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Bis[hydrotris(1-pyrazolyl)borato-κ^3N,N',N'']iron(III) tetrachloroferrate(III) acetonitrile solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.127
Data-to-parameter ratio = 13.4

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

In the title compound, $[Fe(C_9H_{10}BN_6)_2][FeCl_4]\cdot C_2H_3N$, extensive weak C—H···Cl/N interactions exist between the octahedral $[Fe(HBpz_3)_2]^+$ cations [$HBpz_3^-$ is hydrotris(pyrazolyl)borate], tetrahedral $FeCl_4^-$ anions and acetonitrile solvent molecules.

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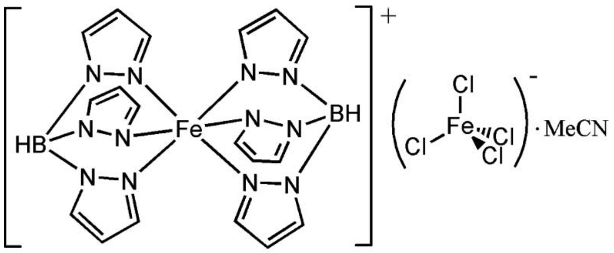
Comment

Tridentate hydrotris(pyrazoly1)borates ($HBpz_3^-$) have been widely used in coordination chemistry, and their complexes with most metals or metalloids of the periodic table have been prepared (Trofimenko, 1993). The title compound, (I), was obtained in an attempt to prepare a polynuclear iron(III) complex.



(I)

The crystal structure of (I) consists of $[Fe(HBpz_3)_2]^+$ cations, $FeCl_4^-$ anions and acetonitrile solvent molecules (Fig. 1). Atom Fe1 in the $[Fe(HBpz_3)_2]^+$ cation is coordinated in an octahedral geometry by six pyrazolyl N atoms from two $HBpz_3^-$ ligands. The Fe—N bond lengths of 1.943 (3)–1.950 (3) Å are similar to those found in tris{bis[hydrotris(1-pyrazolyl)borato-$\kappa^3N^2,N^{2'},N^{2''}$]iron(III)} hexaisothio-

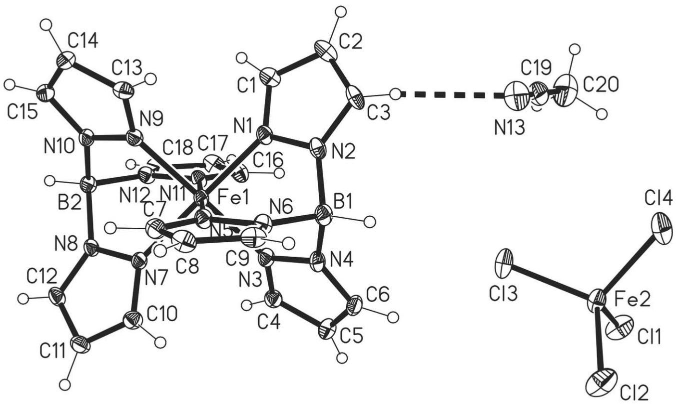


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. The broken line indicates the C—H···N interaction.

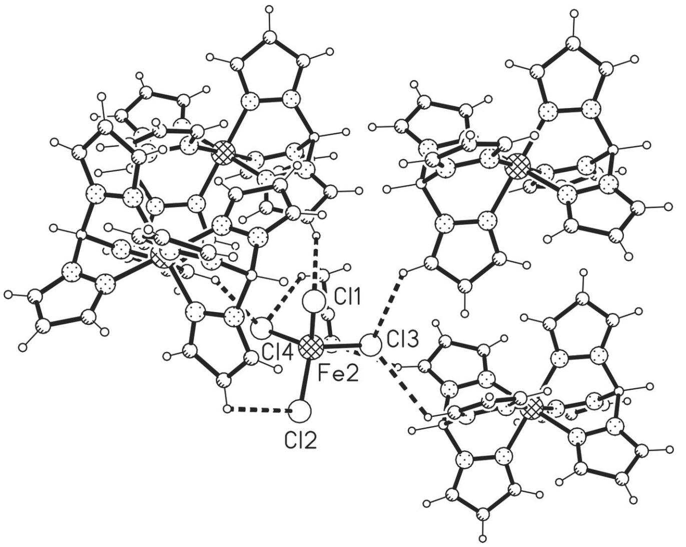


Figure 2
Plot of the hydrogen-bonding interactions among $Fe(HBpz)_2^+$, $FeCl_4^-$ and MeCN. Broken lines indicate C—H···Cl interactions.

cyanatoiron(III) [Fe—N = 1.941 (2)–1.953 (2) Å; Wang *et al.*, 2004], bis[hydridotris(1*H*-pyrazol-1-yl)borato]iron(III) tetrafluoroborate [Fe—N = 1.948 (6)–1.964 (6) Å; Calogero *et al.*, 1994], and bis[hydrogentris(pyrazolyl)borate-*N*,*N*′,*N*″]-iron(III) trichloro[hydrogentris(pyrazolyl)borate-*N*,*N*′,*N*″]-iron(III) [Fe—N = 1.945 (3)–1.961 (3) Å; Cho *et al.*, 1992]. The geometry about atom Fe2 in the $FeCl_4^-$ anion is tetrahedral. The Fe—Cl bond lengths of 2.1741 (14)–2.1994 (14) Å are similar to those observed in [1,4,7-tris(pyrazol-3-ylmethyl)-1,4,7-triazacyclononane]iron(II) tetrachloroferrate(III) chloride [2.179 (3)–2.206 (3) Å; Di Vaira *et al.*, 1997].

The structure is consolidated by extensive weak C—H···Cl/N interactions (Table 2 and Fig. 2). These interactions connect the $[Fe(HBpz_3)_2]^+$ cations, $FeCl_4^-$ anions and MeCN solvent molecules into a three-dimensional molecular architecture. The H···Cl distances (2.87–3.15 Å) and C—H···Cl angles (112–160°) in (I) are comparable with those (H···Cl = 2.47–3.30 Å; C—H···Cl = 90–180°) of reported organic crystal structures with C—H···Cl—*M* contacts in a Cambridge Structural Database (Version 5.19; Allen, 2002) analysis (Thallapally & Nangia, 2001).

Experimental

Potassium hydrotris(1-pyrazolyl)borate (0.025 g, 0.1 mmol) dissolved in acetonitrile (5 ml) was mixed with an acetonitrile solution (2 ml) of iron(III) chloride hexahydrate (0.027 g, 0.1 mmol). Red block-shaped crystals of (I) separated from the solution after 2 d (yield 65%).

Crystal data

$[Fe(C_9H_{10}BN_6)_2][FeCl_4]\cdot C_2H_3N$
M_r = 720.63
Monoclinic, $P2_1/c$
a = 9.779 (4) Å
b = 20.122 (8) Å
c = 15.667 (6) Å
β = 100.011 (5)°
V = 3036 (2) Å^3
Z = 4
D_x = 1.577 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3568 reflections
θ = 3.1–25.0°
μ = 1.34 mm^{-1}
T = 293 (2) K
Block, red
0.32 × 0.26 × 0.18 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
T_{min} = 0.673, T_{max} = 0.794
9914 measured reflections
5080 independent reflections
4174 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°
$h = -11 \rightarrow 5$
$k = -23 \rightarrow 21$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.127
S = 1.06
5080 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.73 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N7	1.943 (3)	Fe1—N1	1.950 (3)
Fe1—N11	1.945 (3)	Fe2—Cl2	2.1741 (14)
Fe1—N3	1.946 (3)	Fe2—Cl3	2.1915 (12)
Fe1—N9	1.947 (3)	Fe2—Cl1	2.1933 (14)
Fe1—N5	1.947 (3)	Fe2—Cl4	2.1994 (14)
N7—Fe1—N11	88.70 (11)	N11—Fe1—N1	91.67 (11)
N7—Fe1—N3	90.69 (11)	N3—Fe1—N1	88.74 (11)
N11—Fe1—N3	91.52 (11)	N9—Fe1—N1	91.57 (11)
N7—Fe1—N9	89.00 (11)	N5—Fe1—N1	88.36 (11)
N11—Fe1—N9	88.13 (11)	Cl2—Fe2—Cl3	110.02 (5)
N3—Fe1—N9	179.54 (11)	Cl2—Fe2—Cl1	109.67 (6)
N7—Fe1—N5	91.27 (11)	Cl3—Fe2—Cl1	108.29 (5)
N11—Fe1—N5	179.79 (12)	Cl2—Fe2—Cl4	110.63 (6)
N3—Fe1—N5	88.70 (11)	Cl3—Fe2—Cl4	109.01 (6)
N9—Fe1—N5	91.66 (11)	Cl1—Fe2—Cl4	109.17 (5)
N7—Fe1—N1	179.33 (10)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N13	0.93	2.54	3.398 (6)	154
C2—H2···Cl1^{i}	0.93	2.87	3.752 (4)	160
C17—H17···Cl2^{ii}	0.93	3.15	3.599 (4)	112
C6—H6···Cl3	0.93	2.99	3.546 (4)	120
C9—H9···Cl3^{iii}	0.93	3.10	3.684 (4)	122
C4—H4···Cl4^{ii}	0.93	3.08	3.642 (4)	121
C20—H20*C*···Cl4	0.96	2.95	3.764 (6)	143

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+1, y, z$.

All H atoms were included in geometrically calculated positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = $1.2U_{eq}$(C) for the pyridyl groups, and C—H = 0.96 Å and U_{iso}(H) = $1.5U_{eq}$(C) for the methyl group.

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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