

catena-Poly[[[tetraaquairon(II)]- μ -5,5'-diazenediyl]ditetrazolido] dihydrate]

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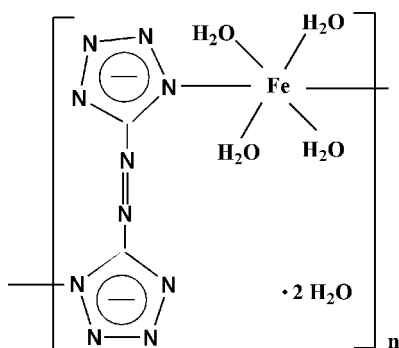
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{N}-\text{C}) = 0.003$ Å; R factor = 0.026; wR factor = 0.070; data-to-parameter ratio = 10.0.

In the title compound, $[\text{Fe}(\text{C}_2\text{N}_{10})(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$, the coordination geometry around the Fe(II) atom, which lies on a center of inversion, is distorted octahedral, with bonds to four O atoms and two N atoms. The azotetrazolate ligand displays a bridging coordination mode, forming an infinite zigzag chain. Intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonding and offset face-to-face $\pi-\pi$ stacking interactions [centroid-centroid distance = 3.4738 (13) Å] lead to a three-dimensional network.

Related literature

For energetic complexes, see: Hammerl *et al.* (2001, 2002); Jiao *et al.* (2007).



Experimental

Crystal data

$[\text{Fe}(\text{C}_2\text{N}_{10})(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$
 $M_r = 328.07$

Triclinic, $P\bar{1}$
 $a = 6.2449$ (5) Å

$b = 6.9764$ (6) Å
 $c = 7.8256$ (6) Å
 $\alpha = 76.424$ (1)°
 $\beta = 74.135$ (1)°
 $\gamma = 69.844$ (1)°
 $V = 304.11$ (4) Å³

$Z = 1$
Mo $K\alpha$ radiation
 $\mu = 1.29$ mm⁻¹
 $T = 273$ K
 $0.30 \times 0.18 \times 0.12$ mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.699$, $T_{\max} = 0.861$

1564 measured reflections
1061 independent reflections
973 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.012$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.070$
 $S = 1.13$
1061 reflections
106 parameters
6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2A} \cdots \text{O3}$	0.85 (1)	1.86 (1)	2.699 (2)	170 (2)
$\text{O1}-\text{H1A} \cdots \text{O3}^{\text{i}}$	0.85 (1)	1.87 (1)	2.715 (2)	171 (3)
$\text{O1}-\text{H1B} \cdots \text{N5}$	0.85 (1)	2.23 (2)	2.926 (2)	139 (2)
$\text{O3}-\text{H3B} \cdots \text{N3}^{\text{ii}}$	0.84 (1)	2.01 (1)	2.839 (2)	169 (2)
$\text{O3}-\text{H3A} \cdots \text{N2}^{\text{iii}}$	0.85 (1)	2.00 (1)	2.843 (2)	173 (2)
$\text{O2}-\text{H2B} \cdots \text{N3}^{\text{iv}}$	0.85 (1)	2.69 (2)	3.439 (2)	148 (2)
$\text{O2}-\text{H2B} \cdots \text{N4}^{\text{iv}}$	0.85 (1)	1.99 (1)	2.840 (2)	173 (2)

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5036).

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supplementary materials

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***catena*-Poly[[[tetraaquairon(II)]- μ -5,5'-diazenediylditetrazolido] dihydrate]**

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Comment

After Thiele prepared metallic salts of azotetrazole and claimed these for use in initiators, salts of AT^{2-} ($AT = 5,5'$ -azotetrazolate) have been extensively investigated and have often been considered for practical use as a class of energetic materials. We report here the crystal structure of the title compound, $[Fe(C_2N_{10})(H_2O)_4 \cdot 2H_2O]_n$, (I).

Single-crystal analysis shows that (I) exists as a one-dimensional infinite chain. As shown in Figure 1, the coordination geometry around Fe^{2+} cation can be described a disordered octahedral arrangement with coordination number of 6, where O1, O2, O1A and O2A form the equatorial plane, and axial positions are occupied by N1 and N1A. Additionally, each AT^{2-} provides two terminal nitrogen atoms (N1 and N1A) acting as bridging ligand to connect two $[Fe(H_2O)_4]^{2+}$ to form an infinite zigzag chain.

In the crystal structure, the interactions of hydrogen bonding between the water molecules and the N atoms in the terazole rings, the off-set face to face π - π stacking interactions of the terazole rings link the complex to a three dimensional structure, as shown in Figure 2.

Experimental

Brown block-like crystal for X-ray diffraction analysis was obtained from the mixture of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ (0.392 g, 1 mmol), $Na_2AT \cdot 5H_2O$ (0.304 g, 1 mmol) and distilled H_2O (20 ml), which was allowed to evaporate at room temperature for one week.

Refinement

H atoms attached to O atoms were placed in calculated positions, with O—H distances of 0.86 Å. The $U_{iso}(H)$ values were constrained to be -1.5Ueq of the carrier atom.

Figures

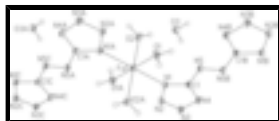


Fig. 1. A view of the molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.



Fig. 2. Three dimensional network of the title complex.

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Crystal data

$[\text{Fe}(\text{C}_2\text{N}_{10})(\text{H}_2\text{O})_4]\cdot 2\text{H}_2\text{O}$	$Z = 1$
$M_r = 328.07$	$F(000) = 168$
Triclinic, PT	$D_x = 1.791 \text{ Mg m}^{-3}$
$a = 6.2449 (5) \text{ \AA}$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$b = 6.9764 (6) \text{ \AA}$	Cell parameters from 1061 reflections
$c = 7.8256 (6) \text{ \AA}$	$\theta = 2.7\text{--}25.1^\circ$
$\alpha = 76.424 (1)^\circ$	$\mu = 1.29 \text{ mm}^{-1}$
$\beta = 74.135 (1)^\circ$	$T = 273 \text{ K}$
$\gamma = 69.844 (1)^\circ$	Block, brown
$V = 304.11 (4) \text{ \AA}^3$	$0.30 \times 0.18 \times 0.12 \text{ mm}$

Data collection

Bruker SMART APEX CCD diffractometer	1061 independent reflections
Radiation source: fine-focus sealed tube graphite	973 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.012$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2002)	$\theta_{\text{max}} = 25.1^\circ$, $\theta_{\text{min}} = 2.7^\circ$
$T_{\text{min}} = 0.699$, $T_{\text{max}} = 0.861$	$h = -7 \rightarrow 6$
1564 measured reflections	$k = -7 \rightarrow 8$
	$l = -8 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.026$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.070$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.13$	$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.0115P]$
1061 reflections	where $P = (F_o^2 + 2F_c^2)/3$
106 parameters	$(\Delta/\sigma)_{\text{max}} = 0.007$
6 restraints	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.5000	0.5000	1.0000	0.02375 (17)
N1	0.5314 (3)	0.6482 (3)	0.7101 (2)	0.0240 (4)
N2	0.3404 (3)	0.7685 (3)	0.6503 (2)	0.0287 (4)
N3	0.3949 (3)	0.8147 (3)	0.4742 (2)	0.0323 (4)
N4	0.6210 (3)	0.7259 (3)	0.4135 (2)	0.0287 (4)
N5	0.9299 (3)	0.5062 (3)	0.5738 (2)	0.0253 (4)
O1	0.8288 (3)	0.2802 (3)	0.9369 (2)	0.0340 (4)
O2	0.6617 (3)	0.6912 (3)	1.0508 (2)	0.0403 (4)
O3	0.8598 (3)	0.9497 (3)	0.7946 (2)	0.0342 (4)
C1	0.7009 (3)	0.6250 (3)	0.5618 (3)	0.0228 (4)
H1A	0.829 (4)	0.172 (3)	0.903 (3)	0.034*
H2A	0.710 (4)	0.784 (3)	0.975 (3)	0.034*
H3A	1.002 (2)	0.886 (3)	0.754 (3)	0.034*
H1B	0.922 (4)	0.329 (4)	0.851 (2)	0.034*
H2B	0.658 (4)	0.707 (4)	1.1563 (18)	0.034*
H3B	0.797 (4)	1.008 (3)	0.705 (2)	0.034*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0192 (2)	0.0327 (3)	0.0151 (2)	-0.00481 (17)	-0.00252 (16)	-0.00163 (17)
N1	0.0186 (9)	0.0307 (9)	0.0172 (9)	-0.0021 (7)	-0.0026 (7)	-0.0026 (7)
N2	0.0214 (9)	0.0379 (10)	0.0198 (9)	-0.0024 (8)	-0.0035 (7)	-0.0022 (8)
N3	0.0273 (10)	0.0406 (10)	0.0218 (10)	-0.0020 (8)	-0.0073 (8)	-0.0014 (8)
N4	0.0242 (9)	0.0389 (10)	0.0168 (9)	-0.0043 (8)	-0.0021 (7)	-0.0034 (8)
N5	0.0211 (9)	0.0336 (9)	0.0162 (8)	-0.0053 (7)	-0.0002 (6)	-0.0033 (7)
O1	0.0260 (8)	0.0398 (9)	0.0283 (9)	-0.0044 (7)	-0.0020 (7)	-0.0033 (7)
O2	0.0526 (11)	0.0589 (11)	0.0188 (9)	-0.0333 (9)	-0.0034 (8)	-0.0035 (8)
O3	0.0234 (8)	0.0433 (9)	0.0255 (9)	-0.0014 (7)	-0.0055 (7)	0.0017 (7)
C1	0.0210 (10)	0.0285 (10)	0.0154 (10)	-0.0059 (8)	-0.0023 (8)	-0.0010 (8)

Geometric parameters (\AA , $^\circ$)

Fe1—O2	2.0868 (15)	N4—C1	1.335 (3)
Fe1—O2 ⁱ	2.0868 (15)	N5—N5 ⁱⁱ	1.245 (3)
Fe1—O1 ⁱ	2.1081 (16)	N5—C1	1.400 (3)
Fe1—O1	2.1081 (16)	O1—H1A	0.854 (10)

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Fe1—N1	2.2474 (16)	O1—H1B	0.851 (10)
Fe1—N1 ⁱ	2.2474 (16)	O2—H2A	0.848 (10)
N1—N2	1.333 (2)	O2—H2B	0.850 (10)
N1—C1	1.338 (3)	O3—H3A	0.852 (10)
N2—N3	1.315 (3)	O3—H3B	0.844 (10)
N3—N4	1.331 (3)		
O2—Fe1—O2 ⁱ	180.0	N2—N1—Fe1	119.64 (12)
O2—Fe1—O1 ⁱ	90.39 (7)	C1—N1—Fe1	134.93 (13)
O2 ⁱ —Fe1—O1 ⁱ	89.61 (7)	N3—N2—N1	109.13 (16)
O2—Fe1—O1	89.61 (7)	N2—N3—N4	110.32 (16)
O2 ⁱ —Fe1—O1	90.39 (7)	N3—N4—C1	104.10 (16)
O1 ⁱ —Fe1—O1	180.0	N5 ⁱⁱ —N5—C1	114.3 (2)
O2—Fe1—N1	91.16 (6)	Fe1—O1—H1A	116.1 (17)
O2 ⁱ —Fe1—N1	88.84 (6)	Fe1—O1—H1B	112.4 (17)
O1 ⁱ —Fe1—N1	89.55 (6)	H1A—O1—H1B	104 (2)
O1—Fe1—N1	90.45 (6)	Fe1—O2—H2A	126.3 (16)
O2—Fe1—N1 ⁱ	88.84 (6)	Fe1—O2—H2B	123.0 (16)
O2 ⁱ —Fe1—N1 ⁱ	91.16 (6)	H2A—O2—H2B	109 (2)
O1 ⁱ —Fe1—N1 ⁱ	90.45 (6)	H3A—O3—H3B	107 (2)
O1—Fe1—N1 ⁱ	89.55 (6)	N4—C1—N1	111.80 (17)
N1—Fe1—N1 ⁱ	180.0	N4—C1—N5	127.64 (18)
N2—N1—C1	104.65 (15)	N1—C1—N5	120.57 (17)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2A \cdots O3	0.85 (1)	1.86 (1)	2.699 (2)	170 (2)
O1—H1A \cdots O3 ⁱⁱⁱ	0.85 (1)	1.87 (1)	2.715 (2)	171 (3)
O1—H1B \cdots N5	0.85 (1)	2.23 (2)	2.926 (2)	139 (2)
O3—H3B \cdots N3 ^{iv}	0.84 (1)	2.01 (1)	2.839 (2)	169 (2)
O3—H3A \cdots N2 ^v	0.85 (1)	2.00 (1)	2.843 (2)	173 (2)
O2—H2B \cdots N3 ^{vi}	0.85 (1)	2.69 (2)	3.439 (2)	148 (2)
O2—H2B \cdots N4 ^{vi}	0.85 (1)	1.99 (1)	2.840 (2)	173 (2)

Symmetry codes: (iii) $x, y-1, z$; (iv) $-x+1, -y+2, -z+1$; (v) $x+1, y, z$; (vi) $x, y, z+1$.

Fig. 1

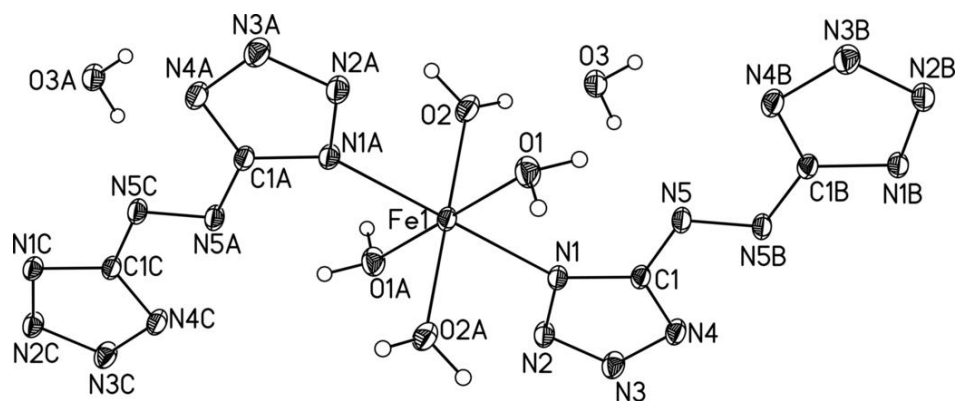


Fig. 2

