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catena-Poly[[*(2,2'*-bipyridine)iron(II)]- μ -oxalato]

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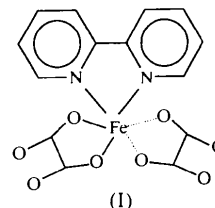
Abstract

The crystal structure of *catena*-poly[[*(2,2'*-bipyridine-*N,N'*)iron(II)]- μ -(oxalato-*O*¹,*O*²:*O*¹,*O*^{2'})], [Fe(C₂O₄)(C₁₀H₈N₂)]_n, is reported. The Fe^{II} atom is six-coordinated with distorted octahedral geometry and the bipyridine ring is in a planar form. The oxalate anion bridges the metal ions forming infinite chains.

Comment

Recently, oxalate-bridged polymeric compounds, especially those with higher dimensionalities, have attracted much attention due to their interesting magnetic properties (Decurtins *et al.*, 1994, and references therein). Using *2,2'*-bipyridine (*2,2'*-bipy) as a terminal ligand, chain compounds can be obtained which contain ox-

alate anions as bridging ligands. So far, there have been only two such compounds reported, *viz.* [Cu(C₂O₄)(*2,2'*-bipy)]·2H₂O (Fitzgerald *et al.*, 1982) and [Mn(C₂O₄)(*2,2'*-bipy)] (Deguenon *et al.*, 1990). We report herein an Fe^{II} compound, [Fe(C₂O₄)(*2,2'*-bipy)], (I), which is isostructural with the Mn^{II} compound.



An examination of the relative geometrical positions of the planar *2,2'*-bipy and oxalate ligands (Fig. 1) shows no particular kind of stacking, although the planes are nearly coplanar. The crystal structure of the title compound consists of neutral [Fe(C₂O₄)(*2,2'*-bipy)] units, with the Fe^{II} ions linked by C₂O₄ ligands to form infinite 'zigzag' chains along the *a* axis. The Fe^{II} atom has a distorted octahedral coordination environment which is made up of two N atoms from the *2,2'*-bipyridine ligand and four O atoms from the oxalate ligands. The Fe—N bond lengths [2.159 (4) and 2.173 (4) Å] and the N—Fe—N bite angle [75.7 (1)°] are comparable to corresponding values in [Fe₄O₂(O₂CCH₃)₇(bipy)₂]ClO₄·0.25CH₂Cl₂·H₂O [2.145 (6), 2.185 (6) Å and 74.9 (2)°; McCusker *et al.*,

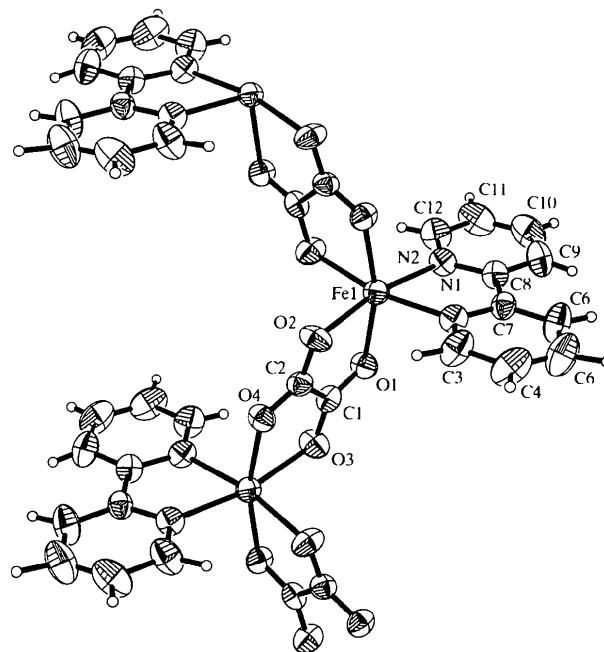


Fig. 1. Part of the polymer chain of (I), showing 50% probability displacement ellipsoids, with the asymmetric unit labelled.

1991]. The Fe—O distances [2.092 (3)–2.162 (4) Å] and the O—Fe—O bite angles [77.9 (1) and 93.4 (1)°] are in agreement with those in [Fe(bipy)₃][Fe₂(C₂O₄)₃] [2.128 (2), 2.122 (2) Å and 78.8 (1)°; Decurtins *et al.*, 1993]. The bite angles of the two oxalate anions are also close to those in [Cu(C₂O₄)(2,2'-bipy)]·2H₂O [77.9 (2) and 77.4 (2)°; Fitzgerald *et al.*, 1982] and [Mn(C₂O₄)(2,2'-bipy)] [76.5 (2) and 76.8 (2)°; Deguenon *et al.*, 1990]. The small N1—C7—C8 and N2—C8—C7 bond angles [average 115.7 (5)°] compared with C6—C7—C8 and C7—C8—C9 [average 123.1 (6)°] show the distortion of the 2,2'-bipyridine ligand due to the coordination. The dihedral angle between the planar pyridyl rings is 2.0 (2)°. The oxalate anion is planar, with the largest deviation being −0.024 (4) Å for O3. The shortest Fe···Fe distance within the chain is 5.529 (1) Å and that between chains is 7.557 (2) Å. Neighboring chains are connected to each other *via* van der Waals interactions, with the bipyridyl ligands stacked between the chains.

Experimental

Dark-red crystals of the title compound were obtained by heating a mixture containing K₃Fe(C₂O₄)₃·3H₂O (0.5 mmol), 2,2'-bipyridine (0.5 mmol) and H₂O (10 ml) at 453 K for 20 h. Analysis found: C 47.72, H 2.33, N 9.41%; C₁₂H₈FeN₂O₄ requires: C 48.02, H 2.67, N 9.34%.

Crystal data

[Fe(C ₂ O ₄)(C ₁₀ H ₈ N ₂)]	Mo K α radiation
$M_r = 300.05$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 2958 reflections
Pna_2_1	$\theta = 2.89$ – 33.66°
$a = 9.3229$ (3) Å	$\mu = 1.252$ mm ^{−1}
$b = 9.2536$ (1) Å	$T = 293$ (2) K
$c = 14.0823$ (4) Å	Plate
$V = 1214.89$ (5) Å ³	$0.36 \times 0.10 \times 0.02$ mm
$Z = 4$	Red
$D_x = 1.640$ Mg m ^{−3}	
D_m not measured	

Data collection

Siemens SMART CCD area-detector diffractometer	1977 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.069$
Absorption correction: empirical (SADABS; Sheldrick, 1996)	$\theta_{max} = 30^\circ$
$T_{min} = 0.637$, $T_{max} = 0.975$	$h = 0 \rightarrow 13$
8714 measured reflections	$k = 0 \rightarrow 13$
1836 independent reflections (plus 1555 Friedel-related reflections)	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$\Delta\rho_{max} = 0.314$ e Å ^{−3}
$wR(F^2) = 0.092$	$\Delta\rho_{min} = -0.312$ e Å ^{−3}

$S = 1.112$
3390 reflections
204 parameters
All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0117P)^2 + 0.1653P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.04 (3)

Table 1. Selected geometric parameters (Å, °)

Fe1—O4 ⁱ	2.092 (3)	Fe1—N1	2.159 (4)
Fe1—O1	2.103 (3)	Fe1—O3 ⁱ	2.162 (4)
Fe1—O2	2.150 (4)	Fe1—N2	2.173 (4)
O4 ⁱ —Fe1—O1	164.26 (10)	N1—Fe1—O3 ⁱ	168.3 (2)
O4 ⁱ —Fe1—O2	90.84 (13)	O4 ⁱ —Fe1—N2	97.60 (14)
O1—Fe1—O2	77.93 (13)	O1—Fe1—N2	95.04 (14)
O4 ⁱ —Fe1—N1	95.67 (14)	O2—Fe1—N2	168.9 (2)
O1—Fe1—N1	96.53 (14)	N1—Fe1—N2	75.70 (13)
O2—Fe1—N1	96.4 (2)	O3 ⁱ —Fe1—N2	95.3 (2)
O4 ⁱ —Fe1—O3 ⁱ	77.88 (13)	C1—O3—Fe1 ⁱⁱ	113.1 (3)
O1—Fe1—O3 ⁱ	91.66 (14)	C2—O4—Fe1 ⁱⁱ	115.7 (3)
O2—Fe1—O3 ⁱ	93.41 (13)		

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

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99%. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections; it was found to be negligible.

Data collection: SMART (Siemens, 1996a). Cell refinement: SAINT (Siemens, 1996b). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Siemens, 1996c). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

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Aminoguanidinium (ethylenediamine-*N,N,N',N'*-tetraacetato)antimonate(III) monohydrate

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Abstract

In the title complex, (CH₇N₄)[Sb(C₁₀H₁₂N₂O₈)]·H₂O, the lone-pair electrons of the Sb atom occupy an axial site in the ψ -pentagonal bipyramidal polyhedron [Sb—N 2.265 (2)–2.507 (2) and Sb—O 2.144 (1)–2.543 (2) Å]. The (ethylenediaminetetraacetato)antimonate anion, the aminoguanidinium cation and the lattice water are linked by hydrogen bonds into a two-dimensional network structure.

Comment

For six-coordinate antimony(III) compounds, the VSEPR (valence-shell electron-pair repulsion) model (Gillespie, 1992) predicts a ψ -pentagonal bipyramidal geometry at the metal atom in which the lone pair of electrons occupies an axial site, a feature that has been docu-

mented in, for example, the tris(oxalato)antimonate(III) ion (Poore & Russell, 1971). Hexacoordination can be forced on the Sb atom by the use of a single diaminetetracarboxylate polyanion, which chelates to the Sb atom through four carboxyl O and two amino N atoms; polyanions that have been used for this purpose include the ethylenediaminetetraacetate (edta), propylenediaminetetraacetate and *trans*-cyclohexanediaminetetraacetate tetraanions (Hu *et al.*, 1998).

Among the ethylenediaminetetraacetate complexes, the lone pair is axially positioned in [(CH₃)₄N][Sb(edta)]·2H₂O (Ilyukhin & Davidovich, 1999), as predicted by the VSEPR model. However, the lone pair is equatorially positioned in [NH₄][Sb(edta)]·H₂O (Ilyukhin & Davidovich, 1999) and in neutral [Sb(Hedta)]·2H₂O (Shimoi *et al.*, 1980). The difference in the geometry of the metal atom in the two salts can be ascribed to the more polarizable nature of the ammonium cation compared with that of the tetramethylammonium cation, since polarizability discourages localization of the lone pair. In the sodium salt Na[Sb(edta)]·3H₂O, the [Sb(edta)][−] ion lies on a twofold axis, and the pentagonal plane that is formed by the two O atoms, the two N atoms and the lone pair is planar. From its equatorial position, the lone pair distorts the axial O—Sb—O skeleton [O—Sb—O = 145.3 (2)°; Xie & Hu, 1991]. In the less polarizable guanidinium cation, the charge formally resides on the central C atom; a study of [CH₆N₃][Sb(edta)]·2H₂O has documented a ψ -pentagonal bipyramidal geometry in which the lone pair is axially disposed. The three amino groups of the cation are all bifurcated, and hydrogen bonds link the cation, the lattice water molecules and the anion into a one-dimensional chain structure. In addition, the crystal structure shows two weak Sb···O interactions (3.464 and 3.467 Å; Mistryukov *et al.*, 1987). The lone pair also lies in the equatorial position in [NH₄][Sb(cdta)]·[NH₄]Cl (H₄cdta = *trans*-cyclohexane-1,2-diaminetetraacetic acid; Hu *et al.*, 1998).

The introduction of another amino group to the guanidinium cation to give the title compound, (I), should lead to further delocalization of the positive charge; although (I) crystallizes with only one lattice water, the N-bonded amino group does not engage in hydrogen bonding with either the lattice water or any of the carbonyl O atoms. The N-bonded amino N atom is, in fact,

