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Poly[diaqua-μ_3-malonato-μ-pyrazine-diiron(II)]

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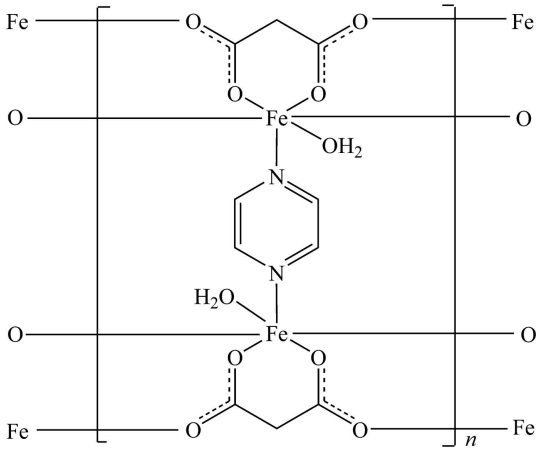
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.040; wR factor = 0.106; data-to-parameter ratio = 11.1.

The title compound, $[Fe_2(C_3H_2O_4)_2(C_4H_4N_2)(H_2O)_2]_n$, prepared by hydrothermal synthesis, is isostructural with its Co^{II}, Ni^{II}, Zn^{II} and Cd^{II} analogues. The Fe^{II} atoms are linked *via* coordinated malonates into two-dimensional sheets containing cavities. The sheets are connected by bridging pyrazine ligands which lie on centres of inversion. The coordination geometry around Fe^{II} is a tetragonally elongated octahedron, in which pyrazine N and aqua O atoms occupy the axial positions. The coordinated water molecules form O—H···O hydrogen bonds to the malonate ligands.

Related literature

For the isostructural analogues, see: Co^{II} (Delgado *et al.*, 2003); Ni^{II} (Liu *et al.*, 2005); Zn^{II} (Zhang *et al.*, 2003; Delgado *et al.*, 2003); Cd^{II} (Mao *et al.*, 2004).



Experimental

Crystal data

$[Fe_2(C_3H_2O_4)_2(C_4H_4N_2)(H_2O)_2]$
M_r = 431.92
Monoclinic, $P2_1/n$
a = 6.9652 (10) Å
b = 14.589 (2) Å
c = 7.3212 (10) Å
β = 92.179 (1)°
V = 743.39 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.01 mm^{-1}
T = 293 (2) K
0.36 × 0.28 × 0.24 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.532, T_{max} = 0.644
2391 measured reflections
1293 independent reflections
1100 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.106
S = 1.00
1293 reflections
116 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H2W···O1^{i}	0.83 (4)	1.93 (3)	2.689 (4)	152 (6)
O5—H2W···O2^{i}	0.83 (4)	2.78 (6)	3.031 (5)	99 (5)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2221).

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supplementary materials

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Poly[diaqua-μ_3-malonato-μ-pyrazine-diiron(II)]

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Comment

The title compound is isostructural with its Co^{II} (Delgado *et al.*, 2003), Ni^{II} (Liu *et al.*, 2005), Zn^{II} (Zhang *et al.*, 2003; Delgado *et al.*, 2003) and Cd^{II} (Mao *et al.*, 2004) analogues. The Fe^{II} atom exhibits sixfold coordination, chelated by two O atoms from one malonate ligand to form a six-membered boat-type ring, and by two O atoms from two neighbouring malonates, one water molecule and one N atom from the bridging pyrazine ligand (Fig. 1). The Fe—*O*(carboxylate) and Fe—N bond lengths are in the range 2.064 (4)–2.202 (4) and 2.272 (4) Å, respectively.

The [Fe(malonate)(H_2O)] units form two-dimensional networks parallel to the (010) planes. These are linked by the bridging pyrazine ligands into a three-dimensional structure. O—H···O hydrogen bonds (Table 1) are formed between the coordinated water molecules and the malonate ligands.

Experimental

A mixture of $FeSO_4$ (0.5 mmol), malonic acid (0.5 mmol), NaOH (1 mmol), pyrazine (1 mmol) and H_2O (8 ml) in a 25 ml Teflon-lined stainless steel autoclave was heated at 443 K for two days, and then cooled to room temperature. Light green block crystals of the title compound were obtained with a yield of 22%. Elemental analysis: calculated C 27.91, H 2.79, N 6.51%; found: C 27.88, H 2.75, N 6.47%.

Refinement

All H atoms on C atoms were generated geometrically and refined as riding atoms with C—H = 0.93 or 0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C). The H atoms of the water molecule were located from difference Fourier maps and were refined with distance restraints of d(H—H) = 1.38 (2) Å, d(O—H) = 0.82 (1) Å.

Figures

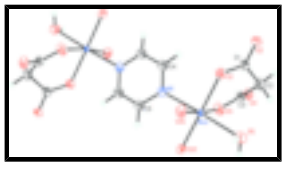

Fig. 1. Part of the polymeric structure of the title complex showing displacement ellipsoids at 30% probability for non-H atoms. Atoms labeled with the suffix I are generated by the symmetry operator (−*x* + 1, *y* + 1/2,-*z* + 3/2).

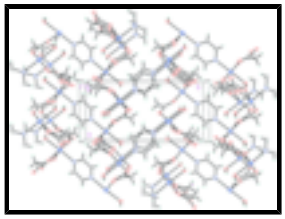

Fig. 2. Packing diagram for the title complex viewed along the *c*-axis.

Poly[diaqua-μ$_3$-malonato-μ-pyrazine-diiron(II)]

Crystal data

$[Fe_2(C_3H_2O_4)_2(C_4H_4N_2)(H_2O)_2]$	F_{000} = 436
M_r = 431.92	D_x = 1.930 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1293 reflections
a = 6.9652 (10) Å	θ = 2.8–25.0º
b = 14.589 (2) Å	μ = 2.01 mm^{-1}
c = 7.3212 (10) Å	T = 293 (2) K
β = 92.179 (1)º	Block, green
V = 743.39 (18) Å^3	0.36 × 0.28 × 0.24 mm
Z = 2	

Data collection

Bruker APEXII CCD diffractometer	1293 independent reflections
Radiation source: fine-focus sealed tube	1100 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −8→3
T_{min} = 0.532, T_{max} = 0.644	k = −13→16
2391 measured reflections	l = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.106	$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 1.1979P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.010
1293 reflections	$\Delta\rho_{max}$ = 0.65 e Å^{-3}
116 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
3 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.039 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5845 (6)	0.3316 (3)	0.7563 (6)	0.0316 (9)
C2	0.6129 (7)	0.3912 (3)	0.5893 (6)	0.0397 (10)
H2A	0.5238	0.4423	0.5912	0.048*
H2B	0.7422	0.4160	0.5955	0.048*
C3	0.5827 (6)	0.3401 (3)	0.4125 (6)	0.0317 (9)
C4	0.4533 (7)	0.0742 (3)	0.5913 (7)	0.0473 (12)
H4	0.4138	0.1251	0.6565	0.057*
C5	0.6788 (6)	−0.0045 (3)	0.4450 (7)	0.0437 (11)
H5	0.8030	−0.0107	0.4042	0.052*
Fe1	0.83758 (7)	0.19136 (3)	0.58103 (6)	0.0177 (3)
N1	0.6323 (5)	0.0716 (2)	0.5376 (5)	0.0357 (8)
O1	0.6634 (4)	0.2528 (2)	0.7697 (4)	0.0397 (8)
O2	0.4864 (4)	0.3672 (2)	0.8748 (4)	0.0403 (8)
O3	0.6700 (5)	0.2644 (2)	0.3900 (4)	0.0429 (8)
O4	0.4752 (4)	0.3774 (2)	0.2946 (4)	0.0393 (7)
O5	1.0315 (5)	0.3106 (2)	0.5861 (4)	0.0439 (8)
H2W	1.090 (8)	0.308 (4)	0.490 (4)	0.080*
H1W	1.101 (7)	0.298 (4)	0.677 (5)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.034 (2)	0.029 (2)	0.032 (2)	0.0001 (18)	0.0005 (16)	−0.0053 (18)
C2	0.056 (3)	0.034 (2)	0.029 (2)	0.005 (2)	0.0022 (19)	0.0010 (19)
C3	0.036 (2)	0.028 (2)	0.032 (2)	0.0035 (18)	0.0042 (17)	0.0045 (18)
C4	0.044 (3)	0.039 (3)	0.060 (3)	−0.005 (2)	0.018 (2)	−0.014 (2)
C5	0.038 (2)	0.034 (3)	0.060 (3)	−0.002 (2)	0.012 (2)	−0.009 (2)
Fe1	0.0219 (3)	0.0169 (4)	0.0145 (3)	0.00068 (19)	0.0018 (2)	−0.00062 (19)
N1	0.0357 (18)	0.033 (2)	0.038 (2)	−0.0020 (16)	0.0017 (15)	−0.0011 (16)
O1	0.0516 (18)	0.0358 (18)	0.0325 (16)	0.0069 (14)	0.0116 (13)	0.0027 (13)
O2	0.0454 (17)	0.0379 (18)	0.0387 (17)	0.0043 (14)	0.0168 (13)	0.0000 (14)
O3	0.054 (2)	0.042 (2)	0.0325 (16)	0.0114 (15)	−0.0052 (14)	−0.0031 (14)

O4	0.0476 (17)	0.0335 (17)	0.0363 (16)	0.0027 (14)	−0.0060 (13)	0.0027 (14)
O5	0.0467 (19)	0.053 (2)	0.0325 (17)	−0.0013 (15)	0.0019 (14)	0.0000 (15)

Geometric parameters (Å, °)

C1—O2	1.239 (5)	C5—C4^{i}	1.390 (7)
C1—O1	1.276 (5)	C5—H5	0.930
C1—C2	1.519 (6)	Fe1—O2^{ii}	2.050 (3)
C2—C3	1.501 (6)	Fe1—O4^{iii}	2.064 (3)
C2—H2A	0.970	Fe1—O1	2.076 (3)
C2—H2B	0.970	Fe1—O3	2.081 (3)
C3—O4	1.246 (5)	Fe1—O5	2.202 (4)
C3—O3	1.275 (6)	Fe1—N1	2.272 (4)
C4—N1	1.322 (6)	O2—Fe1^{iv}	2.050 (3)
C4—C5^{i}	1.390 (7)	O4—Fe1^{v}	2.064 (3)
C4—H4	0.930	O5—H2W	0.83 (4)
C5—N1	1.347 (6)	O5—H1W	0.83 (4)
O2—C1—O1	124.8 (4)	O4^{iii}—Fe1—O3	172.32 (12)
O2—C1—C2	114.6 (4)	O1—Fe1—O3	84.20 (12)
O1—C1—C2	120.6 (4)	O2^{ii}—Fe1—O5	90.84 (12)
C3—C2—C1	113.1 (4)	O4^{iii}—Fe1—O5	95.99 (12)
C3—C2—H2A	109.0	O1—Fe1—O5	91.21 (12)
C1—C2—H2A	109.0	O3—Fe1—O5	86.27 (14)
C3—C2—H2B	109.0	O2^{ii}—Fe1—N1	84.81 (12)
C1—C2—H2B	109.0	O4^{iii}—Fe1—N1	90.23 (13)
H2A—C2—H2B	107.8	O1—Fe1—N1	92.56 (13)
O4—C3—O3	124.5 (4)	O3—Fe1—N1	88.03 (13)
O4—C3—C2	116.2 (4)	O5—Fe1—N1	172.81 (13)
O3—C3—C2	119.3 (4)	C4—N1—C5	114.9 (4)
N1—C4—C5^{i}	123.2 (4)	C4—N1—Fe1	122.1 (3)
N1—C4—H4	118.4	C5—N1—Fe1	122.9 (3)
C5^{i}—C4—H4	118.4	C1—O1—Fe1	126.8 (3)
N1—C5—C4^{i}	122.0 (4)	C1—O2—Fe1^{iv}	130.5 (3)
N1—C5—H5	119.0	C3—O3—Fe1	128.0 (3)
C4^{i}—C5—H5	119.0	C3—O4—Fe1^{v}	124.2 (3)
O2^{ii}—Fe1—O4^{iii}	97.03 (13)	Fe1—O5—H2W	106 (4)
O2^{ii}—Fe1—O1	173.95 (12)	Fe1—O5—H1W	100 (5)
O4^{iii}—Fe1—O1	88.42 (13)	H2W—O5—H1W	113 (3)
O2^{ii}—Fe1—O3	90.26 (13)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O5—H2W···O1^{ii}	0.83 (4)	1.93 (3)	2.689 (4)	152 (6)
O5—H2W···O2^{ii}	0.83 (4)	2.78 (6)	3.031 (5)	99 (5)

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

Fig. 1

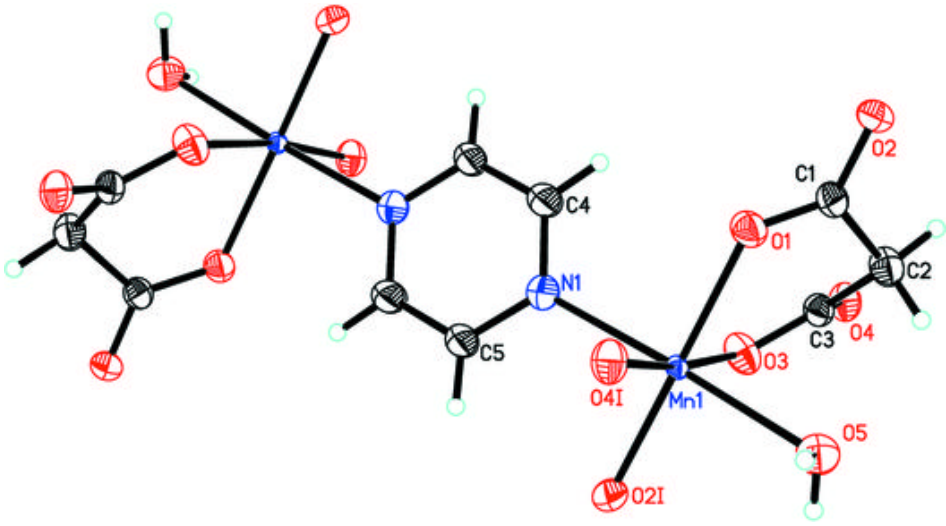

Fig. 2