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Butyltriethylammonium tetrachloridoferrate(III)

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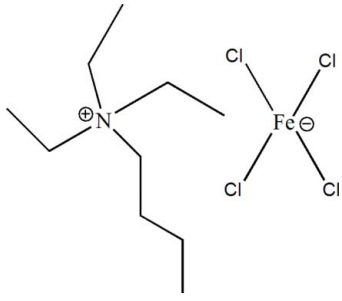
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.117; data-to-parameter ratio = 25.2.

In the title compound, $(C_{10}H_{24}N)[FeCl_4]$, no classical hydrogen bonds are observed. The butyltriethylammonium cations interact with the tetrahedral $[FeCl_4]^-$ anion through weak C—H···Cl interactions, forming a three-dimensional network.

Related literature

For background to molecular–ionic and ferroelectric–dielectric compounds, see: Hay & Geib (2005); Zhang *et al.* (2010).



Experimental

Crystal data

$(C_{10}H_{24}N)[FeCl_4]$ M_r = 355.95
Monoclinic, $P2_1/c$
a = 7.6197 (15) Å
b = 11.671 (2) Å
c = 18.473 (4) Å
β = 91.51 (3)°
V = 1642.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.55 mm^{-1}
T = 293 K
0.28 × 0.24 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.655, T_{max} = 0.734
16819 measured reflections
3754 independent reflections
2766 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.117
S = 1.10
3754 reflections
149 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3B···Cl3^{i}	0.96	2.87	3.790 (4)	162
C4—H4A···Cl1	0.97	2.92	3.859 (3)	163

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

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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2288).

References

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Zhang, W., Chen, L. Z., Gou, M., Li, Y. H., Fu, D. W. & Xiong, R. G. (2010). *Cryst. Growth Des.* **10**, 1025–1027.

supplementary materials

Acta Cryst. (2012). E**68**, m656 [doi:10.1107/S1600536812017047]

Butyltriethylammonium tetrachloridoferrate(III)

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Comment

In our laboratory, we synthesize simple molecular–ionic compounds containing organic-ammonium cations and anions due to the tunability of their special structural features and their ferroelectric-dielectric properties (Hay & Geib, 2005; Zhang *et al.*, 2010). Herein, the crystal structure of the title compound is reported.

The asymmetric unit of the title compound, $(C_{10}H_{24}N^{+}).(FeCl_4^{-})$ consists of one tetrachloroferrate anion unit and one butyltriethylammonium cation (Fig 1). In the structure, the Fe^{III} ion adopts a distorted tetrahedral geometry surrounded by four Cl^{-} anions with the Fe—Cl bond distances being in the range of 2.1611 (9)–2.1823 (10)? and the Cl–Fe–Cl bond angles being in the range of 109.26 (5)–110.18 (5) (5)°. There are no classic hydrogen bonds found, although weak intermolecular C—H···Cl interactions link the butyltriethylammonium cations and the tetrahedral $FeCl_4^{-}$ anion into a three-dimensional network.

Experimental

In room temperaturebutyltriethylammonium(5 mmol,1.17 g) were dissolved in 30 ml water, then $FeCl_3.6H_2O$ (5 mmol, 1.35 g) was added into the previous solution slowly with properly sirring. An orange solid appeared immediately and the solid was collected by filtration. Plate orange single crystals suitable for X-ray structure analysis were obtained by the slow evaporation of the above filtrate after a week in air.

The dielectric constant of the compound as a function of temperature indicates that the permittivity is basically temperature-independent ($\varepsilon = C/(T–T_0)$), suggesting that this compound is not ferroelectric or there may be no distinct phase transition occurring within the measured temperature (below the melting point).

Refinement

H atoms were placed in calculated positions C—H = 0.96 Å and 0.97 Å for Csp^3 atoms), assigned fixed U_{iso} values [U_{iso} = 1.2Ueq(Csp^2/N) and 1.5Ueq(Csp^3)] and allowed to ride.

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); 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: *SHELXTL* (Sheldrick, 2008).

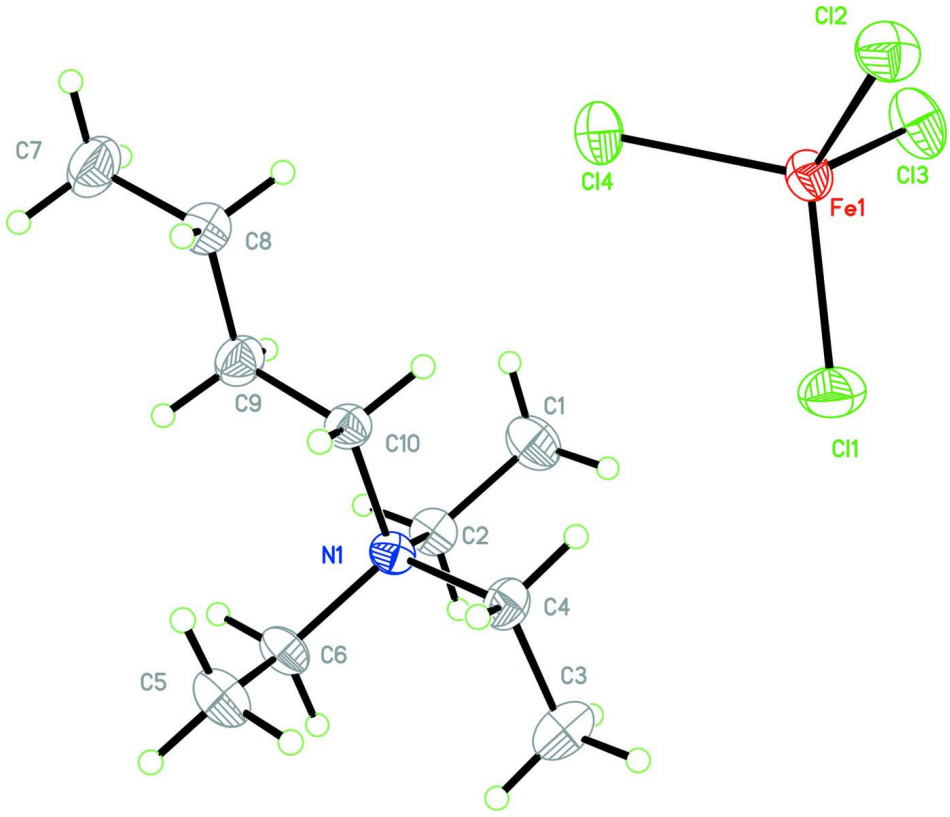

Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme with 30% probability displacement ellipsoids.

Butyltriethylammonium tetrachloridoferrate(III)

Crystal data

$(C_{10}H_{24}N)[FeCl_4]$
$M_r = 355.95$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.6197\ (15)$ Å
$b = 11.671\ (2)$ Å
$c = 18.473\ (4)$ Å
$\beta = 91.51\ (3)°$
$V = 1642.2\ (6)$ Å^3
$Z = 4$
$F(000) = 740$
$D_x = 1.440$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 3.2–27.5°$
$\mu = 1.55$ mm^{-1}
$T = 293$ K
Block, orange
0.28 × 0.24 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.655$, $T_{max} = 0.734$
16819 measured reflections
3754 independent reflections
2766 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -9\rightarrow9$
$k = -15\rightarrow15$
$l = -23\rightarrow23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.117$
$S = 1.10$
3754 reflections
149 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.8101P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3896 (4)	0.6311 (3)	0.58003 (19)	0.0585 (9)
H1A	0.3166	0.6969	0.5712	0.088*
H1B	0.3229	0.5726	0.6033	0.088*
H1C	0.4312	0.6025	0.5349	0.088*
C2	0.5419 (4)	0.6641 (2)	0.62761 (16)	0.0415 (7)
H2A	0.6114	0.5960	0.6375	0.050*
H2B	0.4974	0.6906	0.6734	0.050*
C3	0.8471 (5)	0.6102 (3)	0.5330 (2)	0.0643 (10)
H3A	0.9425	0.6209	0.5673	0.096*
H3B	0.8928	0.5938	0.4862	0.096*
H3C	0.7754	0.5475	0.5481	0.096*
C4	0.7386 (4)	0.7171 (3)	0.52879 (16)	0.0424 (7)
H4A	0.6438	0.7052	0.4935	0.051*
H4B	0.8112	0.7787	0.5110	0.051*
C5	0.9389 (4)	0.8585 (3)	0.63699 (19)	0.0554 (9)
H5A	0.9974	0.8332	0.5944	0.083*
H5B	1.0227	0.8654	0.6765	0.083*
H5C	0.8849	0.9315	0.6278	0.083*
C6	0.8021 (4)	0.7737 (3)	0.65603 (16)	0.0441 (7)
H6A	0.7472	0.7990	0.7001	0.053*
H6B	0.8589	0.7010	0.6664	0.053*
C7	0.2880 (5)	1.0847 (3)	0.6797 (2)	0.0670 (10)
H7A	0.2027	1.0362	0.7017	0.101*
H7B	0.2314	1.1524	0.6612	0.101*
H7C	0.3765	1.1057	0.7152	0.101*
C8	0.3716 (4)	1.0216 (3)	0.61889 (18)	0.0512 (8)

H8A	0.4515	1.0728	0.5947	0.061*
H8B	0.2811	0.9988	0.5839	0.061*
C9	0.4705 (4)	0.9172 (3)	0.64418 (17)	0.0473 (8)
H9A	0.5563	0.9387	0.6815	0.057*
H9B	0.3897	0.8629	0.6649	0.057*
C10	0.5615 (4)	0.8626 (2)	0.58253 (15)	0.0372 (6)
H10A	0.6427	0.9180	0.5631	0.045*
H10B	0.4744	0.8461	0.5447	0.045*
Cl1	0.44676 (12)	0.65139 (8)	0.36000 (6)	0.0695 (3)
Cl2	0.19519 (11)	0.85280 (7)	0.25806 (4)	0.0545 (2)
Cl3	−0.01438 (13)	0.61475 (8)	0.33742 (5)	0.0689 (3)
Cl4	0.15637 (12)	0.84172 (8)	0.44790 (4)	0.0573 (2)
Fe1	0.19494 (6)	0.73961 (4)	0.35064 (2)	0.04185 (15)
N1	0.6607 (3)	0.75470 (18)	0.59891 (11)	0.0334 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0511 (19)	0.062 (2)	0.062 (2)	−0.0149 (16)	−0.0058 (17)	0.0060 (18)
C2	0.0437 (16)	0.0405 (16)	0.0406 (16)	−0.0044 (13)	0.0042 (13)	0.0069 (13)
C3	0.070 (2)	0.054 (2)	0.070 (2)	0.0133 (18)	0.019 (2)	−0.0038 (18)
C4	0.0472 (17)	0.0459 (17)	0.0345 (16)	0.0019 (13)	0.0081 (13)	−0.0029 (13)
C5	0.0479 (19)	0.063 (2)	0.054 (2)	−0.0124 (16)	−0.0105 (16)	0.0036 (17)
C6	0.0431 (17)	0.0526 (19)	0.0360 (17)	−0.0029 (14)	−0.0102 (13)	0.0059 (13)
C7	0.059 (2)	0.060 (2)	0.082 (3)	0.0044 (17)	0.013 (2)	−0.024 (2)
C8	0.0496 (18)	0.0484 (19)	0.055 (2)	0.0031 (14)	−0.0012 (16)	−0.0073 (15)
C9	0.0500 (18)	0.0488 (19)	0.0435 (18)	0.0055 (14)	0.0069 (15)	−0.0042 (14)
C10	0.0403 (15)	0.0357 (15)	0.0356 (15)	0.0025 (12)	0.0003 (12)	0.0038 (12)
Cl1	0.0624 (6)	0.0570 (6)	0.0882 (7)	0.0174 (4)	−0.0131 (5)	−0.0005 (5)
Cl2	0.0583 (5)	0.0617 (5)	0.0438 (4)	0.0026 (4)	0.0076 (4)	0.0148 (4)
Cl3	0.0789 (6)	0.0675 (6)	0.0597 (6)	−0.0330 (5)	−0.0095 (5)	0.0051 (5)
Cl4	0.0683 (6)	0.0638 (6)	0.0397 (4)	−0.0043 (4)	0.0026 (4)	−0.0073 (4)
Fe1	0.0475 (3)	0.0410 (3)	0.0368 (3)	−0.00288 (18)	−0.00255 (19)	0.00161 (18)
N1	0.0337 (12)	0.0370 (13)	0.0294 (12)	0.0002 (10)	0.0008 (10)	0.0057 (9)

Geometric parameters (Å, º)

C1—C2	1.488 (4)	C6—H6B	0.9700
C1—H1A	0.9600	C7—C8	1.498 (5)
C1—H1B	0.9600	C7—H7A	0.9600
C1—H1C	0.9600	C7—H7B	0.9600
C2—N1	1.498 (3)	C7—H7C	0.9600
C2—H2A	0.9700	C8—C9	1.501 (4)
C2—H2B	0.9700	C8—H8A	0.9700
C3—C4	1.497 (4)	C8—H8B	0.9700
C3—H3A	0.9600	C9—C10	1.492 (4)
C3—H3B	0.9600	C9—H9A	0.9700
C3—H3C	0.9600	C9—H9B	0.9700
C4—N1	1.504 (3)	C10—N1	1.496 (3)
C4—H4A	0.9700	C10—H10A	0.9700

C4—H4B	0.9700	C10—H10B	0.9700
C5—C6	1.486 (4)	Cl1—Cl1	0.0000 (18)
C5—H5A	0.9600	Cl1—Fe1	2.1804 (10)
C5—H5B	0.9600	Cl2—Fe1	2.1611 (9)
C5—H5C	0.9600	Cl3—Fe1	2.1695 (10)
C6—N1	1.504 (3)	Cl4—Fe1	2.1823 (10)
C6—H6A	0.9700	Fe1—Cl1	2.1804 (10)
C2—C1—H1A	109.5	C8—C7—H7C	109.5
C2—C1—H1B	109.5	H7A—C7—H7C	109.5
H1A—C1—H1B	109.5	H7B—C7—H7C	109.5
C2—C1—H1C	109.5	C7—C8—C9	112.6 (3)
H1A—C1—H1C	109.5	C7—C8—H8A	109.1
H1B—C1—H1C	109.5	C9—C8—H8A	109.1
C1—C2—N1	116.3 (2)	C7—C8—H8B	109.1
C1—C2—H2A	108.2	C9—C8—H8B	109.1
N1—C2—H2A	108.2	H8A—C8—H8B	107.8
C1—C2—H2B	108.2	C10—C9—C8	110.4 (3)
N1—C2—H2B	108.2	C10—C9—H9A	109.6
H2A—C2—H2B	107.4	C8—C9—H9A	109.6
C4—C3—H3A	109.5	C10—C9—H9B	109.6
C4—C3—H3B	109.5	C8—C9—H9B	109.6
H3A—C3—H3B	109.5	H9A—C9—H9B	108.1
C4—C3—H3C	109.5	C9—C10—N1	116.6 (2)
H3A—C3—H3C	109.5	C9—C10—H10A	108.1
H3B—C3—H3C	109.5	N1—C10—H10A	108.1
C3—C4—N1	115.4 (3)	C9—C10—H10B	108.1
C3—C4—H4A	108.4	N1—C10—H10B	108.1
N1—C4—H4A	108.4	H10A—C10—H10B	107.3
C3—C4—H4B	108.4	Cl1—Cl1—Fe1	0 (10)
N1—C4—H4B	108.4	Cl2—Fe1—Cl3	109.72 (4)
H4A—C4—H4B	107.5	Cl2—Fe1—Cl1	109.41 (5)
C6—C5—H5A	109.5	Cl3—Fe1—Cl1	109.55 (5)
C6—C5—H5B	109.5	Cl2—Fe1—Cl1	109.41 (5)
H5A—C5—H5B	109.5	Cl3—Fe1—Cl1	109.55 (5)
C6—C5—H5C	109.5	Cl1—Fe1—Cl1	0.00 (7)
H5A—C5—H5C	109.5	Cl2—Fe1—Cl4	108.71 (4)
H5B—C5—H5C	109.5	Cl3—Fe1—Cl4	110.18 (5)
C5—C6—N1	115.2 (2)	Cl1—Fe1—Cl4	109.26 (5)
C5—C6—H6A	108.5	Cl1—Fe1—Cl4	109.26 (5)
N1—C6—H6A	108.5	C10—N1—C2	111.0 (2)
C5—C6—H6B	108.5	C10—N1—C6	111.5 (2)
N1—C6—H6B	108.5	C2—N1—C6	106.5 (2)
H6A—C6—H6B	107.5	C10—N1—C4	106.3 (2)
C8—C7—H7A	109.5	C2—N1—C4	110.8 (2)
C8—C7—H7B	109.5	C6—N1—C4	110.8 (2)
H7A—C7—H7B	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*B*···Cl3^{i}	0.96	2.87	3.790 (4)	162
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