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Bis(dimethylammonium) tetrachloridoferrate(II)

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Key indicators: single-crystal X-ray study; T = 173 K; mean σ(e–Cl) = 0.001 Å; R factor = 0.034; wR factor = 0.071; data-to-parameter ratio = 20.6.

The title compound, $(C_2H_8N)_2[FeCl_4]$, is composed of discrete dimethylammonium cations and tetrachloridoferrate(II) anions, which are held together in the crystal structure by N—H···Cl hydrogen bonds. It is isostructural with dimethylammonium tetrachloridocobaltate(II) and dimethylammonium tetrachloridomercurate(II).

Related literature

For the tetrachloridocobaltate, see Williams *et al.* (1992); for the tetrachloridomercurate, see Ben Salah *et al.* (1982).

For related literature, see: Morawitz *et al.* (2007).

$$2[CH_3\text{-}NH_2\text{-}CH_3]^+ [FeCl_4]^{2-}$$

Experimental

Crystal data

$(C_2H_8N)_2[FeCl_4]$
M_r = 289.84
Monoclinic, $P2_1/n$
a = 7.8999 (5) Å
b = 11.2878 (9) Å
c = 14.5064 (9) Å
β = 95.828 (5)°
V = 1286.89 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.96 mm^{-1}
T = 173 (2) K
0.15 × 0.13 × 0.08 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.758, T_{max} = 0.859
18093 measured reflections
2477 independent reflections
2026 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.071
S = 1.02
2477 reflections
120 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl2	0.904 (10)	2.431 (19)	3.240 (2)	149 (3)
N1—H1B···Cl4^{i}	0.907 (10)	2.40 (2)	3.201 (2)	148 (3)
N2—H2A···Cl2^{ii}	0.907 (10)	2.62 (3)	3.291 (2)	131 (3)
N2—H2A···Cl1^{ii}	0.907 (10)	2.76 (3)	3.362 (2)	125 (3)
N2—H2B···Cl3	0.914 (10)	2.323 (13)	3.227 (2)	170 (4)

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2267).

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supplementary materials

Acta Cryst. (2007). E**63**, m1683 [**doi:10.1107/S1600536807022994**]

Bis(dimethylammonium) tetrachloridoferrate(II)

T. Morawitz, H.-W. Lerner and M. Bolte

Comment

We report here the X-ray crystal structure analysis of $[NMe_2H_2]_2[FeCl_4]$. Very recently we have described the solid-state structure of the manganese complex $[NMe_2H_2]_2[MnBr_4]$ (Morawitz *et al.*, 2007). The synthesis protocol of $[NMe_2H_2]_2[FeCl_4]$ was similar to that of the manganese derivative $[NMe_2H_2]_2[MnBr_4]$. X-ray quality crystals of $[NMe_2H_2]_2[FeCl_4]$ were grown by diffusion of hexane into a solution of $[NMe_2H_2]_2[FeCl_4]$ in tetrahydrofuran at ambient temperature.

The title compound is composed of discrete dimethylammonium cations and tetrachloroiron(II) anions, which are held together in the crystal by N—H···Cl hydrogen bonds. It is isostructural with dimethylammonium tetrachlorocobaltate(II) (Williams *et al.*, 1992) and dimethylammonium tetrachloromercurate(II) (Ben Salah *et al.*, 1982).

Experimental

By the reaction (see Fig. 2) of the 1,4-phenylene-bridged Li scorpionate (I) (0.24 g, 0.47 mmol) with $FeCl_2$ (0.12 g, 0.94 mmol) and $[NMe_2H_2][Br]$ (*ca* 0.1 mmol) in 30 ml THF, $[NMe_2H_2]_2[FeCl_4]$ was obtained. X-ray quality crystals of $[NMe_2H_2]_2[FeCl_4]$ were grown by diffusion of hexane into a solution of $[NMe_2H_2]_2[FeCl_4]$ in tetrahydrofuran at ambient temperature.

Refinement

H atoms bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.5U_{eq}(C)] using a riding model with C—H = 0.98 Å. H atoms bonded to N were freely refined with the N—H distances restrained to 0.91 (1) Å.

Figures

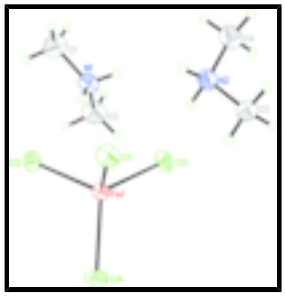


Fig. 1. Perspective view of the title compound with the atom numbering scheme; displacement ellipsoids are at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

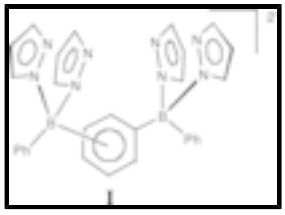


Fig. 2. The anion of the starting compound (I).

Bis(dimethylammonium) tetrachloridoferrate(II)

Crystal data

$(C_2H_8N)_2[FeCl_4]$	F_{000} = 592
M_r = 289.84	D_x = 1.496 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 10454 reflections
a = 7.8999 (5) Å	θ = 3.4–26.0º
b = 11.2878 (9) Å	μ = 1.96 mm^{-1}
c = 14.5064 (9) Å	T = 173 (2) K
β = 95.828 (5)º	Block, colourless
V = 1286.89 (15) Å^3	0.15 × 0.13 × 0.08 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	2477 independent reflections
Radiation source: fine-focus sealed tube	2026 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 173(2) K	θ_{max} = 25.9º
ω scans	θ_{min} = 3.4º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −9→9
T_{min} = 0.758, T_{max} = 0.859	k = −13→13
18093 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.071	$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
2477 reflections	$\Delta\rho_{max}$ = 0.33 e Å^{-3}

120 parameters	$\Delta\rho_{min} = -0.71$ e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Fe1	0.70968 (4)	0.29611 (3)	0.45336 (2)	0.02135 (11)
Cl1	0.71106 (8)	0.50045 (5)	0.44186 (5)	0.03030 (16)
Cl2	0.67392 (8)	0.23483 (6)	0.60487 (4)	0.02816 (15)
Cl3	0.46470 (8)	0.22615 (5)	0.36767 (5)	0.03004 (16)
Cl4	0.95767 (8)	0.21556 (6)	0.41103 (5)	0.03347 (17)
N1	0.2669 (3)	0.1914 (2)	0.57052 (17)	0.0304 (5)
H1A	0.368 (2)	0.227 (3)	0.565 (2)	0.040 (9)*
H1B	0.207 (4)	0.224 (3)	0.5201 (16)	0.048 (10)*
C1	0.2007 (4)	0.2229 (3)	0.6601 (2)	0.0435 (7)
H1C	0.0846	0.1925	0.6606	0.065*
H1D	0.2000	0.3093	0.6671	0.065*
H1E	0.2739	0.1877	0.7115	0.065*
C2	0.2777 (4)	0.0616 (3)	0.5549 (3)	0.0454 (8)
H2C	0.3563	0.0262	0.6038	0.068*
H2D	0.3192	0.0469	0.4945	0.068*
H2E	0.1646	0.0262	0.5559	0.068*
N2	0.3056 (3)	0.4867 (2)	0.32772 (16)	0.0283 (5)
H2A	0.344 (4)	0.535 (2)	0.3754 (16)	0.044 (9)*
H2B	0.344 (5)	0.4129 (17)	0.345 (3)	0.068 (12)*
C3	0.3852 (3)	0.5206 (3)	0.24341 (19)	0.0323 (6)
H3A	0.3535	0.4631	0.1940	0.048*
H3B	0.5093	0.5216	0.2572	0.048*
H3C	0.3456	0.5996	0.2232	0.048*
C4	0.1171 (3)	0.4845 (3)	0.3142 (2)	0.0373 (7)
H4A	0.0749	0.5612	0.2900	0.056*
H4B	0.0724	0.4690	0.3737	0.056*
H4C	0.0793	0.4219	0.2701	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01938 (18)	0.02096 (19)	0.0236 (2)	0.00109 (13)	0.00171 (13)	0.00027 (14)
Cl1	0.0325 (3)	0.0207 (3)	0.0377 (4)	−0.0013 (2)	0.0031 (3)	0.0037 (3)
Cl2	0.0308 (3)	0.0283 (3)	0.0250 (3)	−0.0009 (2)	0.0008 (2)	0.0049 (2)
Cl3	0.0281 (3)	0.0271 (3)	0.0330 (4)	−0.0023 (2)	−0.0066 (3)	−0.0033 (3)
Cl4	0.0247 (3)	0.0349 (4)	0.0416 (4)	0.0068 (2)	0.0077 (3)	−0.0039 (3)
N1	0.0281 (12)	0.0259 (12)	0.0373 (13)	−0.0034 (9)	0.0039 (10)	0.0055 (10)
C1	0.0531 (19)	0.0374 (17)	0.0420 (18)	−0.0013 (13)	0.0151 (15)	0.0073 (14)
C2	0.0532 (19)	0.0236 (14)	0.059 (2)	−0.0013 (13)	0.0033 (16)	0.0007 (14)
N2	0.0283 (11)	0.0287 (12)	0.0272 (12)	0.0016 (9)	−0.0007 (9)	0.0012 (10)
C3	0.0297 (13)	0.0397 (16)	0.0279 (14)	0.0023 (11)	0.0054 (11)	0.0000 (12)
C4	0.0271 (14)	0.0424 (17)	0.0422 (17)	−0.0071 (12)	0.0025 (12)	0.0051 (14)

Geometric parameters (Å, °)

Fe1—Cl4	2.2992 (7)	C2—H2D	0.9800
Fe1—Cl1	2.3128 (7)	C2—H2E	0.9800
Fe1—Cl3	2.3293 (7)	N2—C3	1.482 (4)
Fe1—Cl2	2.3483 (7)	N2—C4	1.483 (3)
N1—C2	1.486 (4)	N2—H2A	0.907 (10)
N1—C1	1.493 (4)	N2—H2B	0.914 (10)
N1—H1A	0.904 (10)	C3—H3A	0.9800
N1—H1B	0.907 (10)	C3—H3B	0.9800
C1—H1C	0.9800	C3—H3C	0.9800
C1—H1D	0.9800	C4—H4A	0.9800
C1—H1E	0.9800	C4—H4B	0.9800
C2—H2C	0.9800	C4—H4C	0.9800
Cl4—Fe1—Cl1	111.39 (3)	N1—C2—H2E	109.5
Cl4—Fe1—Cl3	114.06 (3)	H2C—C2—H2E	109.5
Cl1—Fe1—Cl3	108.02 (3)	H2D—C2—H2E	109.5
Cl4—Fe1—Cl2	108.68 (3)	C3—N2—C4	113.6 (2)
Cl1—Fe1—Cl2	111.30 (3)	C3—N2—H2A	110 (2)
Cl3—Fe1—Cl2	103.14 (3)	C4—N2—H2A	111 (2)
C2—N1—C1	113.5 (2)	C3—N2—H2B	108 (3)
C2—N1—H1A	111 (2)	C4—N2—H2B	109 (3)
C1—N1—H1A	111 (2)	H2A—N2—H2B	105 (3)
C2—N1—H1B	108 (2)	N2—C3—H3A	109.5
C1—N1—H1B	114 (2)	N2—C3—H3B	109.5
H1A—N1—H1B	98 (3)	H3A—C3—H3B	109.5
N1—C1—H1C	109.5	N2—C3—H3C	109.5
N1—C1—H1D	109.5	H3A—C3—H3C	109.5
H1C—C1—H1D	109.5	H3B—C3—H3C	109.5
N1—C1—H1E	109.5	N2—C4—H4A	109.5
H1C—C1—H1E	109.5	N2—C4—H4B	109.5
H1D—C1—H1E	109.5	H4A—C4—H4B	109.5

N1—C2—H2C	109.5	N2—C4—H4C	109.5
N1—C2—H2D	109.5	H4A—C4—H4C	109.5
H2C—C2—H2D	109.5	H4B—C4—H4C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl2	0.904 (10)	2.431 (19)	3.240 (2)	149 (3)
N1—H1B···Cl4^{i}	0.907 (10)	2.40 (2)	3.201 (2)	148 (3)
N2—H2A···Cl2^{ii}	0.907 (10)	2.62 (3)	3.291 (2)	131 (3)
N2—H2A···Cl1^{ii}	0.907 (10)	2.76 (3)	3.362 (2)	125 (3)
N2—H2B···Cl3	0.914 (10)	2.323 (13)	3.227 (2)	170 (4)

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z+1$.

Fig. 1

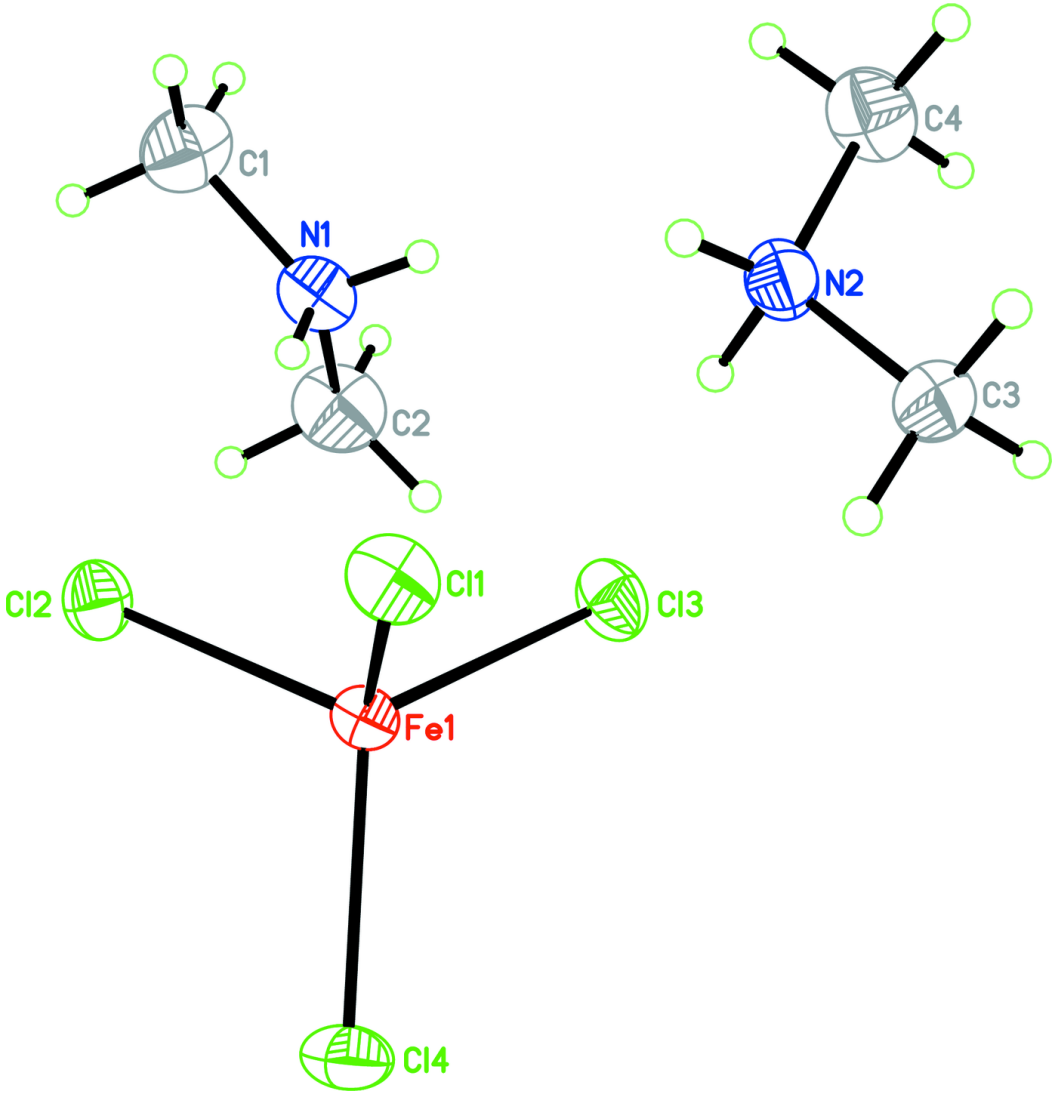
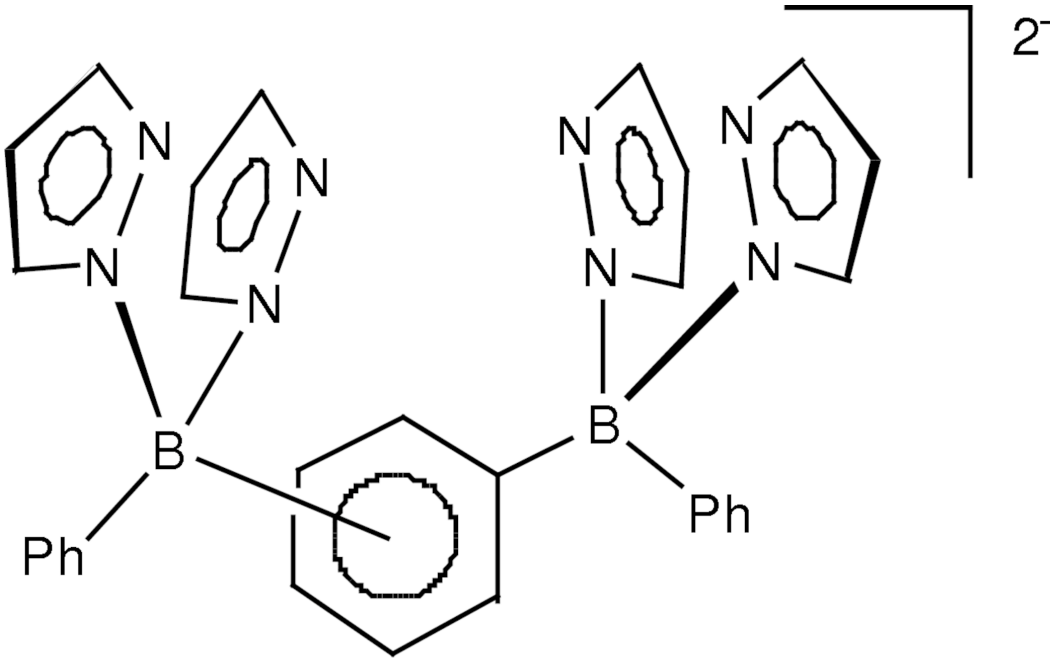


Fig. 2



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