Crystal structure of [diaquabis(4-carboxy-2-ethyl-1*H*-imidazole-5-carboxylato-κ^2N^3,O^4)iron(II)] — *N*,*N*-dimethylformamide (1:2), $Fe(C_7H_7N_2O_4)_2(H_2O)_2 \cdot 2C_3H_7NO$

E-Yu Ji* and Hai-Ling Chen

School of Biological and Chemical Engineering, Nanyang Institute of Technology, Nanyang 473004, P. R. China

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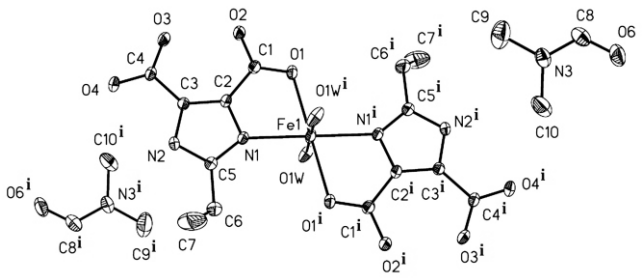

Abstract

$C_{20}H_{32}FeN_6O_{12}$, triclinic, $P\bar{1}$ (no. 2), a = 7.2900(7) Å, b = 8.9289(9) Å, c = 12.0501(1) Å, α = 69.578(1)°, β = 78.015(2)°, γ = 69.990(1)°, V = 687.2 Å^3, Z = 1, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.103, T = 298 K.

Source of material

A mixture of $FeCl_2$ (0.1 mmol, 0.13 g) and 2-ethyl-1*H*-imidazole-4,5-dicarboxylic acid (0.1 mmol, 0.18 g) in 10 ml of DMF solution was sealed in a 15 ml Teflon lined stainless steel container and then heated at 423 K for 2 days. After the mixture was cooled to room temperature, orange block-like crystals were obtained (yield: 36 %).

Experimental details

H atoms of the water molecule were located in a difference Fourier map and refined as riding with a d(O—H) restraint of 0.84(1) Å and U_{iso}(H) = 1.5 U_{eq}. The H—H distances within the water molecules were restrained to 1.39(1) Å (DFIX 1.39 0.001 H1W H2W). Carboxyl H atoms were located in a difference map but were refined as riding on the parent O atoms with d(O—H) = 0.82 Å and U_{iso}(H) = 1.5 U_{eq}(O). Carbon- and nitrogen-bound H atoms were placed at calculated positions and were treated as riding with d(C—H) = 0.96 (methyl), 0.97 (methylene) and d(N—H) = 0.86 Å, U_{iso}(H) = 1.2 or 1.5 U_{eq}(C,N).

Discussion

N,*O*-ligands have been widely used to construct novel supramolecular complexes due to their flexible coordination modes. Several fascinating complexes with imidazole-4,5-dicarboxylate have been isolated [1-3]. Thus, we chose 2-ethyl-1*H*-imidazole-4,5-dicarboxylic acid to obtain a new Fe(II) complex under hydrothermal conditions.

In the asymmetric unit of the title complex , there are one H_2EIDC ligand (H_3EIDC=2-ethyl-1*H*-imidazole-4,5-dicarboxylic acid), half a Fe(II), one coordinated water molecule and one solvent DMF molecule. Each Fe(II) center has slightly distorted octahedral coordination with N_2O_4 donor set formed by two *N*,*O*-chelating rings(O1, O1^i, N1 and N1^i) from two different H_2EIDC ligands and two oxygen atoms (O1W and O1W^i) from two terminal water molecules (symmetry code: i = 1–*x*,1–*y*,1–*z*). The distances within the Fe coordination sphere are d(Fe—O) = 2.091(2) Å and 2.216(2) Å, and d(Fe—N) = 2.168(2) Å. In the crystal packing, the two-dimensional supramolecular network is con-solidated by N–H···O and O–H···O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	orange block, size 0.23 × 0.35 × 0.49 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.18 cm^{-1}
Diffractometer, scan mode:	SMART 1000 CCD, φ/ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3545, 2363
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1967
$N(param)_{refined}$:	181
Programs:	SHELXS-97, SHELXL-97, SHELXTL [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	0.9585	0.1286	0.2464	0.039
H(2A)	2*i*	0.6706	–0.0833	0.6164	0.065
H(1W)	2*i*	0.2211	0.4213	0.4439	0.072
H(2W)	2*i*	0.1670	0.5959	0.4096	0.072
H(6A)	2*i*	0.7851	0.4727	0.1363	0.066
H(6B)	2*i*	0.7571	0.5687	0.2281	0.066
H(7A)	2*i*	1.0866	0.4835	0.2405	0.144
H(7B)	2*i*	1.0511	0.5629	0.1056	0.144
H(7C)	2*i*	1.1221	0.3680	0.1608	0.144
H(8)	2*i*	0.2125	0.8716	1.1715	0.069
H(9A)	2*i*	0.4064	0.6779	1.0850	0.145
H(9B)	2*i*	0.5316	0.7449	0.9668	0.145
H(9C)	2*i*	0.3350	0.7136	0.9617	0.145
H(10A)	2*i*	0.2170	1.0292	0.8384	0.107
H(10B)	2*i*	0.4190	1.0435	0.8523	0.107
H(10C)	2*i*	0.2229	1.1558	0.8999	0.107

* Correspondence author (e-mail: jieyu200899@163.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	1*h*	½	½	½	0.0319(3)	0.0213(3)	0.0384(3)	–0.0037(2)	0.0028(2)	–0.0135(2)
N(1)	2*i*	0.6907(3)	0.3264(2)	0.4064(2)	0.033(1)	0.020(1)	0.032(1)	–0.0047(8)	0.0030(9)	–0.0087(8)
N(2)	2*i*	0.8816(3)	0.1606(2)	0.3037(2)	0.035(1)	0.028(1)	0.029(1)	–0.0037(9)	0.0055(9)	–0.0131(9)
N(3)	2*i*	0.2946(4)	0.9193(3)	1.0035(2)	0.045(1)	0.057(2)	0.046(1)	–0.014(1)	0.008(1)	–0.026(1)
O(1)	2*i*	0.5004(3)	0.2522(2)	0.6291(2)	0.042(1)	0.0273(9)	0.037(1)	–0.0062(8)	0.0099(8)	–0.0146(8)
O(2)	2*i*	0.6025(3)	–0.0196(2)	0.6544(2)	0.059(1)	0.0254(9)	0.038(1)	–0.0120(9)	0.0150(9)	–0.0112(8)
O(3)	2*i*	0.8070(3)	–0.2065(2)	0.5358(2)	0.049(1)	0.0208(9)	0.044(1)	–0.0084(8)	0.0019(9)	–0.0114(8)
O(4)	2*i*	0.9940(3)	–0.1901(2)	0.3624(2)	0.048(1)	0.0277(9)	0.042(1)	0.0012(8)	–0.0015(9)	–0.0181(8)
O(1W)	2*i*	0.2616(3)	0.5064(2)	0.4255(2)	0.041(1)	0.0249(9)	0.079(1)	–0.0036(8)	–0.017(1)	–0.0175(9)
O(6)	2*i*	0.1066(4)	1.1002(3)	1.1026(2)	0.073(2)	0.070(2)	0.056(1)	–0.019(1)	0.020(1)	–0.037(1)
C(1)	2*i*	0.5945(4)	0.1325(3)	0.5917(2)	0.029(1)	0.026(1)	0.034(1)	–0.007(1)	–0.001(1)	–0.009(1)
C(2)	2*i*	0.7009(3)	0.1634(3)	0.4717(2)	0.025(1)	0.022(1)	0.031(1)	–0.0043(9)	–0.002(1)	–0.010(1)
C(3)	2*i*	0.8193(3)	0.0583(3)	0.4083(2)	0.026(1)	0.025(1)	0.028(1)	–0.006(1)	–0.0031(9)	–0.0091(9)
C(4)	2*i*	0.8800(4)	–0.1270(3)	0.4356(2)	0.030(1)	0.024(1)	0.035(1)	–0.002(1)	–0.007(1)	–0.013(1)
C(5)	2*i*	0.8016(4)	0.3204(3)	0.3057(2)	0.038(1)	0.026(1)	0.033(1)	–0.005(1)	0.001(1)	–0.008(1)
C(6)	2*i*	0.8341(5)	0.4685(4)	0.2066(3)	0.068(2)	0.035(2)	0.044(2)	–0.010(2)	0.014(2)	–0.006(1)
C(7)	2*i*	1.0419(7)	0.4709(5)	0.1756(4)	0.095(3)	0.081(3)	0.094(3)	–0.052(3)	–0.003(3)	0.016(2)
C(8)	2*i*	0.2036(5)	0.9577(5)	1.0999(3)	0.056(2)	0.073(2)	0.040(2)	–0.022(2)	0.007(1)	–0.019(2)
C(9)	2*i*	0.4008(7)	0.7497(5)	1.0043(5)	0.083(3)	0.081(3)	0.121(4)	–0.003(2)	0.008(3)	–0.055(3)
C(10)	2*i*	0.2878(6)	1.0479(5)	0.8888(3)	0.083(3)	0.091(3)	0.045(2)	–0.034(2)	0.012(2)	–0.027(2)

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