., Fischer, A. & Edelmann, F. T. (2000). *Z. Anorg. Allg. Chem.* **626**, 1728–1730.
Shapley, P. A., Bigham, W. S. & Hay, M. T. (2003). *Inorg. Chim. Acta*, **345**, 255–260.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.