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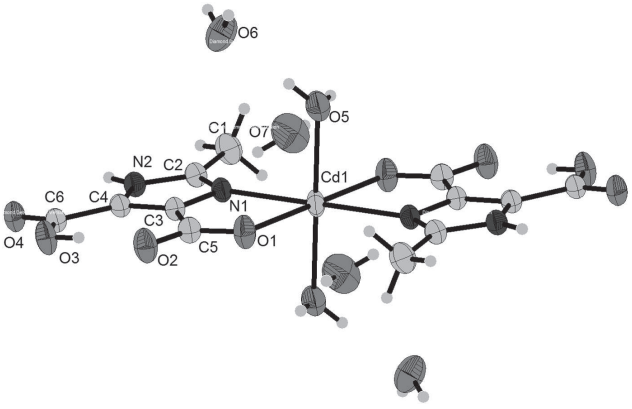
Crystal structure of diaqua-bis(2-methyl-1*H*-imidazole-4,5-dicarboxylato-κ^2-*O*,*N*)cadmium(II) tetrahydrate, $C_{12}H_{22}CdN_4O_{14}$



DOI 10.1515/ncrs-2016-0068
Received March 4, 2016; accepted June 13, 2016; available online June 25, 2016

Abstract

$C_{12}H_{22}CdN_4O_{14}$, triclinic, $P\bar{1}$ (no. 2), $a = 7.188(2)$ Å, $b = 8.895(3)$ Å, $c = 9.771(3)$ Å, $\alpha = 63.148(3)°$, $\beta = 76.750(3)°$, $\gamma = 66.225(3)°$, $V = 509.2(3)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0253$, $wR_{ref}(F^2) = 0.0676$, $T = 296(2)$ K.

CCDC no.: 1484775

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

Source of material

The title compound was synthesized by a hydrothermal method under autogenous pressure. A mixture of $CdCl_2 \cdot H_2O$

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Table 1: Data collection and handling.

Crystal:	Colourless, blocks
	Size 0.22 × 0.20 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.5 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω
$2\theta_{max}$, completeness:	59.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3874, 1879, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1863
$N(param)_{refined}$:	164
Programs:	SHELX [10], Bruker programs [11]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	1.0000	1.0000	0.0000	0.03118(11)
O1	0.9506(3)	1.2382(2)	0.05901(19)	0.0387(4)
O2	0.8494(3)	1.3403(2)	0.2426(2)	0.0408(4)
O4	0.6709(3)	0.9966(3)	0.69875(19)	0.0393(4)
O3	0.7244(3)	1.2393(3)	0.5169(2)	0.0416(4)
H3	0.7573	1.2755	0.4248	0.062*
O5	0.6869(3)	1.1638(4)	−0.1149(3)	0.0604(6)
N1	0.8578(3)	0.9324(2)	0.2449(2)	0.0269(4)
N2	0.7542(3)	0.8366(3)	0.4890(2)	0.0284(4)
C1	0.8039(4)	0.6378(3)	0.3565(3)	0.0393(5)
H1A	0.6999	0.6723	0.2909	0.059*
H1B	0.7753	0.5575	0.4581	0.059*
H1C	0.9337	0.5787	0.3167	0.059*
C2	0.8083(3)	0.7998(3)	0.3627(3)	0.0283(4)
C3	0.8322(3)	1.0586(3)	0.2981(2)	0.0259(4)
C5	0.8804(4)	1.2241(3)	0.1921(3)	0.0304(5)
C4	0.7670(3)	1.0005(3)	0.4507(2)	0.0268(4)
C6	0.7169(3)	1.0797(3)	0.5653(3)	0.0298(5)
O6	0.3235(3)	0.3294(3)	1.0042(2)	0.0526(5)
O7	0.3486(5)	0.4207(4)	0.2413(3)	0.0689(7)
H2	0.716(6)	0.768(5)	0.578(2)	0.083*
H3W	0.251(5)	0.270(4)	1.056(4)	0.083*
H4W	0.263(5)	0.424(3)	0.936(4)	0.083*
H1W	0.692(6)	1.064(3)	−0.100(5)	0.083*
H2W	0.577(4)	1.218(4)	−0.081(5)	0.083*
H5W	0.334(7)	0.521(3)	0.229(4)	0.083*
H6W	0.334(7)	0.411(5)	0.166(3)	0.083*

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(0.029 g, 0.1 mmol), 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid (H_3MIDA) (0.034 g, 0.2 mmol) and distilled water (15 mL) was sealed in a teflon-lined stainless reactor (25 mL) and heated at 120°C for 72 h, and then cooled to room temperature. The red block crystals were filtered and washed with water and ethanol. Yield: 75% (based on H_3MIDA). Analysis calculated for $C_{12}H_{22}CdN_4O_{14}$: C, 25.80; H, 3.97; N, 10.03%; found: C, 24.99; H, 4.19; N, 9.71%.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C—H = 0.96–0.97 Å, O—H = 0.81–0.82 Å and N—H = 0.77 Å, with $U_{iso}(H) = 1.5\,U_{eq}(C)$ for methyl H atoms and 1.2 $U_{eq}(C)$ for all other H atoms.

Discussion

The design and synthesis of metal-organic frameworks (MOFs) have attracted much attention, not only owing to their intriguing variety of architectures but also because of their potential applications as microporous, magnetic, nonlinear optical, and fluorescent materials [1, 2]. Extensive investigation has focused on the construction of coordination polymers using 4,5-imidazoledicarboxylates and derivatives as bridging ligands [3, 4]. However, the reports of coordination polymer with 2-methyl-1H-imidazole-4,5-dicarboxylato ligands are still rare [5–9]. Recently, our group reported five coordination polymers with 2-methyl-1H-imidazole-4,5-dicarboxylate ligand. The reported coordination polymers not only show beautiful and interesting topological structures, for instance 0-D, 1-D, 2-D and 3-D but also exhibits very flexible coordination modes [5–9]. Therefore, we have been engaged in synthesizing new MOFs with H_2MIA-ligand. The structure of the title compound is related to the previously reported cadmium(II) complex [6]. The literature known Cd(II) complex crystallizes in the monoclinic space group $P2_1/c$ and contains no co-crystallized water molecules. The title compound consists of one cadmium(II) ion, two 4-carboxy-2-methyl-1H-imidazole-5-carboxylate anions, two coordinated water molecules, four free water molecules. There are four more free water in the title complex more than the reported cadmium (II) complex. So there are more intermolecular O—H···O hydrogen bonds in the title complex and the intermolecular O—H···O hydrogen bonds link the molecules into a three-dimensional hydrogen bonds network.

Acknowledgements: This work was supported by the Research Foundation of Educational Department of Jiangxi Province[GJJ13261]. X-ray data were collected at Instrumental Analysis Center Nanchang Hangkong University, Nanchang, 330063, People's Republic of China.

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