

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Diaquabis(5-carboxy-1*H*-imidazole-4-carboxylato- κ^2N^3,O^4)iron(II)

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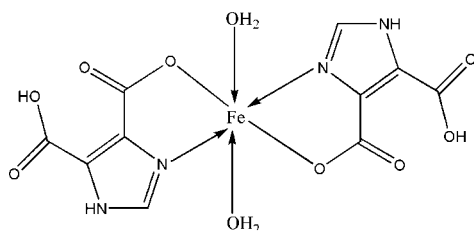
Received 30 May 2011; accepted 23 June 2011

Key indicators: single-crystal X-ray study; $T = 298\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$; R factor = 0.034; wR factor = 0.080; data-to-parameter ratio = 10.7.

In the title compound, $[\text{Fe}(\text{C}_5\text{H}_3\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2]$, the Fe^{II} ion lies on an inversion centre and is coordinated by two N and two O atoms from two 5-carboxy-1*H*-imidazole-4-carboxylate ligands and two water molecules in a distorted octahedral geometry. An intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond occurs. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds form a three-dimensional network, which consolidates the packing.

Related literature

For the diversity of coordination architectures of the metal atom in complexes with 4,5-dicarboxyimidazole, see: Shimizu *et al.* (2004); Fang & Zhang (2006). For the closely related crystal structures of the Zn, Mg and Cd complexes, see: Ma *et al.* (2003), Liu *et al.* (2004) and Zhang *et al.* (2004), respectively.



Experimental

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_3\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2]$
 $M_r = 402.07$
 Monoclinic, $P2_1/c$
 $a = 5.0676(9)\text{ \AA}$
 $b = 22.769(4)\text{ \AA}$
 $c = 6.6725(9)\text{ \AA}$
 $\beta = 113.733(10)^\circ$
 $V = 704.8(2)\text{ \AA}^3$
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.14\text{ mm}^{-1}$
 $T = 298\text{ K}$
 $0.32 \times 0.28 \times 0.25\text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\text{min}} = 0.712$, $T_{\text{max}} = 0.764$
 2872 measured reflections
 1240 independent reflections
 976 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.080$
 $S = 1.05$
 1240 reflections
 116 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25\text{ e \AA}^{-3}$

Table 1

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{O3}^{\text{i}}$	0.86	2.05	2.897 (3)	169
$\text{O3}-\text{H3}\cdots\text{O2}$	0.82	1.74	2.525 (3)	160
$\text{O1W}-\text{H1W}\cdots\text{O2}^{\text{ii}}$	0.85	1.94	2.744 (3)	157
$\text{O1W}-\text{H2W}\cdots\text{O1}^{\text{iii}}$	0.85	1.92	2.710 (3)	155

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5111).

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supporting information

Acta Cryst. (2011). E67, m997 [doi:10.1107/S1600536811024779]

Diaquabis(5-carboxy-1*H*-imidazole-4-carboxylato- κ^2N^3,O^4)iron(II)**Chao-Jun Du, Xing-Hua Song, Li-Sheng Wang and Chao-Ling Du****S1. Comment**

In recent years, the construction of metal complexes based on 1*H*-imidazole-4,5-dicarboxylic acid ligand has been investigated in terms of their intriguing topologies. The diversity of coordination architecture of metal 4,5-dicarboxyimidazole has been described by Shimizu *et al.* (2004) and Fang *et al.* (2006). In order to search for new metal complexes based on 1*H*-imidazole-4,5-dicarboxylic acid ligand, the title complex (I) has been synthesized and its crystal determined.

The crystal structure of (I) is isostructural with the previously reported Zn (Ma *et al.*, 2003), Mg (Zhang *et al.*, 2004) and Cd (Liu *et al.*, 2004) 4,5-dicarboxyimidazole complexes. In the four isostructural complexes, all metal ions lie on an inversion centre being coordinated by two *N,O*-bidentate 1*H*-imidazole-4,5-dicarboxylate monoanionic ligands and two water molecules in a distorted octahedral geometry.

In the crystal structure of (I), intermolecular N—H \cdots O and O—H \cdots O hydrogen bonds (Table 1) form three-dimensional hydrogen-bonding network, which consolidate the crystal packing.

S2. Experimental

A mixture of FeSO₄·7H₂O (0.10 mmol), 1*H*-imidazole-4,5-dicarboxylic acid (0.10 mmol), Et₃N (0.1 ml), EtOH (2 ml) and H₂O (2 ml) was sealed in a 10 ml Teflon-lined stainless-steel reactor and then heated to 393 K for 48 h under autogenous pressure, and then slowly cooled to room temperature at a rate of 5 K/h. Pale-yellow block crystals of the title complex were isolated, washed with distilled water, and dried in air (yield: 48%).

S3. Refinement

H atoms attached to C and N atoms were placed in calculated positions (C—H = 0.93 Å, N—H = 0.86 Å) and refined as riding atoms and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$, respectively. The carboxy and water H atoms were located in a difference map, but placed in idealized positions (O—H = 0.82, 0.85 Å), and refined as riding with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

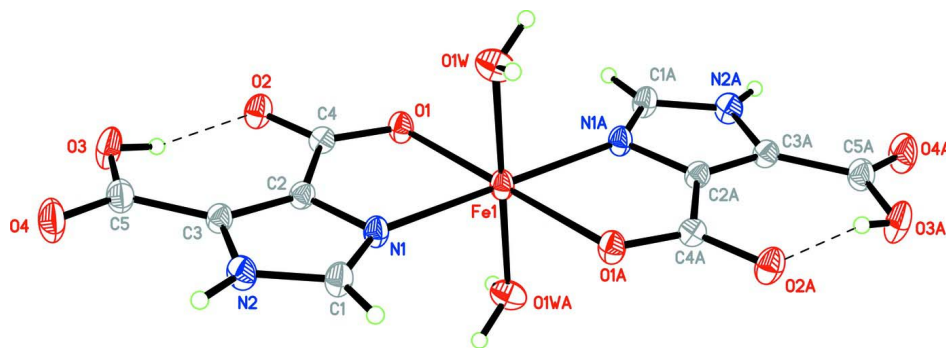


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 40% probability level [symmetry code: (A) $2 - x, 2 - y, 1 - z$].

Diaquabis(5-carboxy-1*H*-imidazole-4-carboxylato- κ^2N^3,O^4)iron(II)

Crystal data

$[\text{Fe}(\text{C}_5\text{H}_3\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2]$

$M_r = 402.07$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 5.0676\ (9)\ \text{\AA}$

$b = 22.769\ (4)\ \text{\AA}$

$c = 6.6725\ (9)\ \text{\AA}$

$\beta = 113.733\ (10)^\circ$

$V = 704.8\ (2)\ \text{\AA}^3$

$Z = 2$

$F(000) = 408$

$D_x = 1.895\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1102 reflections

$\theta = 3.5\text{--}23.7^\circ$

$\mu = 1.14\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, pale-yellow

$0.32 \times 0.28 \times 0.25\ \text{mm}$

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.712, T_{\max} = 0.764$

2872 measured reflections

1240 independent reflections

976 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 25.2^\circ, \theta_{\min} = 3.5^\circ$

$h = -6 \rightarrow 5$

$k = -26 \rightarrow 26$

$l = -4 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.080$

$S = 1.05$

1240 reflections

116 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.0621P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.25\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.25\ \text{e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.2587 (6)	0.86826 (13)	0.4808 (4)	0.0322 (7)
H1	1.4476	0.8718	0.5837	0.039*
C2	0.8201 (6)	0.88774 (11)	0.2661 (4)	0.0265 (6)
C3	0.8639 (6)	0.82984 (12)	0.2373 (4)	0.0279 (6)
C4	0.5611 (5)	0.92523 (12)	0.1742 (4)	0.0268 (6)
C5	0.6789 (7)	0.78182 (14)	0.1066 (5)	0.0369 (7)
Fe1	1.0000	1.0000	0.5000	0.0278 (2)
N1	1.0693 (5)	0.91158 (10)	0.4203 (3)	0.0289 (6)
N2	1.1435 (5)	0.81868 (10)	0.3749 (4)	0.0335 (6)
H2	1.2302	0.7855	0.3906	0.040*
O1	0.5823 (4)	0.97682 (8)	0.2452 (3)	0.0313 (5)
O2	0.3337 (4)	0.90334 (9)	0.0295 (3)	0.0370 (5)
O3	0.4133 (4)	0.79590 (9)	-0.0221 (3)	0.0462 (6)
H3	0.3975	0.8318	-0.0301	0.069*
O4	0.7646 (5)	0.73195 (9)	0.1211 (4)	0.0515 (6)
O1W	0.8685 (4)	0.96305 (9)	0.7355 (3)	0.0392 (5)
H1W	1.0038	0.9508	0.8520	0.059*
H2W	0.7663	0.9851	0.7785	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0267 (15)	0.0259 (17)	0.0363 (16)	0.0013 (13)	0.0045 (12)	-0.0015 (13)
C2	0.0257 (15)	0.0223 (16)	0.0300 (14)	-0.0002 (12)	0.0098 (12)	0.0011 (12)
C3	0.0274 (16)	0.0224 (16)	0.0320 (15)	0.0013 (13)	0.0101 (12)	0.0011 (12)
C4	0.0259 (16)	0.0258 (18)	0.0268 (14)	0.0010 (12)	0.0088 (12)	0.0022 (12)
C5	0.0379 (18)	0.0283 (18)	0.0430 (18)	-0.0011 (15)	0.0144 (14)	-0.0057 (14)
Fe1	0.0267 (3)	0.0203 (3)	0.0314 (3)	0.0024 (3)	0.0067 (2)	-0.0026 (2)
N1	0.0271 (13)	0.0217 (13)	0.0331 (12)	0.0008 (11)	0.0073 (10)	-0.0030 (10)
N2	0.0316 (14)	0.0218 (14)	0.0432 (14)	0.0079 (11)	0.0110 (11)	0.0020 (11)
O1	0.0282 (11)	0.0220 (11)	0.0375 (11)	0.0047 (9)	0.0068 (9)	-0.0019 (9)
O2	0.0277 (11)	0.0317 (12)	0.0402 (11)	0.0007 (10)	0.0018 (9)	0.0000 (9)
O3	0.0367 (12)	0.0286 (13)	0.0570 (14)	-0.0070 (10)	0.0019 (10)	-0.0073 (11)
O4	0.0512 (15)	0.0258 (13)	0.0703 (16)	-0.0008 (11)	0.0169 (12)	-0.0131 (11)
O1W	0.0346 (12)	0.0437 (14)	0.0385 (11)	0.0168 (10)	0.0140 (9)	0.0092 (10)

Geometric parameters (Å, °)

C1—N1	1.321 (3)	C5—O3	1.312 (3)
C1—N2	1.335 (3)	Fe1—O1W ⁱ	2.1128 (19)
C1—H1	0.9300	Fe1—O1W	2.1128 (19)
C2—C3	1.363 (4)	Fe1—N1	2.147 (2)
C2—N1	1.379 (3)	Fe1—N1 ⁱ	2.147 (2)
C2—C4	1.476 (4)	Fe1—O1	2.1801 (18)
C3—N2	1.367 (3)	Fe1—O1 ⁱ	2.1801 (18)
C3—C5	1.475 (4)	N2—H2	0.8600
C4—O1	1.255 (3)	O3—H3	0.8200
C4—O2	1.270 (3)	O1W—H1W	0.8498
C5—O4	1.206 (3)	O1W—H2W	0.8499
N1—C1—N2	111.1 (2)	O1W ⁱ —Fe1—O1	90.82 (7)
N1—C1—H1	124.4	O1W—Fe1—O1	89.18 (7)
N2—C1—H1	124.4	N1—Fe1—O1	77.53 (8)
C3—C2—N1	109.5 (2)	N1 ⁱ —Fe1—O1	102.47 (8)
C3—C2—C4	132.0 (2)	O1W ⁱ —Fe1—O1 ⁱ	89.18 (7)
N1—C2—C4	118.4 (2)	O1W—Fe1—O1 ⁱ	90.82 (7)
C2—C3—N2	105.6 (2)	N1—Fe1—O1 ⁱ	102.47 (8)
C2—C3—C5	134.4 (2)	N1 ⁱ —Fe1—O1 ⁱ	77.53 (7)
N2—C3—C5	120.0 (2)	O1—Fe1—O1 ⁱ	179.999 (1)
O1—C4—O2	124.6 (2)	C1—N1—C2	105.6 (2)
O1—C4—C2	117.2 (2)	C1—N1—Fe1	142.71 (18)
O2—C4—C2	118.2 (2)	C2—N1—Fe1	111.22 (17)
O4—C5—O3	121.6 (3)	C1—N2—C3	108.2 (2)
O4—C5—C3	121.9 (3)	C1—N2—H2	125.9
O3—C5—C3	116.5 (3)	C3—N2—H2	125.9
O1W ⁱ —Fe1—O1W	179.998 (1)	C4—O1—Fe1	115.45 (16)
O1W ⁱ —Fe1—N1	93.27 (8)	C5—O3—H3	109.5
O1W—Fe1—N1	86.73 (8)	Fe1—O1W—H1W	115.5
O1W ⁱ —Fe1—N1 ⁱ	86.73 (8)	Fe1—O1W—H2W	115.7
O1W—Fe1—N1 ⁱ	93.27 (8)	H1W—O1W—H2W	105.2
N1—Fe1—N1 ⁱ	179.999 (1)		
N1—C2—C3—N2	-0.4 (3)	O1W ⁱ —Fe1—N1—C1	96.8 (3)
C4—C2—C3—N2	-176.4 (3)	O1W—Fe1—N1—C1	-83.2 (3)
N1—C2—C3—C5	176.3 (3)	O1—Fe1—N1—C1	-173.1 (3)
C4—C2—C3—C5	0.3 (5)	O1 ⁱ —Fe1—N1—C1	6.9 (3)
C3—C2—C4—O1	174.6 (3)	O1W ⁱ —Fe1—N1—C2	-93.03 (18)
N1—C2—C4—O1	-1.1 (4)	O1W—Fe1—N1—C2	86.97 (18)
C3—C2—C4—O2	-5.5 (4)	O1—Fe1—N1—C2	-2.91 (16)
N1—C2—C4—O2	178.8 (2)	O1 ⁱ —Fe1—N1—C2	177.09 (16)
C2—C3—C5—O4	-174.3 (3)	N1—C1—N2—C3	0.1 (3)
N2—C3—C5—O4	1.9 (5)	C2—C3—N2—C1	0.2 (3)
C2—C3—C5—O3	3.9 (5)	C5—C3—N2—C1	-177.1 (3)
N2—C3—C5—O3	-179.8 (3)	O2—C4—O1—Fe1	178.5 (2)

N2—C1—N1—C2	-0.3 (3)	C2—C4—O1—Fe1	-1.6 (3)
N2—C1—N1—Fe1	170.2 (2)	O1W ⁱ —Fe1—O1—C4	95.72 (19)
C3—C2—N1—C1	0.4 (3)	O1W—Fe1—O1—C4	-84.28 (19)
C4—C2—N1—C1	177.0 (2)	N1—Fe1—O1—C4	2.55 (18)
C3—C2—N1—Fe1	-173.45 (18)	N1 ⁱ —Fe1—O1—C4	-177.45 (18)
C4—C2—N1—Fe1	3.2 (3)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O3 ⁱⁱ	0.86	2.05	2.897 (3)	169
O3—H3 \cdots O2	0.82	1.74	2.525 (3)	160
O1W—H1W \cdots O2 ⁱⁱⁱ	0.85	1.94	2.744 (3)	157
O1W—H2W \cdots O1 ^{iv}	0.85	1.92	2.710 (3)	155

Symmetry codes: (ii) $x+1, -y+3/2, z+1/2$; (iii) $x+1, y, z+1$; (iv) $-x+1, -y+2, -z+1$.