Crystal structure of aqua(1*H*-imidazol)-(μ_3-5-isonicotinamido-isophthalato-κ^2O^1,O^3-κ^1N^1)iron(II) monohydrate, $Fe(H_2O)(C_3H_4N_2)(C_{14}H_8N_2O_5) \cdot H_2O$

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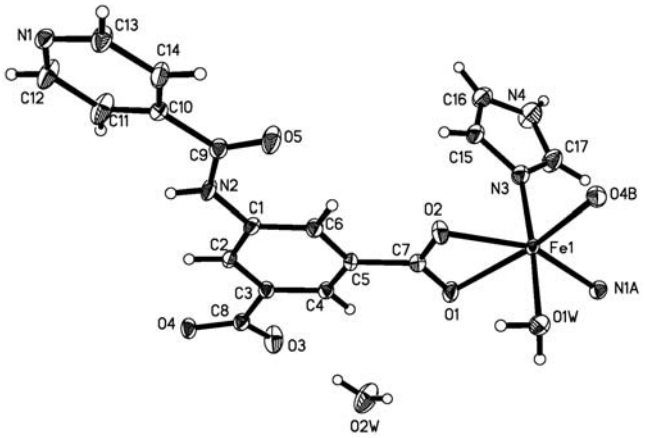

Abstract

$C_{17}H_{16}FeN_4O_7$, monoclinic, $P12_1/c1$ (no. 14), a = 7.696(5) Å, b = 17.96(1) Å, c = 12.778(9) Å, β = 102.001(2)°, V = 1727.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.118, T = 296 K.

Source of material

A mixture of $FeSO_4 \cdot 7H_2O$ (0.028 g, 0.1 mmol), 5-isonicotinamidoisophthalic acid (H_2L, 0.028 g, 0.1 mmol), NaOH (0.081 g, 0.2 mmol), imidazol (Him, 0.007 g, 0.1 mmol), CH_3CH_2OH (4 mL) and H_2O (6 mL) was sealed in a 15 ml Teflon-lined stainless steel reactor, which was heated at 393 K for 72 h and then cooled to room temperature. Red needle-like crystals of the title compound were collected.

Experimental details

H atoms were placed geometrically and treated as riding with d(C—H) = 0.93 Å, d(O—H) = 0.85 Å and U_{iso}(H) = 1.2 U_{eq}(C,O).

Discussion

In recent years, much interest in transition metal complex assembly has been devoted to the development of rational synthetic routes to novel one-, two- and three-dimensional crystal frameworks, due to their potential applications in many areas [1]. Particularly, iron(II) complexes with the heterocycle carboxylate have been investigated rarely to date [2-4]. However, it is well known that carboxylate pyridine as well as the amide groups have good coordination capacity [5-8].

In the title compound, the central Fe(II) ion is six-coordinated by two N atoms from the Him and one L^{2-} ligand, three carboxylate O atoms from two L^{2-} ligands and one O atom from water in a distorted octahedral arrangement. Furthermore, two carboxylate and one pyridine groups of the L^{2-} ligand interconnect three Fe(II) atoms. The carboxylate groups of L^{2-} ligand adopt bidentate chelating and mono-dentate modes, to interlink the title complexes into an infinite layer structure. The layers generate 3D frameworks architecture through O–H···O as well as N–H···O hydrogen bonding interactions.

Table 1. Data collection and handling.

Crystal:	red needle, size 0.10 × 0.16 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.26 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8698, 3113
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2581
$N(param)_{refined}$:	263
Programs:	SHELXS-97, SHELXL-97 [9]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.2742	0.3717	–0.0335	0.031
H(4)	4*e*	0.5779	0.3947	0.2601	0.031
H(6)	4*e*	0.3072	0.5700	0.1134	0.033
H(11)	4*e*	0.0589	0.4916	–0.2800	0.061
H(12)	4*e*	–0.1152	0.5169	–0.4433	0.056
H(13)	4*e*	–0.2521	0.7071	–0.3448	0.049
H(14)	4*e*	–0.0835	0.6873	–0.1782	0.047
H(15)	4*e*	0.7139	0.6503	0.1988	0.040
H(16)	4*e*	1.0089	0.6667	0.1551	0.051
H(17)	4*e*	1.0629	0.6566	0.4692	0.055
H(2)	4*e*	0.1338	0.4719	–0.1216	0.037
H(4A)	4*e*	1.2148	0.6700	0.3262	0.066
H(1WB)	4*e*	0.3367	0.6014	0.4572	0.042
H(1WA)	4*e*	0.4240	0.5809	0.5510	0.042
H(2WB)	4*e*	0.6815	0.2625	0.3333	0.084
H(2WA)	4*e*	0.6788	0.2990	0.4279	0.084

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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)	4*e*	0.63115(5)	0.63585(2)	0.43799(3)	0.0252(3)	0.0192(3)	0.0138(3)	–0.0002(2)	–0.0020(2)	0.0004(1)
C(1)	4*e*	0.2766(4)	0.4761(2)	0.0233(2)	0.028(2)	0.027(2)	0.018(1)	0.002(1)	0.001(1)	0.002(1)
C(2)	4*e*	0.3209(4)	0.4014(2)	0.0252(2)	0.033(2)	0.026(2)	0.018(1)	–0.002(1)	0.001(1)	–0.002(1)
C(3)	4*e*	0.4336(4)	0.3699(2)	0.1130(2)	0.026(2)	0.022(2)	0.024(2)	0.000(1)	0.003(1)	–0.000(1)
C(4)	4*e*	0.4995(4)	0.4145(2)	0.2014(2)	0.029(2)	0.023(2)	0.021(1)	0.000(1)	–0.002(1)	0.002(1)
C(5)	4*e*	0.4485(4)	0.4883(2)	0.2021(2)	0.027(2)	0.026(2)	0.019(1)	–0.003(1)	0.001(1)	–0.003(1)
C(6)	4*e*	0.3387(4)	0.5201(2)	0.1130(2)	0.033(2)	0.022(2)	0.024(2)	0.001(1)	0.001(1)	–0.001(1)
C(7)	4*e*	0.5109(4)	0.5377(2)	0.2971(2)	0.028(2)	0.028(2)	0.024(2)	–0.004(1)	0.004(1)	–0.002(1)
C(8)	4*e*	0.4784(4)	0.2878(2)	0.1131(2)	0.036(2)	0.025(2)	0.024(2)	0.001(1)	0.004(1)	0.000(1)
C(9)	4*e*	0.1111(4)	0.5744(2)	–0.0959(2)	0.030(2)	0.027(2)	0.025(2)	0.002(1)	0.000(1)	0.001(1)
C(10)	4*e*	0.0062(4)	0.5862(2)	–0.2081(2)	0.029(2)	0.028(2)	0.022(2)	–0.001(1)	0.000(1)	0.003(1)
C(11)	4*e*	–0.0045(5)	0.5360(2)	–0.2907(2)	0.070(3)	0.041(2)	0.033(2)	0.030(2)	–0.010(2)	–0.004(2)
C(12)	4*e*	–0.1092(5)	0.5519(2)	–0.3890(2)	0.069(3)	0.041(2)	0.024(2)	0.021(2)	–0.006(2)	–0.007(1)
C(13)	4*e*	–0.1891(5)	0.6628(2)	–0.3314(2)	0.056(2)	0.030(2)	0.030(2)	0.009(2)	–0.009(2)	–0.000(1)
C(14)	4*e*	–0.0881(5)	0.6512(2)	–0.2309(2)	0.055(2)	0.029(2)	0.025(2)	0.004(2)	–0.008(2)	–0.004(1)
C(15)	4*e*	0.8212(4)	0.6533(2)	0.2480(2)	0.033(2)	0.040(2)	0.025(2)	–0.003(1)	0.001(1)	0.000(1)
C(16)	4*e*	0.9841(5)	0.6626(2)	0.2231(3)	0.044(2)	0.053(2)	0.031(2)	–0.007(2)	0.009(2)	0.001(2)
C(17)	4*e*	1.0112(5)	0.6567(2)	0.3966(3)	0.035(2)	0.069(3)	0.030(2)	–0.007(2)	0.001(2)	–0.001(2)
N(1)	4*e*	–0.2016(3)	0.6136(1)	–0.4108(2)	0.033(1)	0.027(1)	0.022(1)	0.003(1)	0.001(1)	0.003(1)
N(2)	4*e*	0.1681(3)	0.5042(2)	–0.0719(2)	0.041(2)	0.029(2)	0.018(1)	0.004(1)	–0.006(1)	–0.001(1)
N(3)	4*e*	0.8412(3)	0.6491(1)	0.3578(2)	0.031(2)	0.036(2)	0.027(1)	–0.003(1)	0.003(1)	–0.001(1)
N(4)	4*e*	1.1017(4)	0.6646(2)	0.3178(2)	0.028(2)	0.092(3)	0.044(2)	–0.011(2)	0.004(1)	–0.003(2)
O(1)	4*e*	0.6102(3)	0.5125(1)	0.3808(2)	0.045(1)	0.032(1)	0.022(1)	–0.002(1)	–0.0062(9)	–0.0002(9)
O(2)	4*e*	0.4654(3)	0.6047(1)	0.2907(2)	0.037(1)	0.027(1)	0.034(1)	0.001(1)	–0.0013(9)	–0.0085(9)
O(3)	4*e*	0.5987(3)	0.2636(1)	0.1848(2)	0.065(2)	0.031(1)	0.048(2)	0.014(1)	–0.019(1)	–0.001(1)
O(4)	4*e*	0.3879(3)	0.2483(1)	0.0399(2)	0.052(1)	0.021(1)	0.030(1)	–0.003(1)	–0.003(1)	–0.0047(9)
O(5)	4*e*	0.1376(3)	0.6263(1)	–0.0324(2)	0.066(2)	0.032(1)	0.030(1)	0.010(1)	–0.014(1)	–0.003(1)
O(1W)	4*e*	0.4100(3)	0.6189(1)	0.5105(2)	0.038(1)	0.037(1)	0.028(1)	–0.002(1)	0.0015(9)	0.0045(9)
O(2W)	4*e*	0.7198(4)	0.2614(2)	0.4006(2)	0.101(2)	0.065(2)	0.043(2)	0.031(2)	0.013(2)	–0.009(1)

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