metal-organic compounds

catena-Poly[[[diaquairon(II)]-*μ*-pyrazine-2,3-dicarboxylato] dihydrate]

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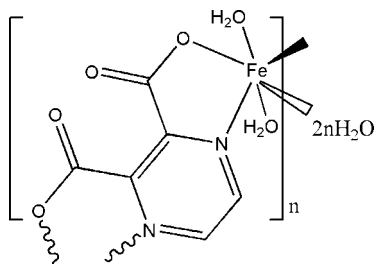Correspondence e-mail: xuhyun1970@sohu.com

The crystal structure of the title compound, $\{[Fe(C_6H_2N_2O_4)(H_2O)_2]\cdot2H_2O\}_n$, was synthesized by a diffusion method. It has a one-dimensional polymeric chain structure and the chains are further connected into a three-dimensional structure by hydrogen bonds. The Fe^{II} ion has a distorted octahedral coordination environment, with two N and two O atoms from the pyrazine-2,3-dicarboxylate ligands in the equatorial plane and with two water molecules in axial positions. The Fe atom lies on a crystallographic centre of symmetry and a twofold rotation axis passes through the pyrazine ring.

Related literature

For related literature, see: Kondo *et al.* (1999); Kitaura *et al.* (2002); Zheng *et al.* (2002); Mao *et al.* (1996); Castillo *et al.* (2003); Konar *et al.* (2004); Muranishi & Okabe (2003); Richard *et al.* (1973); Xiang *et al.* (2004); Zou *et al.* (1999).

Experimental

Crystal data

$[Fe(C_6H_2N_2O_4)(H_2O)_2]\cdot2H_2O$	c = 11.8314 (2) Å
M_r = 294.01	β = 110.759 (1)°
Monoclinic, $C2/c$	V = 1044.77 (3) Å^3
a = 12.5650 (2) Å	Z = 4
b = 7.5158 (1) Å	Mo $K\alpha$ radiation
μ = 1.48 mm^{-1}	0.23 × 0.20 × 0.18 mm
T = 298 (2) K	

Data collection

Siemens SMART CCD area-detector diffractometer	$wR(F^2)$ = 0.088
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.727, T_{max} = 0.777	S = 1.03
	1291 reflections
	80 parameters
5477 measured reflections	H-atom parameters constrained
1291 independent reflections	$\Delta\rho_{max}$ = 0.59 e Å^{-3}
1219 reflections with $I > 2\sigma(F^2)$] = 0.029	$\Delta\rho_{min}$ = −0.70 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4A···O1^i	0.85	2.17	2.890 (3)	142
O4—H4A···O2^i	0.85	2.59	3.227 (3)	133
O4—H4B···O1^{ii}	0.85	2.20	3.045 (3)	174
O3—H3A···O4^{iii}	0.85	2.41	3.210 (3)	156
O3—H3B···O2^{iv}	0.85	1.98	2.720 (2)	145
C3—H3···O2^v	0.93	2.51	3.232 (3)	135

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2063).

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supplementary materials

catena-Poly[[[diaquairon(II)]-*μ*-pyrazine-2,3-dicarboxylato] dihydrate]

H. Xu, H. Ma, M. Xu, W. Zhao and B. Guo

Comment

Recently, the effective combination of coordination bond and hydrogen bond has been applied in the engineering study of inorganic-organic hybrid material and the construction of metal-organic coordination supramolecular complexes. The suitable organic ligand makes the complex not only to possess novel structure but also produces unique optical, electric and magnetic properties. Pyrazine-2,3-dicarboxylic acid (pzdcH2) has proved to be well suited for the construction of multidimensional frameworks, due to the presence of two adjacent carboxylate groups (O donor atoms) as substituents on the N-heterocyclic pyrazine ring (N donor atoms). A series of one-dimensional, two-dimensional and three-dimensional metal-organic coordination supramolecular complexes have been synthesized and characterized. Now, we report the crystal structure of the title compound (I), and the crystal structure is similar to the structures reported by Mao *et al.* (1996). In compound 1, the iron atom is hexacoordinate where the sphere about any iron atom includes the N1, N1A, O1, O1A, O3 and O3A atoms. The Fe atom lies on a crystallographic center of symmetry and that the ligand lies on a crystallographic twofold axis. Two coordinated water molecules are on the axis. The coordination distances for the Fe—O1 2.054 (1) Å are similar with the usual carboxyl oxygen to iron distance of 2.091 Å. The pzdc dianion ligands bridge Fe ions to form extended linear chains. In this structure, the pzdc dianion ligand coordinates to two metal centers *via* chelate interactions involving each nitrogen N(1) and oxygen O(1) from the adjacent carboxylate substituent (Fig. 1). As shown in Fig. 2, the chains are linked in a 3-D surpramolecular network by O—H⋯O hydrogen-bonding interactions.

Experimental

The title compound was obtained by a diffusion method. In one arm of U-tube was placed (C$_6$H$_2$N$_2$O$_4$)Na$_2$ (42 mg, 0.2 mmol) in water/ethanol (1:1; 10 ml) and in the other H$_{12}$Cl$_2$O$_{14}$Fe (73 mg, 0.2 mmol) in water/ethanol (1:1; 10 ml). The red crystals were collected by filtration, washed with distilled water, followed by ethanol and dried under reduced pressure for 2 h.

Analysis found: C 24.39, H 3.41, N 9.26%; C$_6$H$_{10}$N$_2$O$_8$Fe requires: C 24.51, H 3.43, N 9.53%.

Refinement

The H-atoms were included in the riding-model approximation with C—H = 0.93 - 0.96 Å and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C-aromatic).
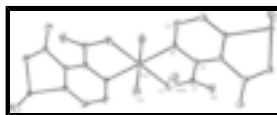
Figures

Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. The H atoms are omitted for clarity.
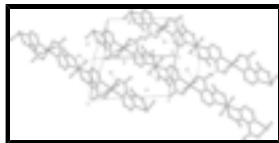
supplementary materials

Fig. 2. three-dimensional supramolecular network of (I). O—H···O hydrogen bonds interactions shown.

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Crystal data

[Fe(C$_6$H$_2$N$_2$O$_4$)(H$_2$O)$_2$]·2H$_2$O	$F_{000} = 600$
$M_r = 294.01$	$D_x = 1.869$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 3554 reflections
$a = 12.5650$ (2) Å	$\theta = 3.3–28.2º$
$b = 7.5158$ (1) Å	$\mu = 1.48$ mm^{-1}
$c = 11.8314$ (2) Å	$T = 298$ (2) K
$\beta = 110.759$ (1)º	Block, red
$V = 1044.77$ (3) Å^3	$0.23 \times 0.20 \times 0.18$ mm
$Z = 4$	

Data collection

CCD area-detector diffractometer	1291 independent reflections
Radiation source: fine-focus sealed tube	1219 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 298(2)$ K	$\theta_{max} = 28.3º$
φ and ω scans	$\theta_{min} = 3.2º$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12\rightarrow16$
$T_{min} = 0.727$, $T_{max} = 0.777$	$k = -10\rightarrow10$
5477 measured reflections	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 1.9364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.59$ e Å^{-3}
1291 reflections	$\Delta\rho_{min} = -0.70$ e Å^{-3}
80 parameters	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.028 (2)

Secondary atom site location: difference Fourier map

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\mathring{A}^2$)

	x	y	z	$U_{iso}*/U_{eq}$
Fe1	0.2500	0.2500	0.5000	0.01865 (18)
C1	0.08321 (16)	−0.0176 (3)	0.38935 (17)	0.0225 (4)
C2	0.04250 (15)	0.1436 (3)	0.30732 (16)	0.0202 (4)
C3	0.04814 (18)	0.4486 (3)	0.30161 (19)	0.0289 (5)
H3	0.0840	0.5557	0.3320	0.035*
N1	0.09020 (14)	0.2975 (2)	0.35810 (15)	0.0228 (3)
O1	0.18127 (13)	−0.0012 (2)	0.47077 (14)	0.0283 (3)
O2	0.01979 (14)	−0.1458 (2)	0.37732 (17)	0.0378 (4)
O3	0.32048 (15)	0.2010 (3)	0.36718 (16)	0.0387 (4)
H3A	0.3336	0.0941	0.3525	0.046*
H3B	0.3619	0.2805	0.3524	0.046*
O4	0.1817 (2)	0.2742 (3)	0.14299 (19)	0.0509 (6)
H4A	0.1531	0.1832	0.1004	0.061*
H4B	0.2226	0.3406	0.1168	0.061*

Atomic displacement parameters ($\mathring{A}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0141 (2)	0.0209 (3)	0.0166 (2)	−0.00144 (12)	0.00016 (15)	−0.00229 (12)
C1	0.0209 (8)	0.0219 (9)	0.0224 (8)	0.0007 (7)	0.0047 (7)	0.0014 (7)
C2	0.0158 (8)	0.0194 (9)	0.0222 (9)	−0.0005 (6)	0.0030 (7)	0.0004 (7)
C3	0.0279 (10)	0.0193 (9)	0.0318 (11)	−0.0031 (8)	0.0010 (9)	−0.0023 (8)
N1	0.0181 (7)	0.0223 (8)	0.0226 (8)	−0.0007 (6)	0.0005 (5)	−0.0013 (6)
O1	0.0244 (7)	0.0240 (6)	0.0276 (7)	−0.0012 (5)	−0.0018 (6)	0.0040 (5)
O2	0.0301 (8)	0.0273 (8)	0.0484 (10)	−0.0073 (6)	0.0047 (7)	0.0081 (7)
O3	0.0317 (9)	0.0544 (11)	0.0337 (9)	−0.0109 (8)	0.0160 (7)	−0.0108 (8)
O4	0.0651 (14)	0.0589 (13)	0.0310 (9)	−0.0257 (10)	0.0200 (10)	−0.0104 (8)

Geometric parameters (Å, °)

Fe1—O1^i	2.0539 (15)	C2—N1	1.343 (3)
Fe1—O1	2.0539 (15)	C2—C2^{ii}	1.398 (3)
Fe1—O3	2.0919 (17)	C3—N1	1.329 (3)
Fe1—O3^i	2.0919 (17)	C3—C3^{ii}	1.381 (4)
Fe1—N1^i	2.1420 (17)	C3—H3	0.9300
Fe1—N1	2.1420 (17)	O3—H3A	0.8500
C1—O2	1.226 (3)	O3—H3B	0.8500
C1—O1	1.273 (2)	O4—H4A	0.8500
C1—C2	1.523 (3)	O4—H4B	0.8501
O1^i—Fe1—O1	180.0	O2—C1—C2	119.56 (17)
O1^i—Fe1—O3	91.35 (7)	O1—C1—C2	114.93 (16)
O1—Fe1—O3	88.65 (7)	N1—C2—C2^{ii}	120.03 (11)
O1^i—Fe1—O3^i	88.65 (7)	N1—C2—C1	113.86 (16)
O1—Fe1—O3^i	91.35 (7)	C2^{ii}—C2—C1	125.94 (10)
O3—Fe1—O3^i	180.000 (1)	N1—C3—C3^{ii}	120.82 (11)
O1^i—Fe1—N1^i	78.46 (6)	N1—C3—H3	119.6
O1—Fe1—N1^i	101.54 (6)	C3^{ii}—C3—H3	119.6
O3—Fe1—N1^i	91.76 (7)	C3—N1—C2	118.46 (17)
O3^i—Fe1—N1^i	88.24 (7)	C3—N1—Fe1	129.20 (14)
O1^i—Fe1—N1	101.54 (6)	C2—N1—Fe1	110.65 (13)
O1—Fe1—N1	78.46 (6)	C1—O1—Fe1	116.94 (13)
O3—Fe1—N1	88.24 (7)	Fe1—O3—H3A	118.9
O3^i—Fe1—N1	91.76 (7)	Fe1—O3—H3B	118.9
N1^i—Fe1—N1	180.0	H3A—O3—H3B	116.4
O2—C1—O1	125.39 (19)	H4A—O4—H4B	116.0

Symmetry codes: (i) −x+1/2, −y+1/2, −z+1; (ii) −x, y, −z+1/2.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O1^{iii}	0.85	2.17	2.890 (3)	142
O4—H4A···O2^{iii}	0.85	2.59	3.227 (3)	133
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O3—H3B···O2^{vi}	0.85	1.98	2.720 (2)	145
C3—H3···O2^{vii}	0.93	2.51	3.232 (3)	135

Symmetry codes: (iii) x, −y, z−1/2; (iv) −x+1/2, y+1/2, −z+1/2; (v) −x+1/2, y−1/2, −z+1/2; (vi) x+1/2, y+1/2, z; (vii) x, y+1, z.

Fig. 1

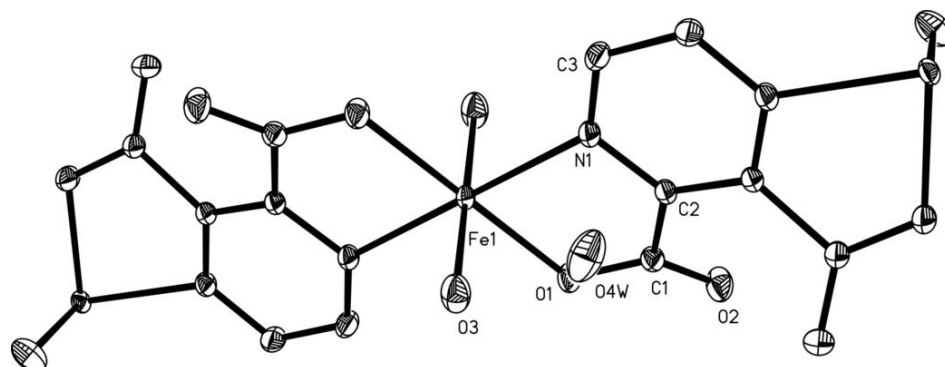**Fig. 2**