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Aquachloro(dimethyl sulfoxide-κO)(pyridine-2,6-dicarboxylato-$\kappa^3 O,N,O'$)iron(III)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.083
Data-to-parameter ratio = 22.5

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

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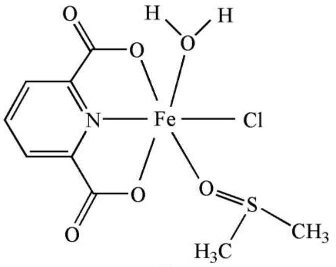
In the molecule of the title compound, $[FeCl(C_7H_3NO_4)(C_2H_6OS)(H_2O)]$, the Fe ion is six-coordinate and has a distorted octahedral geometry. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds, linking the molecules in chains.

Comment

Recently, we reported the synthesis and crystal structures of the $\{[Ce(pydcH)(pydc)(H_2O)_2]\cdot 4H_2O\}_n$ and $\{[Sm(pydcH)(pydc)(H_2O)_2]\cdot 4H_2O\}_n$ (Rafizadeh, Amani, Iravani & Neumuller, 2005) and $[Tl(pydcH]_n$ complexes (Rafizadeh, Amani & Neumuller, 2005) (where H$_2$pydc is 2,6-pyridinedicarboxylic acid). In these complexes, pydc acts as an N– and O-atom donor ligand, forming coordination polymers in the solid state.



(I)

A literature survey reveals that attempts have been made to synthesize and characterize transition metal complexes of the pydc ligand. Complexes of this ligand with mono-, bi- and trivalent transition metals, such as $[Ag(pydcH)(pydcH_2)]\cdot H_2O$ (Drew *et al.*, 1970), $[Ni(pydcH)_2]\cdot 3H_2O$ (Cıngı *et al.*, 1972) and $[Fe_2(pydc)_2(H_2O)_6]\cdot 2pydcH_2$ (Laine *et al.*, 1995), have been reported. There are also reports on the reaction of H$_2$pydc with lanthanides in the presence of a Lewis base such as pyda (2,6-diaminopyridine), which lead to the complexes $(H_5O_2)(Hpyda)_2[(Yb(pydc)_3)]\cdot 2H_2O$ (Rafizadeh *et al.*, 2004), $[(H_5O_2)(pydaH)_2(Gd(pydc)_3)]\cdot 2H_2O$ (Rafizadeh, Ranjbar & Amani, 2005) and $[(H_5O_2)(pyda\cdot H)_2(Dy(pydc)_3)]\cdot 2H_2O$ (Rafizadeh *et al.*, 2006). Here, we report the crystal structure of the title compound, (I).

The coordination of the Fe atom in (I) is distorted octahedral. The sites occupied by the O atoms of the pydc ligand show a distortion in the equatorial plane. The O1—Fe1—O3 angle is distorted from linearity by about 29°. The other four sites around the Fe atom are occupied by two O atoms of dimethyl sulfoxide and water molecules, one N atom of the pydc ligand and one Cl atom, producing a square without any distortion. The Fe—Cl, Fe—N and mean Fe—O bond lengths

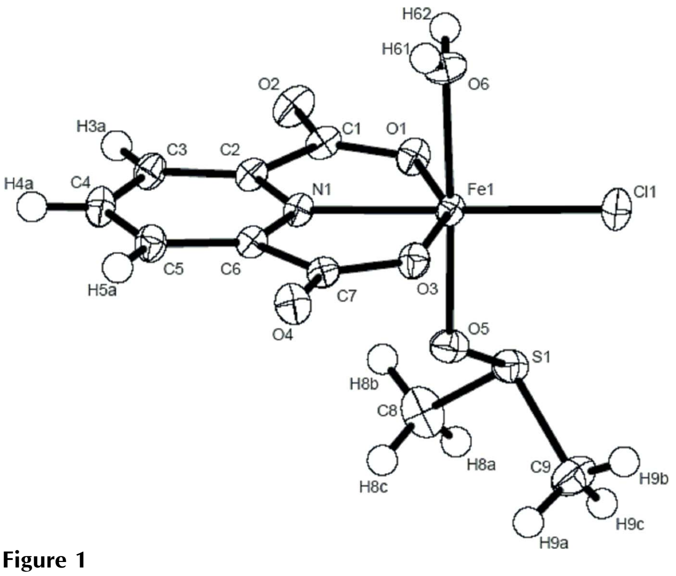


Figure 1
The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

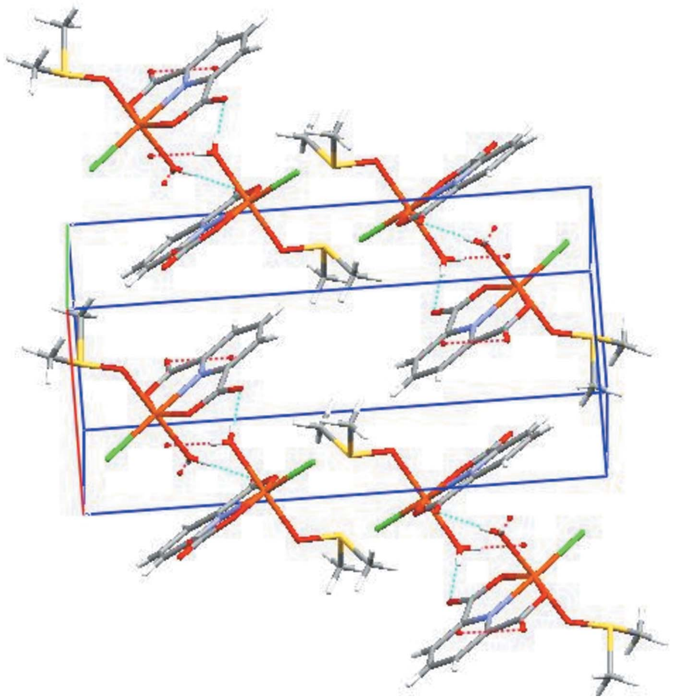


Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(Table 1) are in agreement with the corresponding ones in $[Fe_2(pydc)_2(H_2O)_6]\cdot 2pydcH_2$ (Laine *et al.*, 1995).

The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds (Table 2), linking the molecules in chains (Fig. 2).

Experimental

To a solution of H_2pydc (0.7 g, 4.2 mmol) in DMSO (20 ml) was added a solution of $FeCl_3\cdot 6H_2O$ (1.135 g, 4.2 mmol) in water (40 ml) and the resulting pale-yellow solution was refluxed at 343 K for 5 h. This solution was left to evaporate slowly at room temperature. After six months, green–yellow prismatic crystals were isolated (yield 1.04 g, 70.2%; m.p. 489 K).

Crystal data

$[FeCl(C_7H_3NO_4)(C_2H_6OS)(H_2O)]$
$M_r = 352.55$
Monoclinic, $P2_1/n$
$a = 7.2641$ (4) Å
$b = 10.3018$ (4) Å
$c = 17.7667$ (10) Å
$\beta = 90.014$ (5)°
$V = 1329.54$ (12) Å^3
$Z = 4$
$D_x = 1.761$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.51$ mm^{-1}
$T = 296$ (2) K
Prism, green–yellow
0.60 × 0.45 × 0.31 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2004)
$T_{min} = 0.554$, $T_{max} = 0.732$
27972 measured reflections
4048 independent reflections
3563 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 30.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.083$
$S = 1.04$
4048 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.6263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O5	2.0140 (14)	Fe1—O1	2.0497 (12)
Fe1—O3	2.0203 (12)	Fe1—N1	2.0746 (13)
Fe1—O6	2.0427 (14)	Fe1—Cl1	2.2669 (5)
O5—Fe1—O3	92.17 (5)	O6—Fe1—N1	88.09 (6)
O5—Fe1—O6	174.66 (6)	O1—Fe1—N1	75.30 (5)
O3—Fe1—O6	91.86 (6)	O5—Fe1—Cl1	91.52 (5)
O5—Fe1—O1	88.39 (6)	O3—Fe1—Cl1	102.70 (4)
O3—Fe1—O1	151.27 (5)	O6—Fe1—Cl1	91.01 (5)
O6—Fe1—O1	86.40 (6)	O1—Fe1—Cl1	106.01 (4)
O5—Fe1—N1	89.48 (6)	N1—Fe1—Cl1	178.37 (4)
O3—Fe1—N1	75.98 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H61···O2^{i}	0.75 (3)	1.92 (3)	2.669 (2)	176 (3)
O6—H62···O4^{ii}	0.73 (3)	1.93 (3)	2.6620 (19)	176 (3)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Water H atoms were located in a difference sythesis and refined isotropically. The remaining H atoms were positioned geometrically [with C—H = 0.93 and 0.96Å for aromatic and methyl H atoms, respectively] and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s)

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used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-STEP* (Stoe & Cie, 2000); software used to prepare material for publication: *SHELXL97*.

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