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Tris(1,10-phenanthroline-κ^2N,N')iron(II) dichloride methanol hexasolvate

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The title complex, $[Fe(C_{12}H_8N_2)_3]Cl_2\cdot 6CH_3OH$, contains a slightly distorted octahedral $[Fe(phen)_3]^{2+}$ cation (phen is 1,10-phenanthroline). Chloride anions and solvent methanol molecules form two distinct hydrogen-bonded anionic clusters, formulated as $[Cl(MeOH)_2]^-$ and $[Cl(MeOH)_4]^-$, which complete the crystal structure.

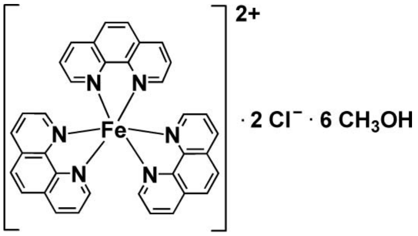
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.164
Data-to-parameter ratio = 13.6

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

Comment

Metal complexes with 1,10-phenanthroline (phen) as a ligand are well known, but structures of these where the $[Fe(phen)_3]^{2+}$ cation is accompanied by simple (X^-) halide counter-ions are surprisingly scarce. A search of the Cambridge Structural Database (CSD, Version 5.28, updated January 2007; Allen, 2002) revealed only two iodides. The first is a dihydrate nitrobenzene solvate (refcode CEMPEP; Fujiwara *et al.*, 1984) and the second is a dihydrate (refcode TPHOLF; Johansson *et al.*, 1978), where the water molecules and iodide ions form chains through hydrogen bonds. In both cases, the anions and solvent molecules are located between the layers formed by the metal complex cations.



(I)

The title complex, (I), prepared from $FeCl_2$ and phenanthroline in methanol as a solvent (see *Experimental*), incorporates six molecules of methanol in the asymmetric unit (Fig. 1). The coordination of the Fe^{II} ion in the discrete complex cation $[Fe(phen)_3]^{2+}$ is almost perfect octahedral, with *cis* N—Fe—N angles approaching 90° and all Fe—N bond lengths spanning a small range (Table 1), which compares well with those observed in the above-quoted solvated iodides (1.96–1.99 Å).

All methanol molecules interact through hydrogen bonds, either with Cl^- anions or with another methanol molecule within the asymmetric unit (Table 2). As expected, the O—H···Cl contacts are longer than O—H···O, by *ca* 0.2–0.4 Å. These interactions lead to the formation of two well separated anionic clusters, $[Cl(MeOH)_2]^-$, with an O—H···O—H···Cl interaction, and $[Cl(MeOH)_4]^-$, where one Cl^- ion interacts simultaneously with three methanol molecules (Fig. 2). The

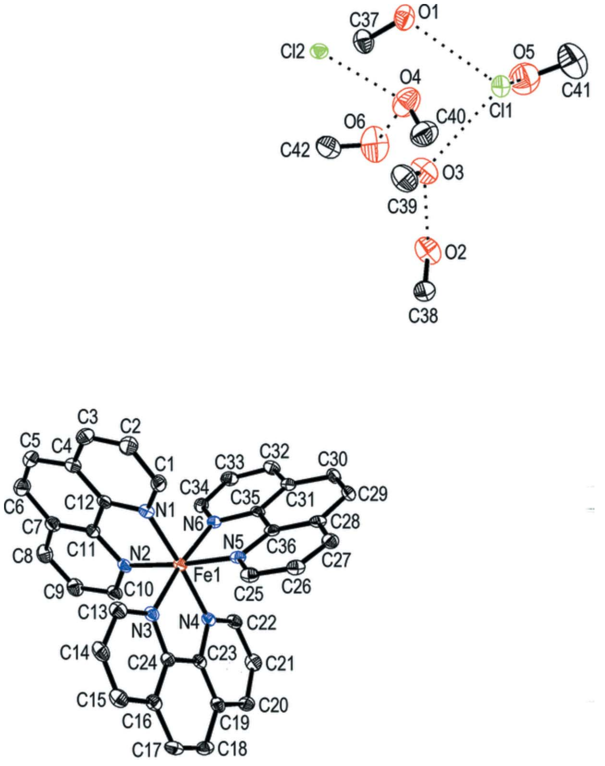


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

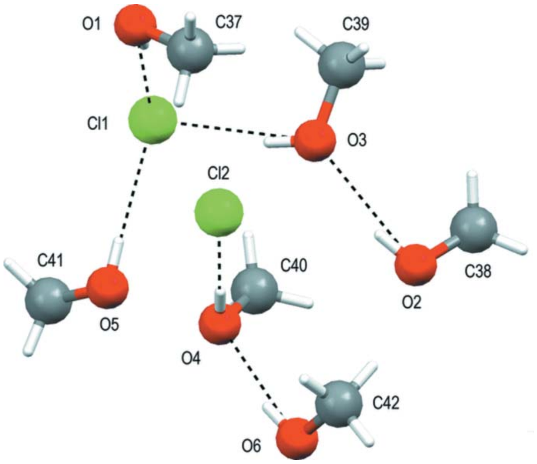


Figure 2
Hydrogen-bonded $[Cl(MeOH)_4]^-$ and $[Cl(MeOH)_2]^-$ clusters. Dashed lines represent O···O and O···Cl contacts.

anions are packed within the crystal structure without the formation of any obvious layers, and are ordered as rods along the [100] axis (Fig. 3).

Experimental

$FeCl_2{\cdot}4H_2O$ (100 mg, 0.5 mmol) was dissolved in methanol (4 ml). Traces of impurities were removed by filtration under an N_2 atmosphere using standard Schlenk techniques. Next, phen·H_2O (120 mg, 0.6 mmol) was added, which caused the colour of the solution to turn red and a dark-red crystalline solid to precipitate. The mixture was left undisturbed for some days at room temperature for crystallization, affording large dark-red crystals (*ca* 4 mm) of (I).

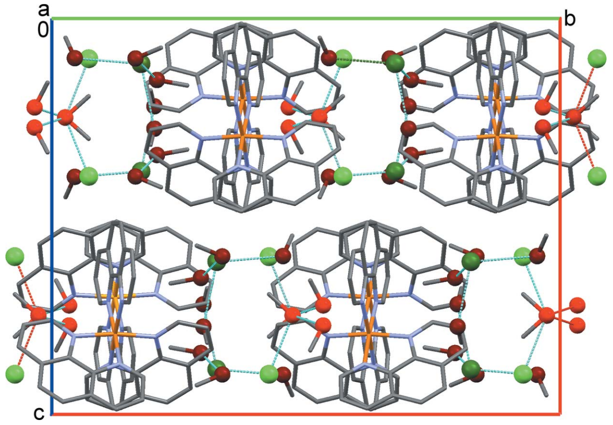


Figure 3
A packing diagram for (I), viewed along the [100] axis. Dashed lines indicate O···O and O···Cl contacts in hydrogen bonds. For clarity, H atoms have been omitted and C atoms are shown as sticks. The spheres represent hydrogen-bond partners within $[Cl(MeOH)_4]^-$ (darker) and $[Cl(MeOH)_2]^-$ (lighter) assemblies. C atoms are shown in grey, N atoms in blue, Fe atoms in orange, Cl atoms in green and O atoms in red.

Crystal data

$[Fe(C_{12}H_8N_2)_3]Cl_2{\cdot}6CH_4O$
$M_r = 859.61$
Orthorhombic, *Pbca*
$a = 18.5808\ (9)$ Å
$b = 23.6715\ (14)$ Å
$c = 18.4205\ (11)$ Å
$V = 8102.0\ (8)$ Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.56$ mm^{-1}
$T = 120\ (2)$ K
0.31 × 0.19 × 0.19 mm

Data collection

Oxford Diffraction KM-4-CCD area-detector diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.898$, $T_{max} = 0.956$
46880 measured reflections
7162 independent reflections
5069 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.164$
$S = 1.03$
7162 reflections
526 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.33$ e Å^{-3}
$\Delta\rho_{min} = -0.71$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N1	1.971 (3)	Fe1—N4	1.970 (3)
Fe1—N2	1.978 (3)	Fe1—N5	1.971 (3)
Fe1—N3	1.979 (3)	Fe1—N6	1.975 (3)
N1—Fe1—N2	83.10 (12)	N2—Fe1—N6	93.39 (12)
N1—Fe1—N3	93.45 (13)	N3—Fe1—N4	83.11 (12)
N1—Fe1—N4	175.70 (13)	N3—Fe1—N5	92.91 (12)
N1—Fe1—N5	93.47 (12)	N3—Fe1—N6	173.18 (12)
N1—Fe1—N6	92.04 (12)	N4—Fe1—N5	89.30 (12)
N2—Fe1—N3	91.28 (12)	N4—Fe1—N6	91.58 (12)
N2—Fe1—N4	94.37 (12)	N5—Fe1—N6	82.72 (12)
N2—Fe1—N5	174.73 (12)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
O1—H1*A*$\cdots$Cl1	0.84	2.33	3.157 (3)	167
O2—H2*A*$\cdots$O3	0.84	2.06	2.873 (5)	164
O3—H3*A*$\cdots$Cl1	0.84	2.22	3.046 (4)	166
O4—H4*A*$\cdots$Cl2	0.84	2.17	3.011 (4)	178
O5—H5*A*$\cdots$Cl1	0.84	2.35	3.164 (5)	164
O6—H6*A*$\cdots$O4	0.84	1.88	2.711 (7)	168

All H atoms were placed in calculated positions, with C—H distances of 0.95 for aryl CH and 0.98 Å for methyl CH_3, and with O—H distances of 0.84 Å, and refined as riding on their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}(C,O)$ or $1.5U_{eq}$(methyl C). Methyl and hydroxyl functionalities in MeOH molecules were treated as rigid groups but were allowed to rotate about their common C—O bonds. The highest peak is located 1.27 Å from O6.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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