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(Tris{2-[(1-methylimidazol-2-yl)methylimino]ethyl}amine)iron(II) diperchlorate acetonitrile solvate

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Key indicators

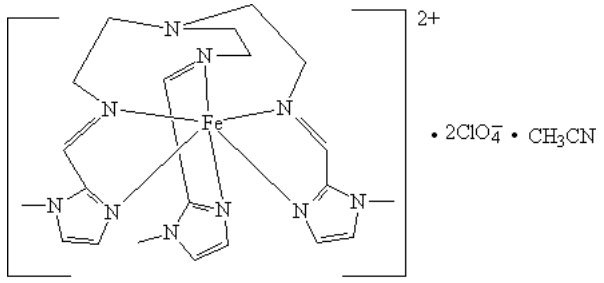
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.163
Data-to-parameter ratio = 16.5

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

In the cation of the title complex, $[Fe(C_{21}H_{30}N_{10})](ClO_4)_2\cdot CH_3CN$, the Fe^{II} atom is coordinated by three imine N atoms and three imidazole N atoms in a distorted octahedral geometry, with Fe—N bond distances ranging from 2.189 (3) to 2.236 (3) Å.

Comment

As a part of our ongoing studies of transition metal complexes incorporating imidazole and related ligands (Chen *et al.*, 2003, 2005; Yang *et al.*, 1999, 2000, 2001, 2004), the title compound, (I), has been prepared and its X-ray crystal structure is presented here.



(I)

The crystal structure of (I) consists of $[FeL]^{2+}$ complex cations (where L is tri{2-[(1-methylimidazol-2-yl)methylimino]ethyl}amine), perchlorate anions and acetonitrile solvent molecules. The Fe^{II} atom is coordinated by three imine N atoms and three imidazole N atoms in a distorted octahedral geometry (Fig. 1 and Table 1). Although L is a potentially heptadentate ligand, the Fe···N10 distance of 2.834 (3) Å suggests no bonding interaction between the N10 and Fe^{II} atoms.

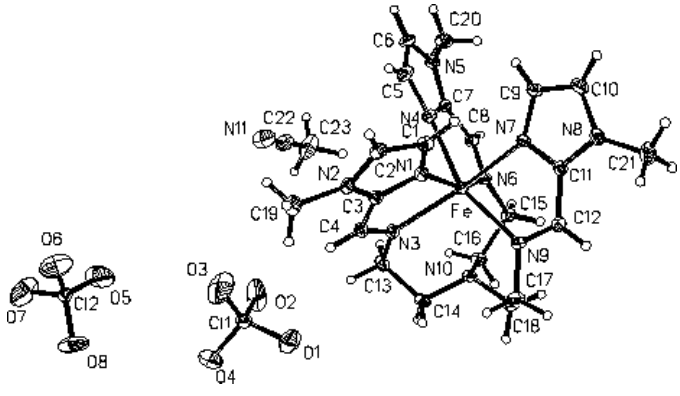


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 35% probability level and H atoms shown as small spheres of arbitrary radii.

The two equilateral triangles, one formed by the three imine N atoms (N1, N4 and N7) and the other formed by imidazole N atoms (N3, N6 and N9), are nearly parallel to each other. This is similar to the situation found in structures reported previously (Yang *et al.*, 2001; Kichner *et al.*, 1987; Sim & Sinn, 1978).

The packing of (I) is shown in Fig. 2. Weak C—H···O hydrogen bonding occurs between the cations and anions (Table 2).

Experimental

1-Methylimidazole-2-carbaldehyde (3 mmol, 0.330 g) was added to a solution of tri(2-aminoethyl)amine (1 mmol, 0.146 g) in dried methanol. The mixture was refluxed for 5 h, and then an acetonitrile solution of $FeCl_3·6H_2O$ (1 mmol, 0.263 g) and an aqueous methanol solution (10 ml, 1:1, *v*/*v*) of $NaClO_4$ (2 mmol, 0.250 g) were added. After filtration, the solution was allowed to stand at room temperature in air. Red single crystals of (I) were obtained from the filtrate after 6 d.

Crystal data

$[Fe(C_{21}H_{30}N_{10})](ClO_4)_2·C_2H_3N$
M_r = 718.35
Orthorhombic, *Pbca*
a = 16.5457 (9) Å
b = 16.7931 (9) Å
c = 22.4135 (12) Å
V = 6227.7 (6) Å^3
Z = 8
D_x = 1.532 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6256 reflections
θ = 2.4–22.5°
μ = 0.72 mm^{-1}
T = 295 (2) K
Block, red
0.52 × 0.42 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.706, T_{max} = 0.758
35111 measured reflections
6787 independent reflections
4589 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 27.0°
$h = -20 \rightarrow 21$
$k = -19 \rightarrow 21$
$l = -18 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.163
S = 0.99
6787 reflections
411 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0916P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.042
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.00023 (2)

Table 1
Selected geometric parameters (Å, °).

Fe—N1	2.206 (2)	Fe—N6	2.198 (3)
Fe—N3	2.189 (3)	Fe—N7	2.205 (3)
Fe—N4	2.236 (3)	Fe—N9	2.212 (3)
N3—Fe—N6	105.7 (1)	N7—Fe—N9	74.07 (9)
N3—Fe—N7	157.9 (1)	N1—Fe—N9	95.3 (1)
N6—Fe—N7	96.1 (1)	N3—Fe—N4	97.3 (1)
N3—Fe—N1	74.4 (1)	N6—Fe—N4	74.5 (1)
N6—Fe—N1	160.4 (1)	N7—Fe—N4	84.9 (1)
N7—Fe—N1	83.9 (1)	N1—Fe—N4	86.0 (1)
N3—Fe—N9	103.6 (1)	N9—Fe—N4	158.7 (1)
N6—Fe—N9	103.6 (1)		

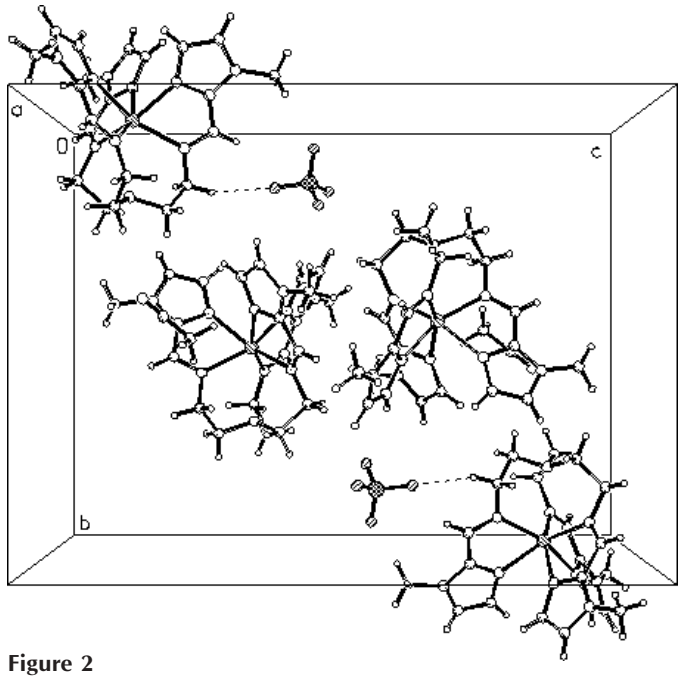


Figure 2
A perspective view of the packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O8^{i}	0.93	2.58	3.367 (5)	142
C15—H15A···O7^{ii}	0.97	2.49	3.461 (7)	176
C15—H15B···O2^{iii}	0.97	2.56	3.462 (5)	156
C21—H21B···O8^{i}	0.96	2.59	3.483 (5)	154
C21—H21C···O6^{ii}	0.96	2.50	3.337 (5)	146

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

H atoms were positioned geometrically (C—H = 0.93, 0.96 or 0.97 Å) and refined using the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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