Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Dimethyl-1*H*-1,2,3-benzotriazol-3-ium tetrachloridoferrate(III)

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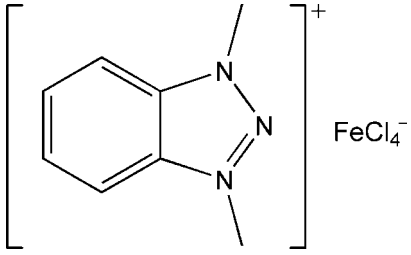
Received 26 January 2013; accepted 6 February 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.057; wR factor = 0.183; data-to-parameter ratio = 20.5.

The asymmetric unit of the title salt, $(C_8H_{10}N_3)[FeCl_4]$, contains one 1,3-dimethyl-1*H*-1,2,3-benzotriazol-3-ium cation and one tetrachloridoferrate anion. The Fe^{III} atom in the anion is tetrahedrally coordinated by four Cl atoms. In the crystal, interactions are observed between the Cl atoms and the triazolium ring [Cl···centroid distances = 3.587 (3) and 3.866 (3) Å].

Related literature

For related iron complexes, see: Hay *et al.* (2003); Liu *et al.* (2000); Lorenz *et al.* (2000); Shapley *et al.* (2003).



Experimental

Crystal data

$(C_8H_{10}N_3)[FeCl_4]$
M_r = 345.84
Orthorhombic, *Pbca*
a = 10.2920 (5) Å
b = 12.5518 (6) Å
c = 22.5857 (9) Å
V = 2917.7 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.74 mm^{-1}
T = 293 K
0.32 × 0.28 × 0.25 mm

Data collection

Oxford Diffraction SuperNova (Dual, Cu at zero, Eos) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2012)
T_{min} = 0.578, T_{max} = 0.647
8114 measured reflections
3016 independent reflections
1828 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.183
S = 1.05
3016 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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*.

We are thankful for support of this study by the National Natural Science Foundation of China (grant No. J0730425) and the Gansu Provincial Natural Science Foundation of China (grant No. 0710RJZA113).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2615).

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supporting information

Acta Cryst. (2013). E**69**, m148 [doi:10.1107/S1600536813003711]

1,3-Dimethyl-1*H*-1,2,3-benzotriazol-3-ium tetrachloridoferrate(III)

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S1. Comment

Iron-containing compounds are ubiquitous throughout the field of coordination chemistry. Recently, a variety of iron compounds have been added to the list of iron coordination complexes (Hay *et al.*, 2003; Liu *et al.*, 2000; Lorenz *et al.*, 2000; Shapley *et al.*, 2003).

In the title compound (Fig. 1), the Fe^{III} atom in the $[FeCl_4]^-$ anion is four-coordinated in a distorted tetrahedral geometry. The Cl—Fe—Cl bond angles are in the range of 108.35 (6)–111.33 (8) °, while the Fe—Cl bond lengths are in the range of 2.1634 (17)–2.1867 (14) Å. Three Cl—Fe—Cl angles are smaller than tetrahedral and the other three are greater than tetrahedral one. In the crystal, interactions between the Cl atoms and the triazolium rings are present [Cl···centroid distances = 3.587 (3) and 3.866 (3) Å].

S2. Experimental

$FeCl_3.6H_2O$ (0.1 mmol, 27.0 mg) was dissolved in 15 ml CH_3OH. To this yellow solution, one equivalent of 1,3-dimethyl-1*H*-benzo[1,2,3]triazolenium chlorate (0.1 mmol, 18.4 mg) was added with stirring. The mixture was filtered and held at room temperature to allow slow evaporation of solvent after stirring 30 min. Block crystals suitable for X-ray diffraction were obtained after one week (yield: 64%).

S3. Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 (CH) and 0.96 Å (CH_3), and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups or $1.2U_{eq}(C)$ otherwise.

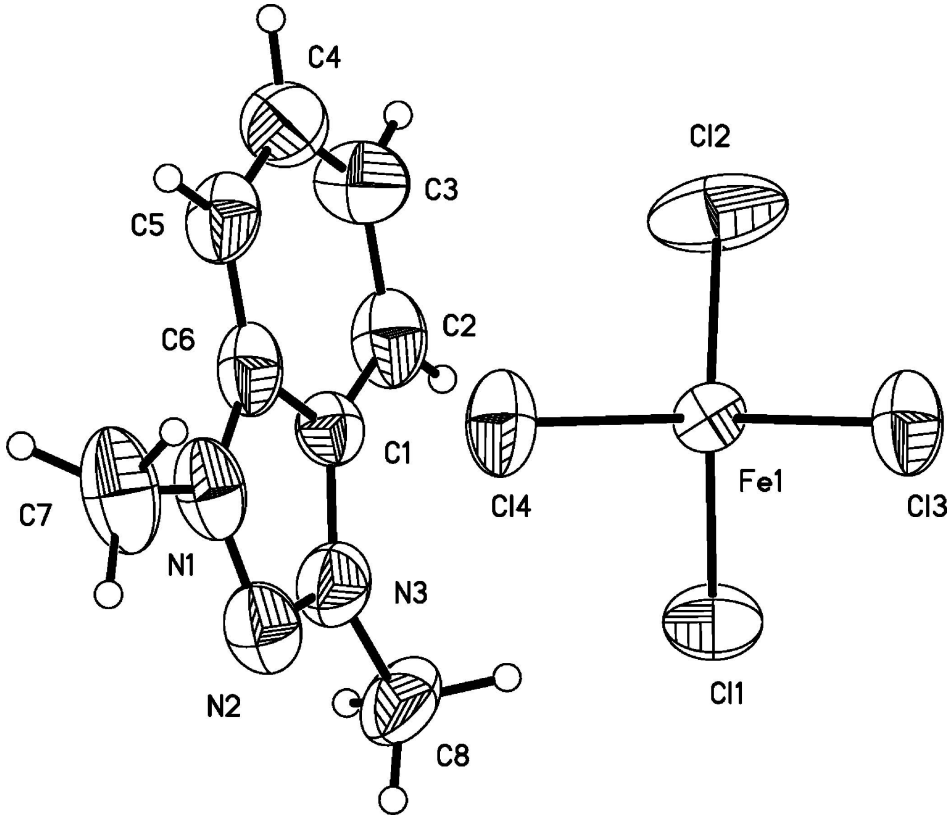

Figure 1

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

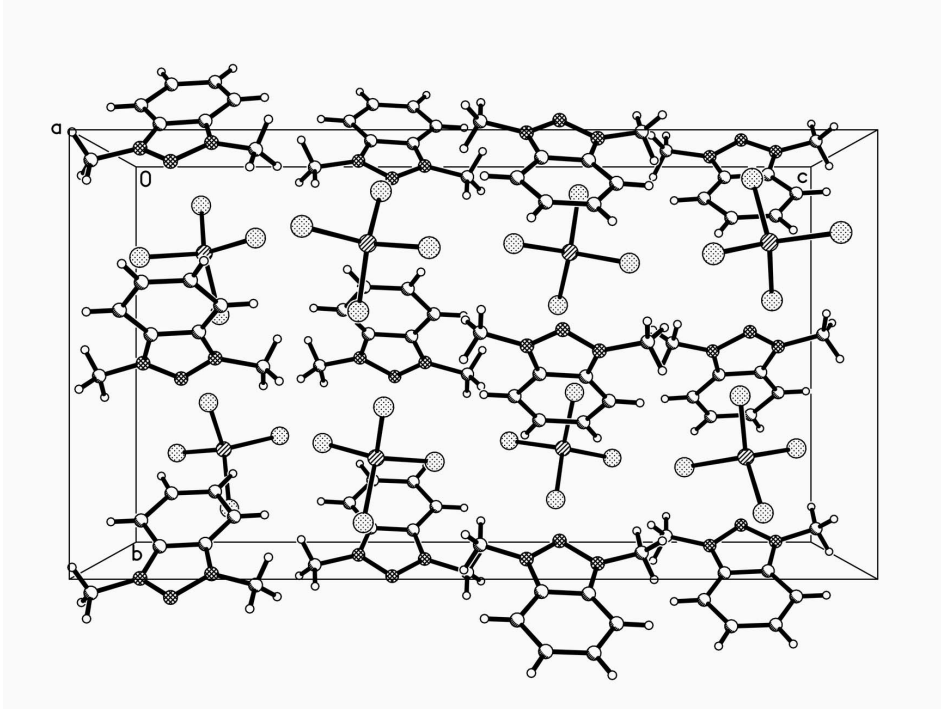

Figure 2

Crystal packing of the title compound viewed along the *a* axis.

1,3-Dimethyl-1*H*-1,2,3-benzotriazol-3-ium tetrachloridoferrate(III)

Crystal data

$(C_8H_{10}N_3)[FeCl_4]$
M_r = 345.84
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 10.2920 (5) Å
b = 12.5518 (6) Å
c = 22.5857 (9) Å
V = 2917.7 (2) Å^3
Z = 8

F(000) = 1384
D_x = 1.575 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 1751 reflections
θ = 3.1–28.5°
μ = 1.74 mm^{-1}
T = 293 K
Block, yellow
0.32 × 0.28 × 0.25 mm

Data collection

Oxford Diffraction SuperNova (Dual, Cu at zero, Eos) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0733 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2012)

T_{min} = 0.578, T_{max} = 0.647
8114 measured reflections
3016 independent reflections
1828 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.5°, θ_{min} = 3.1°
h = −12→6
k = −15→12
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.183$
$S = 1.05$
3016 reflections
147 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
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 1.072P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2130 (5)	0.5085 (4)	0.3377 (2)	0.0934 (14)
N2	0.1550 (5)	0.5572 (4)	0.3863 (2)	0.0907 (14)
N3	0.2259 (5)	0.5203 (4)	0.4309 (2)	0.0912 (13)
C1	0.3237 (4)	0.4541 (4)	0.4130 (2)	0.0650 (11)
C2	0.4177 (6)	0.3997 (5)	0.4452 (2)	0.0927 (17)
H2	0.4238	0.4034	0.4863	0.111*
C3	0.5016 (6)	0.3393 (5)	0.4106 (3)	0.0986 (17)
H3	0.5669	0.3006	0.4293	0.118*
C4	0.4922 (6)	0.3345 (6)	0.3510 (3)	0.1023 (18)
H4	0.5522	0.2937	0.3302	0.123*
C5	0.3950 (6)	0.3889 (5)	0.3193 (2)	0.0877 (16)
H5	0.3874	0.3845	0.2783	0.105*
C6	0.3140 (6)	0.4478 (5)	0.3524 (2)	0.0823 (15)
C7	0.1582 (6)	0.5291 (6)	0.2780 (3)	0.124 (3)
H7A	0.1317	0.4630	0.2604	0.186*
H7B	0.2230	0.5622	0.2535	0.186*
H7C	0.0844	0.5755	0.2814	0.186*
C8	0.1916 (6)	0.5507 (5)	0.4913 (2)	0.111 (2)
H8A	0.1229	0.6023	0.4903	0.166*
H8B	0.2662	0.5808	0.5106	0.166*
H8C	0.1632	0.4888	0.5127	0.166*
Fe1	0.53140 (7)	0.75129 (5)	0.36820 (3)	0.0621 (3)
Cl1	0.40700 (15)	0.76662 (12)	0.44622 (6)	0.0934 (5)
Cl2	0.6707 (2)	0.62448 (18)	0.38165 (8)	0.1478 (9)
Cl3	0.63300 (15)	0.90130 (13)	0.35368 (7)	0.0997 (5)

Cl4	0.40818 (16)	0.71994 (13)	0.29142 (6)	0.1007 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.089 (3)	0.084 (3)	0.108 (4)	−0.025 (3)	−0.029 (3)	0.027 (3)
N2	0.086 (3)	0.079 (3)	0.107 (4)	−0.005 (3)	−0.020 (3)	0.016 (3)
N3	0.094 (3)	0.083 (3)	0.097 (3)	−0.019 (3)	0.010 (3)	−0.012 (3)
C1	0.064 (3)	0.070 (3)	0.060 (3)	−0.009 (2)	0.001 (2)	0.004 (2)
C2	0.105 (4)	0.108 (4)	0.064 (3)	−0.027 (4)	−0.012 (3)	0.008 (3)
C3	0.100 (4)	0.116 (5)	0.080 (4)	0.019 (4)	−0.001 (3)	0.007 (4)
C4	0.097 (4)	0.127 (5)	0.083 (4)	0.001 (4)	0.002 (3)	−0.012 (4)
C5	0.105 (4)	0.108 (4)	0.050 (3)	−0.030 (3)	0.009 (3)	−0.009 (3)
C6	0.089 (4)	0.087 (4)	0.071 (3)	−0.036 (3)	−0.003 (3)	0.003 (3)
C7	0.127 (5)	0.136 (6)	0.110 (5)	−0.039 (4)	−0.062 (4)	0.046 (4)
C8	0.130 (5)	0.109 (5)	0.093 (4)	−0.037 (4)	0.042 (4)	−0.043 (4)
Fe1	0.0725 (5)	0.0589 (5)	0.0548 (4)	0.0043 (3)	0.0006 (3)	0.0041 (3)
Cl1	0.1005 (10)	0.1047 (11)	0.0749 (9)	0.0156 (8)	0.0246 (8)	0.0135 (7)
Cl2	0.1852 (19)	0.1443 (18)	0.1138 (14)	0.1036 (16)	0.0155 (12)	0.0183 (11)
Cl3	0.1123 (11)	0.1022 (11)	0.0847 (10)	−0.0438 (9)	−0.0079 (8)	0.0060 (8)
Cl4	0.1173 (11)	0.1100 (11)	0.0748 (9)	−0.0393 (9)	−0.0193 (8)	0.0016 (8)

Geometric parameters (Å, º)

N1—C6	1.332 (7)	C4—H4	0.9300
N1—N2	1.390 (7)	C5—C6	1.342 (7)
N1—C7	1.485 (7)	C5—H5	0.9300
N2—N3	1.327 (6)	C7—H7A	0.9600
N3—C1	1.367 (6)	C7—H7B	0.9600
N3—C8	1.460 (6)	C7—H7C	0.9600
C1—C6	1.373 (7)	C8—H8A	0.9600
C1—C2	1.390 (7)	C8—H8B	0.9600
C2—C3	1.390 (8)	C8—H8C	0.9600
C2—H2	0.9300	Fe1—Cl2	2.1636 (17)
C3—C4	1.351 (8)	Fe1—Cl3	2.1786 (15)
C3—H3	0.9300	Fe1—Cl4	2.1840 (14)
C4—C5	1.408 (8)	Fe1—Cl1	2.1867 (15)
C6—N1—N2	112.9 (5)	N1—C6—C5	131.3 (6)
C6—N1—C7	128.6 (6)	N1—C6—C1	105.8 (5)
N2—N1—C7	118.5 (5)	C5—C6—C1	122.9 (6)
N3—N2—N1	102.2 (4)	N1—C7—H7A	109.5
N2—N3—C1	113.1 (5)	N1—C7—H7B	109.5
N2—N3—C8	119.1 (5)	H7A—C7—H7B	109.5
C1—N3—C8	127.9 (5)	N1—C7—H7C	109.5
N3—C1—C6	106.1 (5)	H7A—C7—H7C	109.5
N3—C1—C2	131.0 (5)	H7B—C7—H7C	109.5
C6—C1—C2	122.9 (5)	N3—C8—H8A	109.5

C3—C2—C1	113.9 (5)	N3—C8—H8B	109.5
C3—C2—H2	123.0	H8A—C8—H8B	109.5
C1—C2—H2	123.0	N3—C8—H8C	109.5
C4—C3—C2	122.7 (5)	H8A—C8—H8C	109.5
C4—C3—H3	118.6	H8B—C8—H8C	109.5
C2—C3—H3	118.6	Cl2—Fe1—Cl3	109.81 (10)
C3—C4—C5	122.4 (6)	Cl2—Fe1—Cl4	111.32 (8)
C3—C4—H4	118.8	Cl3—Fe1—Cl4	108.36 (6)
C5—C4—H4	118.8	Cl2—Fe1—Cl1	109.85 (7)
C6—C5—C4	115.1 (5)	Cl3—Fe1—Cl1	109.04 (7)
C6—C5—H5	122.4	Cl4—Fe1—Cl1	108.41 (7)
C4—C5—H5	122.4		

C6—N1—N2—N3	−1.1 (5)	C3—C4—C5—C6	1.3 (8)
C7—N1—N2—N3	179.0 (4)	N2—N1—C6—C5	−179.6 (5)
N1—N2—N3—C1	0.9 (5)	C7—N1—C6—C5	0.4 (9)
N1—N2—N3—C8	−177.7 (4)	N2—N1—C6—C1	0.9 (5)
N2—N3—C1—C6	−0.4 (5)	C7—N1—C6—C1	−179.2 (5)
C8—N3—C1—C6	178.0 (5)	C4—C5—C6—N1	180.0 (5)
N2—N3—C1—C2	−179.8 (5)	C4—C5—C6—C1	−0.6 (7)
C8—N3—C1—C2	−1.4 (8)	N3—C1—C6—N1	−0.3 (5)
N3—C1—C2—C3	180.0 (5)	C2—C1—C6—N1	179.2 (5)
C6—C1—C2—C3	0.7 (7)	N3—C1—C6—C5	−179.9 (5)
C1—C2—C3—C4	0.1 (8)	C2—C1—C6—C5	−0.4 (7)
C2—C3—C4—C5	−1.1 (9)		