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Crystal structure of diaqua-bis(5-carboxy-1-methyl-1H-imidazole-4-carboxylato- $\kappa^2 N,O$) zinc(II), $C_{12}H_{14}N_4O_{10}Zn$

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Abstract

$C_6H_7N_2O_5Zn_{0.5}$, monoclinic, $C2/c$ (no. 15), $a = 9.0277(4)$ Å, $b = 12.0102(5)$ Å, $c = 15.3939(5)$ Å, $\beta = 95.040(3)^\circ$, $V = 1662.62(11)$ Å³, $Z = 8$, $R_{gt}(F) = 0.0242$, $wR_{ref}(F^2) = 0.0637$, $T = 292(1)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were used as obtained without further purification. A mixture of 1-methyl-1H-imidazole-4,5-dicarboxylic acid (H_2mida) (0.1 mmol, 17.1 mg), trz (1,3,5-triazole) (0.1 mmol, 7.1 mg) $Zn(OAc)_2 \cdot 2H_2O$ (0.2 mmol, 43.9 mg), and H_2O (6.0 mL) was placed in 23 mL Teflon-lined autoclave at 393 K for 3 days, and then slowly cooled down. Colourless block-shaped crystals of compound 1 were obtained.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.33 \times 0.29 \times 0.24$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.5 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.0° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15049, 1453, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1415
$N(\text{param})_{\text{refined}}$:	124
Programs:	SHELX [10, 11], Olex2 [12], CrysAlis ^{PRO} [13]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.5000	0.79859(3)	0.7500	0.03064(13)
O1	0.65209(17)	0.66291(13)	0.72669(9)	0.0387(4)
O1W	0.66578(19)	0.91475(14)	0.74617(10)	0.0494(4)
H1WA	0.7274	0.9214	0.7070	0.074*
H1WB	0.6992	0.9671	0.7787	0.074*
O2	0.7364(2)	0.55802(15)	0.62323(11)	0.0536(5)
O3	0.7149(2)	0.55452(16)	0.46304(12)	0.0633(6)
H3	0.7248	0.5621	0.5229	0.095*
O4	0.6100(2)	0.64977(17)	0.35261(10)	0.0542(5)
N1	0.47837(19)	0.78312(14)	0.61238(10)	0.0306(4)
N2	0.4555(2)	0.78542(15)	0.46875(11)	0.0325(4)
C1	0.6575(2)	0.63633(18)	0.64974(14)	0.0336(5)
C2	0.5661(2)	0.70165(16)	0.58274(13)	0.0275(4)
C3	0.4140(2)	0.83111(19)	0.54216(14)	0.0360(5)
H3A	0.3472	0.8900	0.5433	0.043*
C4	0.5534(2)	0.70121(16)	0.49321(13)	0.0286(4)
C5	0.6280(3)	0.63245(19)	0.43004(14)	0.0380(5)
C6	0.4016(3)	0.8228(2)	0.38023(15)	0.0491(6)
H6A	0.4828	0.8539	0.3520	0.074*
H6B	0.3259	0.8783	0.3841	0.074*
H6C	0.3610	0.7605	0.3471	0.074*

Experimental details

Hydrogen atoms bound to carbon atoms were generated geometrically and refined isotropically with a riding model except for those bound to water molecules which were located from the difference Fourier syntheses.

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Comment

The desirable novel supramolecular complexes depending upon hydrogen bonding π - π stacking interactions, and even van der Waals forces have received considerable attention due to the enormous variety of intriguing structural and the potential properties [1–4]. One promising strategy for the preparation of such supramolecular materials is to use multi-dentate carboxylate ligands, which may adopt diverse binding modes [5–7]. A few studies have been carried out by using 1-methyl-1*H*-imidazole-4,5-dicarboxylic acid (H₂mida), which can probably provide the potential to enrich the unpredictable and interesting structural and functional diversities of network structures [8, 9].

Single-crystal X-ray diffraction reveals that title compound is a mononuclear zinc(II) complex with two mida-ligands. The asymmetric unit of title structure contain one half of a zinc(II) ion, one hmdci[−] anion, and one coordination water molecule (see the figure). Four equatorial oxygen atoms from two mida-ligands (O1 and O1A) and two coordination water (O1W and O1WA) respectively are in equatorial position, whereas, two axial positions are occupied by two N atoms (N1 and N1A). The Zn–N bond length is 2.1185(16) Å, and the Zn–O bond lengths are between 2.0507(16) and 2.1808(16) Å. The carboxyl group remains uncoordinated. The carboxylate, the carboxyl group and the anion and imidazolyl ring are almost parallel. There is one intramolecular hydrogen bond (*cf.* the figure). There are intermolecular hydrogen between adjacent complexes: (a) hydrogen bonds (O1W–H1WA...O4¹: 2.748(2) Å and 146.4°: 3/2 – x, 3/2 – y, 1 – z) connect the mononuclear units to form one chain structure; (b) hydrogen bonds (O1W–H1WB...O2²: 2.733(2) Å and 164.5°: 3/2 – x, 1/2 + y, 3/2 – z) link the adjacent chains to form the entire three-dimensional supramolecular network.

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