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Diaquabis(1*H*-imidazole-4-carboxylato-κ^2N^3,*O*)zinc

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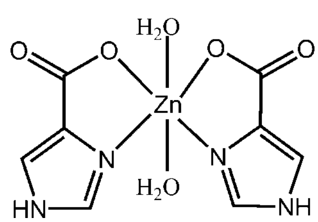
Key indicators: single-crystal X-ray study; *T* = 298 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.023; *wR* factor = 0.066; data-to-parameter ratio = 10.9.

In the title compound, $[Zn(C_4H_3N_2O_2)_2(H_2O)_2]$, the Zn^{II} ion is situated on a twofold rotation axis and exhibits a distorted octahedral coordination configuration. The equatorial plane contains two *cis*-oriented bidentate 1*H*-imidazole-4-carboxylate ligands and the axial positions are occupied by two coordinated water molecules. In the crystal structure, intermolecular O—H···O and N—H···O hydrogen bonds link the molecules into a three-dimensional supramolecular network. There are π–π interactions between the imidazole rings, with a centroid-to-centroid distance of 3.504 (3) Å.

Related literature

For general background, see: Yin *et al.* (2009); Zheng *et al.* (2011); Alkordi *et al.* (2009); Lu *et al.* (2009). For related structures, see: Haggag (2005); Starosta & Leciejewicz (2006); Gryz *et al.* (2007); Yin *et al.* (2009).



Experimental

Crystal data

$[Zn(C_4H_3N_2O_2)_2(H_2O)_2]$
M_r = 323.57
Orthorhombic, *Pccn*
a = 7.1399 (19) Å
b = 11.757 (3) Å
c = 13.508 (4) Å
V = 1133.9 (5) Å^3
Z = 4
Mo *K*α radiation
μ = 2.20 mm^{-1}
T = 298 K
0.35 × 0.32 × 0.30 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.513, T_{max} = 0.558
5336 measured reflections
1037 independent reflections
913 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.066
S = 1.08
1037 reflections
95 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	1.93	2.784 (2)	174
O1*W*—H1*WA*···O2^{ii}	0.83 (2)	2.04 (2)	2.850 (2)	167 (3)
O1*W*—H1*WB*···O2^{iii}	0.82 (2)	1.96 (2)	2.778 (2)	175 (3)

Symmetry codes: (i) $-x+\frac{3}{2}, y, z+\frac{1}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, -y+\frac{3}{2}, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5098).

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supplementary materials

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Diaquabis(1*H*-imidazole-4-carboxylato-κ^2N^3,*O*)zinc

W. Shuai, S. Cai and S. Zheng

Comment

Recently, we were interested in constructing coordination polymers based on *N*-heterocyclic carboxylic acids (Zheng *et al.*, 2011). The imidazole-4-carboxylatic acid (H_2imc), which contains two N atoms of an imidazole group and one carboxylate group, remains largely unexplored, compared with its analogue imidazole-4,5-dicarboxylic acid (Alkordi *et al.*, 2009; Lu *et al.*, 2009). To date, only a few mononuclear complexes based on the H_2imc ligand have been documented (Haggag, 2005; Starosta & Leciejewicz, 2006; Gryz *et al.*, 2007; Yin *et al.*, 2009). For instance, Yin *et al.* (2009) reported the structure of a mononuclear complex [Cd(Himc)$_2$(H_2O)$_2$], which was prepared by the solvent evaporation method. Herein, we report a new Zn^{II} coordination polymer [Zn(Himc)$_2$(H_2O)$_2$], (**I**), which is isomorphous with the Cd^{II} analog.

The asymmetric unit of (**I**) contains a half of [Zn(Himc)$_2$(H_2O)$_2$] formula unit. The Zn^{II} ion exhibits a distorted octahedral geometry (Fig. 1), in which two *cis*-oriented bidentate chelating $Himc^-$ ligands are located in the equatorial plane, forming two stable five-membered rings with metal ion, and the axial sites are occupied by two coordinated water molecules (Fig. 1). The Zn—O distances range from 2.1623 (17) to 2.1626 (14) Å and Zn—N bonds have the value of 2.0751 (16) Å. All Zn—O and Zn—N bond distances are shorter than those of Cd^{II} analog [the axial Cd—O, the equatorial Cd—O and Cd—N bond distances are 2.343 (2), 2.325 (2) and 2.274 (2) Å, respectively].

In the crystal structure, intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) link the molecules into a three-dimensional supramolecular network, which demonstrate π–π interactions between the imidazole rings (Fig. 2) with the centroid-to-centroid distance of 3.504 (3) Å.

Experimental

13.6 mg $ZnCl_2$ (0.10 mmol) and 16.8 mg H_2imc (0.20 mmol) were mixed in 6 ml EtOH/H_2O (1:1). The aqueous NaOH (0.20 *M*) solution was dropwise added to the above solution and the pH was adjusted to about 7. Then, the resulting mixture was sealed into a 10 ml Teflon-lined stainless-steel reactor, which was heated at 100°C for 48 h under autogenous pressure, and then slowly cooled to room temperature at a rate of 2°C/h. Colorless block crystals of **(I)** were isolated, washed with distilled water, and dried in air (yield: 56%). IR (KBr, n/cm^{-1}): 3382*m*, 3147 s, 2997w, 2941w, 2849w, 1688*m*, 1584 s, 15581*m*, 1462*m*, 1402w, 1395 s, 1237 s, 1094*m*, 1003 s, 931w, 847w, 820w, 793*m*, 713w, 656*m*, 610w, 494*m*.

Refinement

C- and N-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 and N—H = 0.86 Å and with U_{iso}(H) = 1.2U_{eq}(C,N). H atoms of the water molecule were located from a difference Fourier map and isotropically refined with O—H bond lenghts restrained to 0.82 (2) Å.

Figures

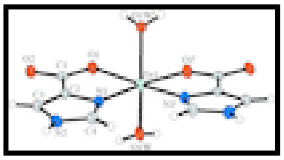

Fig. 1. The molecular structure of (**I**), with displacement ellipsoids drawn at the 30% probability level [symmetry code: (i) $-x + 5/2$, $-y + 3/2$, z.]

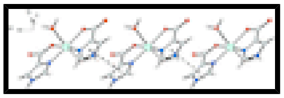

Fig. 2. A portion of the crystal packing showing π–π interactions between the imidazole rings as dashed lines.

Diaquabis(1*H*-imidazole-4-carboxylato-κ^2*N*^{3},*O*)zinc

Crystal data

$[Zn(C_4H_3N_2O_2)_2(H_2O)_2]$

$M_r = 323.57$

Orthorhombic, *Pccn*

$a = 7.1399 (19)$ Å

$b = 11.757 (3)$ Å

$c = 13.508 (4)$ Å

$V = 1133.9 (5)$ Å^3

$Z = 4$

$F(000) = 656$

$D_x = 1.895$ Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2272 reflections

θ = 3.3–27.3°

μ = 2.20 mm^{-1}

$T = 298$ K

Block, colourless

0.35 × 0.32 × 0.30 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)

$T_{min} = 0.513$, $T_{max} = 0.558$

5336 measured reflections

1037 independent reflections

913 reflections with $I > 2\sigma(I)$

$R_{int} = 0.021$

$\theta_{max} = 25.2°$, $\theta_{min} = 3.0°$

$h = -7 \rightarrow 8$

$k = -8 \rightarrow 14$

$l = -14 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.066$

$S = 1.08$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 0.618P]$

where $P = (F_o^2 + 2F_c^2)/3$

1037 reflections	$(\Delta/\sigma)_{max} < 0.001$
95 parameters	$\Delta\rho_{max} = 0.29$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Zn1	1.2500	0.7500	0.37215 (2)	0.02665 (15)
N1	1.0565 (2)	0.81059 (14)	0.47393 (11)	0.0263 (4)
N2	0.8608 (2)	0.87069 (15)	0.58685 (12)	0.0318 (4)
H2	0.8123	0.8836	0.6440	0.038*
C2	0.9100 (3)	0.86538 (16)	0.42860 (13)	0.0229 (4)
C1	0.9084 (3)	0.87026 (16)	0.31884 (14)	0.0229 (4)
C3	0.7885 (3)	0.90319 (19)	0.49823 (15)	0.0291 (4)
H3	0.6781	0.9433	0.4875	0.035*
C4	1.0207 (3)	0.81514 (18)	0.56937 (14)	0.0322 (5)
H4	1.0966	0.7838	0.6182	0.039*
O1	1.04760 (19)	0.82909 (12)	0.27495 (9)	0.0304 (3)
O2	0.76821 (19)	0.91520 (13)	0.27745 (10)	0.0303 (4)
O1W	1.0979 (2)	0.59370 (14)	0.34656 (12)	0.0356 (4)
H1WA	1.150 (4)	0.550 (2)	0.3077 (18)	0.062 (9)*
H1WB	0.989 (3)	0.595 (3)	0.328 (2)	0.072 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0242 (2)	0.0358 (2)	0.0200 (2)	0.00824 (13)	0.000	0.000
N1	0.0267 (9)	0.0336 (10)	0.0187 (8)	0.0059 (7)	0.0010 (7)	0.0021 (7)
N2	0.0338 (10)	0.0426 (11)	0.0189 (8)	0.0024 (8)	0.0070 (7)	−0.0012 (7)
C2	0.0220 (10)	0.0252 (10)	0.0214 (10)	0.0008 (8)	0.0003 (8)	0.0002 (8)
C1	0.0246 (10)	0.0238 (10)	0.0205 (9)	−0.0028 (8)	−0.0016 (8)	0.0009 (8)
C3	0.0254 (10)	0.0344 (11)	0.0275 (11)	0.0040 (9)	0.0007 (9)	−0.0018 (9)
C4	0.0344 (12)	0.0421 (13)	0.0203 (10)	0.0044 (9)	−0.0009 (9)	0.0046 (9)
O1	0.0283 (8)	0.0434 (9)	0.0194 (7)	0.0063 (6)	0.0021 (6)	−0.0011 (6)
O2	0.0249 (8)	0.0433 (9)	0.0227 (7)	0.0044 (6)	−0.0045 (6)	0.0051 (6)
O1W	0.0264 (8)	0.0386 (9)	0.0417 (9)	0.0049 (7)	−0.0033 (7)	−0.0108 (7)

Geometric parameters (Å, °)

Zn1—N1	2.0751 (16)	N2—H2	0.8600
Zn1—N1^{i}	2.0751 (16)	C2—C3	1.354 (3)
Zn1—O1	2.1626 (14)	C2—C1	1.484 (3)
Zn1—O1^{i}	2.1626 (14)	C1—O1	1.255 (2)
Zn1—O1W^{i}	2.1623 (17)	C1—O2	1.262 (2)
Zn1—O1W	2.1623 (17)	C3—H3	0.9300
N1—C4	1.315 (2)	C4—H4	0.9300
N1—C2	1.373 (2)	O1W—H1WA	0.828 (17)
N2—C4	1.336 (3)	O1W—H1WB	0.818 (18)
N2—C3	1.358 (3)		
N1—Zn1—N1^{i}	97.01 (9)	C4—N2—H2	126.1
N1—Zn1—O1	79.04 (6)	C3—N2—H2	126.1
N1^{i}—Zn1—O1	174.10 (5)	C3—C2—N1	109.40 (16)
N1—Zn1—O1^{i}	174.10 (5)	C3—C2—C1	132.55 (17)
N1^{i}—Zn1—O1^{i}	79.04 (6)	N1—C2—C1	118.02 (15)
O1—Zn1—O1^{i}	105.24 (7)	O1—C1—O2	125.47 (17)
N1—Zn1—O1W^{i}	98.53 (7)	O1—C1—C2	116.81 (15)
N1^{i}—Zn1—O1W^{i}	93.64 (7)	O2—C1—C2	117.71 (16)
O1—Zn1—O1W^{i}	82.71 (6)	C2—C3—N2	106.05 (18)
O1^{i}—Zn1—O1W^{i}	86.15 (6)	C2—C3—H3	127.0
N1—Zn1—O1W	93.64 (7)	N2—C3—H3	127.0
N1^{i}—Zn1—O1W	98.54 (7)	N1—C4—N2	111.05 (18)
O1—Zn1—O1W	86.15 (6)	N1—C4—H4	124.5
O1^{i}—Zn1—O1W	82.71 (6)	N2—C4—H4	124.5
O1W^{i}—Zn1—O1W	161.60 (9)	C1—O1—Zn1	114.11 (11)
C4—N1—C2	105.65 (16)	Zn1—O1W—H1WA	114 (2)
C4—N1—Zn1	142.45 (15)	Zn1—O1W—H1WB	121 (2)
C2—N1—Zn1	111.89 (12)	H1WA—O1W—H1WB	104 (3)
C4—N2—C3	107.84 (17)		
N1^{i}—Zn1—N1—C4	−5.0 (2)	C3—C2—C1—O2	−1.8 (3)
O1—Zn1—N1—C4	179.4 (3)	N1—C2—C1—O2	176.18 (18)
O1^{i}—Zn1—N1—C4	42.6 (7)	N1—C2—C3—N2	−0.3 (2)
O1W^{i}—Zn1—N1—C4	−99.8 (2)	C1—C2—C3—N2	177.80 (19)
O1W—Zn1—N1—C4	94.1 (3)	C4—N2—C3—C2	0.0 (2)
N1^{i}—Zn1—N1—C2	175.82 (16)	C2—N1—C4—N2	−0.6 (2)
O1—Zn1—N1—C2	0.25 (13)	Zn1—N1—C4—N2	−179.84 (17)
O1^{i}—Zn1—N1—C2	−136.6 (5)	C3—N2—C4—N1	0.4 (3)
O1W^{i}—Zn1—N1—C2	81.05 (14)	O2—C1—O1—Zn1	−176.15 (15)
O1W—Zn1—N1—C2	−85.11 (14)	C2—C1—O1—Zn1	3.9 (2)
C4—N1—C2—C3	0.6 (2)	N1—Zn1—O1—C1	−2.39 (14)
Zn1—N1—C2—C3	−179.92 (14)	N1^{i}—Zn1—O1—C1	−50.7 (6)

C4—N1—C2—C1	−177.86 (17)	O1^{i}—Zn1—O1—C1	173.43 (15)
Zn1—N1—C2—C1	1.6 (2)	O1W^{i}—Zn1—O1—C1	−102.60 (14)
C3—C2—C1—O1	178.1 (2)	O1W—Zn1—O1—C1	92.07 (14)
N1—C2—C1—O1	−3.9 (2)		

Symmetry codes: (i) −*x*+5/2, −*y*+3/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{ii}	0.86	1.93	2.784 (2)	174
O1W—H1WA···O2^{iii}	0.83 (2)	2.04 (2)	2.850 (2)	167 (3)
O1W—H1WB···O2^{iv}	0.82 (2)	1.96 (2)	2.778 (2)	175 (3)

Symmetry codes: (ii) −*x*+3/2, *y*, *z*+1/2; (iii) −*x*+2, *y*−1/2, −*z*+1/2; (iv) −*x*+3/2, −*y*+3/2, *z*.

Fig. 1

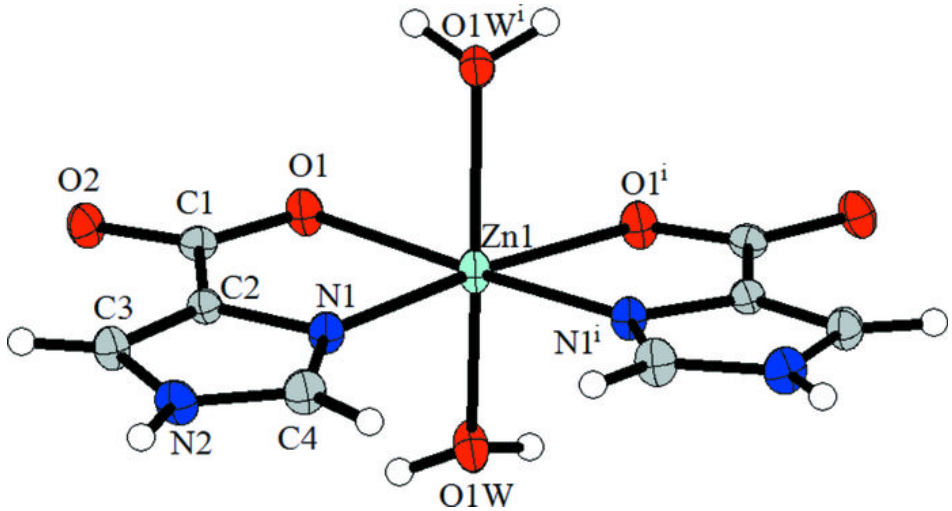

Fig. 2