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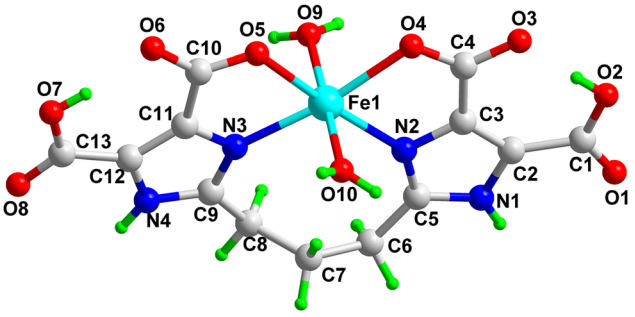
Crystal structure of Diaqua[5,5′-dicarboxy-2,2′-(propane-1,3-diyl)bis(1*H*-imidazole-4-carboxylato-k^4*O*,*O*′,*N*,*N*′)]iron(II), $C_{13}H_{14}FeN_4O_{10}$



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Abstract

$C_{13}H_{14}FeN_4O_{10}$, orthorhombic, *Pcca* (no. 54), a = 13.2154(4) Å, b = 16.9075(6) Å, c = 13.9347(5) Å, V = 3113.56(18) Å^3, Z = 8, $R_{gt}(F)$ = 0.0362, $wR_{ref}(F^2)$ = 0.0971, T = 293(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.13 × 0.10 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.04 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE PHOTON, φ and ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22,485, 3041, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2355
$N(param)_{refined}$:	265
Programs:	SHELX [1], Bruker [2, 3]

Source of material

The reagents and solvents used in this work were purchased from commercial sources and used without further purification. An aqueous solution (2 mL) of $FeSO_4{\cdot}7H_2O$ (0.05 mmol) was added dropwise to an aqueous solution (2 mL) of 2,2′-(propane-1,3-diyl)bis(1*H*-imidazole-4,5-dicarboxylic acid (0.05 mmol, H_6pbidc), resulting in a clear solution. The mixture was then placed in a 25 mL Teflon-lined stainless steel reactor, which was sealed and heated to 413 K for 72 h. After the mixture was cooled to room temperature at a rate of 5 K h^{-1}, yellow crystals of the title compound were obtained (yield 35%, based on Fe).

Experimental details

The propyl group was modelled by splitting it into two parts (C6–C8 and C6′–C8′), the site-occupation factors of which refined in a ratio of 0.688(6): 0.312(6). Hydrogen atoms on carbon and nitrogen atoms were positioned geometrically and refined as riding atoms, with C–H = 0.97 Å and N–H = 0.82 Å. H atoms of the carboxylic acid groups of H_4pbidc^{2-} were refined as riding atoms, with O–H = 0.82 Å. The H atoms of the water molecules were located in a difference Fourier map and the O–H distance was constrained to 0.85 Å. All H atoms were refined with U_{iso}(H) = 1.2 U_{eq}(C, N, O) except for U_{iso}(H) = 1.5 U_{eq}(O) for H atoms of the carboxylic acid.

Comment

The 1*H*-imidazole-4,5-dicarboxylic acid and its 2-substituted analogues have attracted much attention in the construction of coordination compounds (CPs) and have been proven to be excellent building blocks for assembling CPs with

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}^*/U_{eq}
Fe1	0.13185 (3)	0.24519 (2)	0.12381 (2)	0.02320 (13)
N1	0.12635 (14)	0.04766 (11)	−0.08035 (14)	0.0246 (4)
H1A	0.125924	0.022763	−0.134164	0.030*
N2	0.12840 (13)	0.14580 (11)	0.02323 (13)	0.0224 (4)
N3	0.13502 (14)	0.35468 (11)	0.04824 (13)	0.0251 (4)
N4	0.13244 (14)	0.46052 (11)	−0.04216 (14)	0.0271 (4)
H4A	0.133159	0.489933	−0.092505	0.032*
O1	0.12660 (14)	−0.11571 (10)	−0.04958 (14)	0.0400 (5)
O2	0.12081 (14)	−0.09939 (10)	0.10829 (13)	0.0351 (4)
H2	0.122117	−0.061955	0.145676	0.053*
O3	0.12562 (14)	0.01089 (10)	0.22347 (12)	0.0380 (4)
O4	0.13789 (13)	0.14233 (10)	0.21872 (11)	0.0341 (4)
O5	0.12332 (12)	0.33548 (9)	0.23887 (11)	0.0287 (4)
O6	0.11966 (15)	0.46548 (10)	0.26708 (12)	0.0404 (5)
O7	0.10759 (19)	0.58836 (11)	0.16595 (14)	0.0521 (6)
H7	0.107391	0.547489	0.197626	0.078*
O8	0.12452 (14)	0.62210 (10)	0.01443 (14)	0.0412 (5)
O9	0.29190 (14)	0.24693 (9)	0.12551 (12)	0.0339 (4)
H1W	0.327456	0.206409	0.112567	0.041*
H2W	0.323596	0.284379	0.098087	0.041*
O10	−0.02531 (14)	0.24544 (9)	0.12230 (12)	0.0335 (4)
H3W	−0.066486	0.208300	0.109436	0.040*
H4W	−0.065066	0.275069	0.154005	0.040*
C1	0.12416 (16)	−0.07330 (13)	0.02096 (18)	0.0259 (5)
C2	0.12572 (16)	0.01357 (13)	0.00914 (16)	0.0219 (5)
C3	0.12725 (16)	0.07533 (12)	0.07218 (16)	0.0220 (5)
C4	0.13007 (18)	0.07743 (13)	0.17885 (16)	0.0267 (5)
C5	0.12776 (17)	0.12718 (13)	−0.06924 (17)	0.0256 (5)
C6[a]	0.1267 (3)	0.18222 (16)	−0.1535 (2)	0.0481 (8)
H6A[a]	0.088829	0.157377	−0.204926	0.058*
H6B[a]	0.195709	0.188964	−0.175745	0.058*
C7[a]	0.0816 (3)	0.2639 (2)	−0.1350 (3)	0.0371 (11)
H7A[a]	0.036551	0.277150	−0.187580	0.044*
H7B[a]	0.041270	0.261730	−0.076875	0.044*
C8[a]	0.1587 (3)	0.32854 (19)	−0.1247 (2)	0.0608 (10)
H8A[a]	0.225183	0.304877	−0.118251	0.073*
H8B[a]	0.158742	0.360280	−0.182690	0.073*
C6′[b]	0.1267 (3)	0.18222 (16)	−0.1535 (2)	0.0481 (8)
H6′A[b]	0.057813	0.198329	−0.167440	0.058*
H6′B[b]	0.153733	0.155636	−0.209586	0.058*
C7′[b]	0.1945 (6)	0.2586 (4)	−0.1286 (5)	0.024 (2)
H7′A[b]	0.225653	0.248365	−0.066840	0.029*
H7′B[b]	0.248872	0.259761	−0.175338	0.029*
C8′[b]	0.1587 (3)	0.32854 (19)	−0.1247 (2)	0.0608 (10)
H8′A[b]	0.093782	0.325133	−0.156978	0.073*
H8′B[b]	0.201828	0.359292	−0.166870	0.073*
C9	0.14002 (19)	0.38105 (13)	−0.04095 (17)	0.0291 (6)
C10	0.12213 (17)	0.40650 (13)	0.21259 (16)	0.0242 (5)
C11	0.12525 (17)	0.41957 (13)	0.10613 (16)	0.0225 (5)
C12	0.12337 (16)	0.48652 (13)	0.05101 (16)	0.0235 (5)
C13	0.11827 (18)	0.57099 (14)	0.07461 (19)	0.0301 (6)

[a]Occupancy: 0.687(6), [b]Occupancy: 0.313(6).

different structures and useful properties [4–7]. This is because this kind of ligands contain N-atom as well as O-atoms as potential donors, and can be partially or fully deprotonated to generate different anionic ligands at different pH values. Thus, a large number of CPs have been constructed from these kinds of ligands [8–12]. Among them, 2,2′-(propane-1,3-diyl)bis(1*H*-imidazole-4,5-dicarboxylic acid (H_6pbidc) and the corresponding deprotonated anions are excellent ligands [8, 13].

The asymmetric unit of the title structure contains one Fe(II) ion, one 2,2′-(propane-1,3-diyl)bis(1*H*-imidazole-4,5-dicarboxylate (H_4pbidc^{2-}) anion and two water molecules. The Fe1 ion is six-coordinated by two N atoms (N2, N3) and two O atoms (O4, O5) from the tetradentate H_4pbidc^{2-} anion and by two water ligands (O9, O10), featuring a distorted FeN_2O_4 octahedral environment. The Fe–N bond lengths of 2.1301(19) and 2.1887(19) Å and the Fe–O bond lengths of 2.0770(19), 2.1154(19), 2.1864(16) and 2.2166(16) Å are close to those reported in other Fe(II) CPs [14, 15]. The two imidazole rings are nearly co-planar, with a dihedral angle between the two least-square planes N1/C2/C3/N2/C5 and C9/N3/C11/C12/N4 of 6.44(2)°. The dihedral angles between the mean plane defined by the imidazole ring N1/C2/C3/N2/C5 and the planes of the carboxylate groups are 2.26(1)° and 3.99(3)°, respectively. The dihedral angles between the mean plane defined by the imidazole ring C9/N3/C11/C12/N4 and the planes of the carboxylate groups are 2.24(8) and 4.01(6)°, respectively. Intramolecular O–H···O hydrogen bonds between carboxyl and carboxylate groups stabilize the molecular configuration. In addition, there are two kinds of intermolecular N–H···O hydrogen bonds and four kinds of intermolecular O–H···O hydrogen bonds involving imidazole rings, carboxylate groups and coordinated water molecules of adjacent molecules. Adjacent molecules are connected by these hydrogen bonds, resulting in a three-dimensional architecture.

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