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Dimethyl(3-oxo-3-phenylpropyl)azanium tetrachloridoferrate(III)

Lei Jin

College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: jinlei8812@163.com

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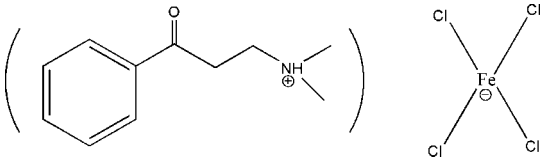
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.010 Å; R factor = 0.071; wR factor = 0.172; data-to-parameter ratio = 19.8.

In the title molecular salt, $(C_{11}H_{16}NO)[FeCl_4]$, an intramolecular N—H···O hydrogen bond in the cation generates an $S(6)$ loop and the conformation of the C(=O)—C—C—N chain is *gauche* [torsion angle = 57.0 (6)°]. The anion is a near-regular tetrahedron [range of Cl—Fe—Cl angles = 107.93 (8)–112.13 (10)°]. There are no directional inter-ionic bonds in the crystal.

Related literature

For related structures, see: Hay & Geib (2005); Ton & Bolte (2004).



Experimental

Crystal data

$(C_{11}H_{16}NO)[FeCl_4]$
M_r = 375.90
Monoclinic, $P2_1/c$
a = 6.3166 (13) Å
b = 15.149 (3) Å
c = 17.293 (4) Å
β = 92.55 (3)°
V = 1653.1 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.55 mm^{-1}
T = 291 K
0.26 × 0.22 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.08, T_{max} = 0.12
15234 measured reflections
3244 independent reflections
1895 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.172
S = 1.11
3244 reflections
164 parameters
61 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—Cl1	2.164 (2)	Fe1—Cl3	2.180 (2)
Fe1—Cl2	2.1854 (18)	Fe1—Cl4	2.179 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1D···O1	0.91	2.06	2.735 (7)	130

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: HB6733).

References

Hay, M. T. & Geib, S. J. (2005). *Acta Cryst.* E**61**, m190–m191.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Ton, C. & Bolte, M. (2004). *Acta Cryst.* E**60**, o616–o617.

supplementary materials

Acta Cryst. (2012). E**68**, m709 [doi:10.1107/S1600536812018600]

Dimethyl(3-oxo-3-phenylpropyl)azanium tetrachloridoferrate(III)

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Experimental

At room temperature, dimethyl-(3-oxo-3-phenyl-propyl)-amine (5 mmol, 0.89 g) was dissolved in 30 ml water, then $FeCl_3.6H_2O$ (5 mmol, 1.35 g) was added slowly with sirring. Orange plates were obtained by the slow evaporation of the above filtrate after a week in air.

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).

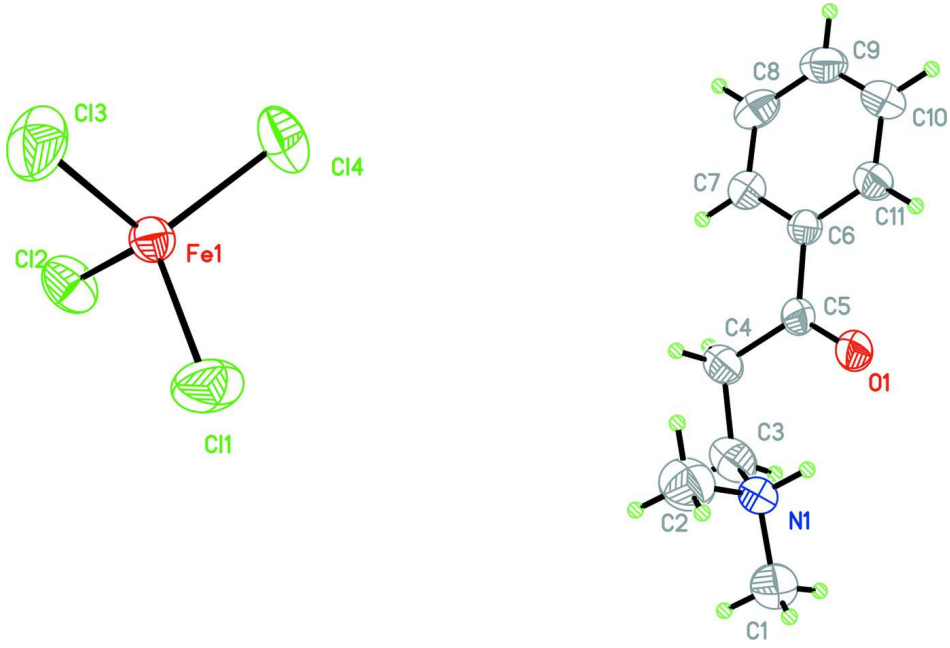

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

Dimethyl(3-oxo-3-phenylpropyl)azanium tetrachloridoferrate(III)

Crystal data

$(C_{11}H_{16}NO)[FeCl_4]$
$M_r = 375.90$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.3166$ (13) Å
$b = 15.149$ (3) Å
$c = 17.293$ (4) Å
$\beta = 92.55$ (3)°
$V = 1653.1$ (6) Å^3
$Z = 4$
$F(000) = 764$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 3.2$–26°
$\mu = 1.55$ mm^{-1}
$T = 291$ K
Block, yellow
0.26 × 0.22 × 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)
$T_{min} = 0.08$, $T_{max} = 0.12$
15234 measured reflections
3244 independent reflections
1895 reflections with $I > 2\sigma(I)$
$R_{int} = 0.080$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
$h = -7 \rightarrow 7$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.172$
$S = 1.11$
3244 reflections
164 parameters
61 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
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 2.3762P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0126 (14)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0240 (17)	0.4076 (6)	0.9427 (6)	0.135 (3)
H1A	−0.0142	0.3960	0.9974	0.203*
H1B	−0.1631	0.4292	0.9285	0.203*

H1C	0.0797	0.4510	0.9301	0.203*
C2	0.0080 (15)	0.3420 (6)	0.8157 (5)	0.120 (3)
H2A	0.0347	0.2878	0.7890	0.180*
H2B	0.1140	0.3847	0.8037	0.180*
H2C	−0.1294	0.3641	0.7995	0.180*
C3	−0.1593 (13)	0.2645 (5)	0.9215 (5)	0.102 (2)
H3A	−0.2943	0.2879	0.9020	0.122*
H3B	−0.1629	0.2603	0.9774	0.122*
C4	−0.1252 (11)	0.1715 (4)	0.8870 (4)	0.0829 (19)
H4A	−0.2390	0.1335	0.9027	0.099*
H4B	−0.1363	0.1760	0.8310	0.099*
C5	0.0813 (10)	0.1284 (4)	0.9097 (3)	0.0627 (16)
C6	0.1018 (9)	0.0308 (4)	0.9021 (3)	0.0547 (14)
C7	−0.0662 (11)	−0.0227 (4)	0.8783 (4)	0.0745 (19)
H7A	−0.1985	0.0019	0.8663	0.089*
C8	−0.0368 (14)	−0.1134 (5)	0.8724 (4)	0.088 (2)
H8A	−0.1491	−0.1495	0.8559	0.106*
C9	0.1574 (15)	−0.1496 (5)	0.8908 (4)	0.088 (2)
H9A	0.1770	−0.2102	0.8863	0.105*
C10	0.3218 (13)	−0.0976 (5)	0.9157 (5)	0.087 (2)
H10A	0.4528	−0.1225	0.9292	0.104*
C11	0.2933 (10)	−0.0084 (4)	0.9206 (4)	0.0701 (18)
H11A	0.4071	0.0269	0.9371	0.084*
Cl1	0.4836 (3)	0.50465 (14)	0.21112 (15)	0.1142 (8)
Cl2	0.0148 (3)	0.38087 (12)	0.17367 (12)	0.0850 (6)
Cl3	0.4451 (4)	0.3546 (2)	0.05173 (13)	0.1258 (9)
Cl4	0.4761 (3)	0.26961 (14)	0.24300 (13)	0.0987 (7)
Fe1	0.36032 (13)	0.37873 (5)	0.17073 (5)	0.0576 (3)
N1	0.0153 (9)	0.3257 (3)	0.9003 (3)	0.0745 (14)
H1D	0.1432	0.3026	0.9159	0.089*
O1	0.2302 (8)	0.1727 (3)	0.9339 (3)	0.0893 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.165 (7)	0.114 (6)	0.123 (6)	0.047 (6)	−0.041 (6)	−0.034 (5)
C2	0.120 (6)	0.129 (6)	0.113 (6)	0.022 (5)	0.023 (5)	−0.006 (5)
C3	0.100 (5)	0.096 (4)	0.109 (5)	0.026 (4)	0.006 (4)	0.010 (4)
C4	0.080 (4)	0.065 (4)	0.102 (4)	0.010 (3)	−0.014 (3)	0.003 (3)
C5	0.074 (4)	0.057 (4)	0.056 (4)	0.002 (3)	−0.011 (3)	0.005 (3)
C6	0.064 (4)	0.055 (4)	0.045 (3)	0.003 (3)	0.003 (3)	0.002 (3)
C7	0.079 (5)	0.071 (5)	0.072 (5)	0.001 (4)	−0.007 (4)	−0.005 (4)
C8	0.103 (6)	0.069 (5)	0.092 (6)	−0.020 (5)	0.001 (4)	−0.018 (4)
C9	0.122 (7)	0.061 (4)	0.081 (5)	0.010 (5)	0.011 (5)	−0.005 (4)
C10	0.089 (5)	0.071 (5)	0.100 (6)	0.022 (4)	0.001 (4)	−0.002 (4)
C11	0.072 (4)	0.062 (4)	0.076 (5)	0.005 (3)	−0.001 (3)	0.004 (3)
Cl1	0.0904 (15)	0.0800 (13)	0.170 (2)	−0.0095 (11)	−0.0163 (14)	−0.0386 (14)
Cl2	0.0490 (9)	0.0881 (13)	0.1176 (16)	0.0042 (9)	0.0013 (9)	0.0156 (11)
Cl3	0.1048 (16)	0.197 (3)	0.0762 (14)	0.0392 (17)	0.0098 (11)	−0.0246 (15)
Cl4	0.0720 (12)	0.0990 (14)	0.1228 (17)	0.0033 (11)	−0.0227 (11)	0.0345 (12)

Fe1	0.0481 (5)	0.0582 (6)	0.0660 (6)	0.0028 (4)	−0.0023 (4)	−0.0042 (4)
N1	0.081 (3)	0.061 (3)	0.081 (3)	0.013 (3)	−0.011 (3)	−0.004 (3)
O1	0.090 (3)	0.061 (3)	0.113 (4)	0.000 (3)	−0.041 (3)	−0.003 (3)

Geometric parameters (Å, º)

C1—N1	1.468 (9)	C6—C11	1.372 (8)
C1—H1A	0.9600	C6—C7	1.383 (8)
C1—H1B	0.9600	C7—C8	1.391 (9)
C1—H1C	0.9600	C7—H7A	0.9300
C2—N1	1.483 (10)	C8—C9	1.368 (11)
C2—H2A	0.9600	C8—H8A	0.9300
C2—H2B	0.9600	C9—C10	1.358 (10)
C2—H2C	0.9600	C9—H9A	0.9300
C3—N1	1.499 (9)	C10—C11	1.367 (9)
C3—C4	1.548 (10)	C10—H10A	0.9300
C3—H3A	0.9700	C11—H11A	0.9300
C3—H3B	0.9700	Fe1—Cl1	2.164 (2)
C4—C5	1.495 (8)	Fe1—Cl2	2.1854 (18)
C4—H4A	0.9700	Fe1—Cl3	2.180 (2)
C4—H4B	0.9700	Fe1—Cl4	2.179 (2)
C5—O1	1.214 (7)	N1—H1D	0.9100
C5—C6	1.492 (8)		
N1—C1—H1A	109.5	C7—C6—C5	122.6 (6)
N1—C1—H1B	109.5	C6—C7—C8	119.8 (7)
H1A—C1—H1B	109.5	C6—C7—H7A	120.1
N1—C1—H1C	109.5	C8—C7—H7A	120.1
H1A—C1—H1C	109.5	C9—C8—C7	120.0 (7)
H1B—C1—H1C	109.5	C9—C8—H8A	120.0
N1—C2—H2A	109.5	C7—C8—H8A	120.0
N1—C2—H2B	109.5	C10—C9—C8	120.4 (7)
H2A—C2—H2B	109.5	C10—C9—H9A	119.8
N1—C2—H2C	109.5	C8—C9—H9A	119.8
H2A—C2—H2C	109.5	C9—C10—C11	119.5 (7)
H2B—C2—H2C	109.5	C9—C10—H10A	120.2
N1—C3—C4	110.6 (6)	C11—C10—H10A	120.2
N1—C3—H3A	109.5	C6—C11—C10	122.0 (7)
C4—C3—H3A	109.5	C6—C11—H11A	119.0
N1—C3—H3B	109.5	C10—C11—H11A	119.0
C4—C3—H3B	109.5	Cl1—Fe1—Cl4	112.13 (10)
H3A—C3—H3B	108.1	Cl1—Fe1—Cl3	110.63 (11)
C5—C4—C3	115.5 (6)	Cl4—Fe1—Cl3	108.91 (10)
C5—C4—H4A	108.4	Cl1—Fe1—Cl2	108.94 (9)
C3—C4—H4A	108.4	Cl4—Fe1—Cl2	107.93 (8)
C5—C4—H4B	108.4	Cl3—Fe1—Cl2	108.17 (9)
C3—C4—H4B	108.4	C2—N1—C1	110.7 (7)
H4A—C4—H4B	107.5	C2—N1—C3	110.7 (6)
O1—C5—C4	120.2 (6)	C1—N1—C3	105.0 (7)
O1—C5—C6	120.6 (6)	C2—N1—H1D	110.1

C4—C5—C6	119.2 (6)	C1—N1—H1D	110.1
C11—C6—C7	118.3 (6)	C3—N1—H1D	110.1
C11—C6—C5	119.1 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···O1	0.91	2.06	2.735 (7)	130