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Diaquabis(3-carboxypyridine-2-carboxylato-κ^2N,O^2)zinc(II)

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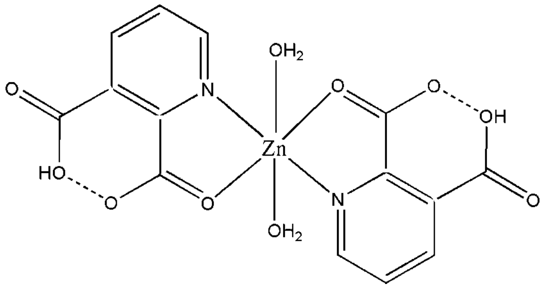
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.069; data-to-parameter ratio = 13.8.

The centrosymmetric title compound, $[Zn(C_{14}H_8N_2O_8)_2(H_2O)_2]$, shows a distorted octahedral coordination. The four donor atoms of the two coplanar 3-carboxypyridine-2-carboxylate or (2,3-pydcH)$^-$ anions form a square-planar arrangement around the Zn^{II} centre. This crystal structure has an infinite three-dimensional framework. The stabilizing interactions existing in the crystal structure are intermolecular O—H···O and very strong intramolecular O—H···O hydrogen bonds. H atoms of coordinated water molecules participate in hydrogen-bonded chains described by $C_2^2(12)$ and $C_1^1(8)$ graph-set descriptors.

Related literature

Several complexes of zinc with pyridine-2,6-dicarboxylic acid have been reported (Aghajani *et al.*, 2006, and references therein). There is also a report of a similar copper compound with pyridine-2,3-dicarboxylic acid (Xiang *et al.*, 2006).



Experimental

Crystal data

$[Zn(C_{14}H_8N_2O_8)(H_2O)_2]$
M_r = 433.63
Monoclinic, $P2_1/n$
a = 9.304 (3) Å
b = 7.909 (3) Å
c = 10.276 (4) Å
β = 95.317 (6)°
V = 752.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.70 mm^{-1}
T = 150 (2) K
0.38 × 0.38 × 0.35 mm

Data collection

Bruker SMART diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
T_{min} = 0.565, T_{max} = 0.588 (expected range = 0.530–0.552)
8225 measured reflections
1706 independent reflections
1575 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.12
1706 reflections
124 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	2.0602 (12)	Zn1—O5	2.1858 (13)
Zn1—N1	2.0691 (14)		
O1—Zn1—N1^i	101.61 (6)	O1—Zn1—N1	78.39 (6)

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O3^{ii}	0.95	1.88	2.8035 (19)	164
O5—H5B···O3^{iii}	0.95	1.89	2.8358 (18)	174
O5—H5B···O4^{iii}	0.95	2.64	3.226 (2)	121
O4—H4A···O2	0.86	1.55	2.4017 (17)	173

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: BQ2016).

References

Aghajani, Z., Sharif, M. A., Aghabozorg, H. & Naderpour, A. (2006). *Acta Cryst.* E**62**, m830–m832.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* (Version 5.059) and *SAINT-Plus* (Version 6.01). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1998). *SADABS*. Version 2.01. University of Göttingen, Germany.
Xiang, J.-F., Li, M., Wu, S.-M., Yuan, L.-J. & Sun, J.-T. (2006). *Acta Cryst.* E**62**, m1122–m1123.

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supplementary materials

Acta Cryst. (2007). E**63**, m1769 [**doi:10.1107/S1600536807025032**]

Diaquabis(3-carboxypyridine-2-carboxylato-$\kappa^2 N,O^2$)zinc(II)

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Comment

The lattice consists of [Zn(2,3-pydcH)$_2$(H$_2$O)$_2$] complexes resulted by a doubly monoprotonated (2,3-pydcH)$^-$ as a bidentate ligand. The presence of this bidentate ligand leads to a neutral complex and does not allow the 2,9-dimethyl-1,10-phenanthroline Lewis base to crystallize in the produced network. The asymmetric unit of compound, [Zn(2,3-pydcH)$_2$(H$_2$O)$_2$] is presented in Figure 1. The metal center is hexacoordinated by two (2,3-pydcH)$^-$ bidentate ligand and two water molecules. The geometry around Zn^{II} center is distorted octahedral. The four donor atoms of the two (2,3-pydcH)$^-$ anions form a square planar arrangement around Zn^{II} center. The rings are almost coplanar (RMS deviation 0.0057 Å). In construction of this crystal which has an infinite three dimensional framework based on [100], [001] and [010] vectors, the hydrogen bonding intermolecular interactions play the essential role. The stabilizing interactions existing in the crystal lattice are intermolecular O–H···O and very strong intramolecular O–H···O type hydrogen bonds.

Hydrogen atoms of coordinated water molecules participate in hydrogen bonded chains described by $C^2_2(12)$ and $C^1_1(8)$ graph set descriptors. Thus, the three-dimensional supramolecule structure for [Zn(2,3-pydcH)$_2$(H$_2$O)$_2$] is confirmed.

Experimental

To a 10 ml of a stirring aqueous solution of 2,9-dimethyl-1,10-phenathroline (0.104 g, 0.5 mmol) and pyridine-2,3-dicarboxylic acid (0.0836 g, 0.5 mmol), was added a 0.5 molar equivalent of ZnSO$_4$. 7H$_2$O (0.0727 g, 0.25 mmol) at room temperature. A neutral zinc(II) complex, [Zn(2,3-pydcH)$_2$(H$_2$O)$_2$], was isolated at pH 3.0 as a colorless crystals. Slow evaporation of the solvent during 10 days resulted in product complexes. Recrystallization of resultants for several times leads to X-ray quality crystals.

Figures

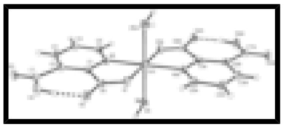

Fig. 1. The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bonds are shown by dashed lines.

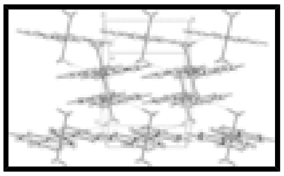

Fig. 2. Packing diagram of (I) with H-bonds with dashed lines. H atoms not involved in H-bonds omitter for clarity.

Diaquabis(3-carboxypyridine-2-carboxylato-κ^2N,O^2)zinc(II)

Crystal data

$[Zn(C_{14}H_8N_2O_8)(H_2O_1)_2]$

$M_r = 433.63$

Monoclinic, $P2_1/n$

$a = 9.304$ (3) Å

$b = 7.909$ (3) Å

$c = 10.276$ (4) Å

$\beta = 95.317$ (6)°

$V = 752.9$ (5) Å^3

$Z = 2$

$F_{000} = 440$

$D_x = 1.913$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 5661 reflections

$\theta = 2.8–27.5°$

$\mu = 1.70$ mm^{-1}

$T = 150$ (2) K

Block, colourless

0.38 × 0.38 × 0.35 mm

Data collection

Bruker SMART
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 100 pixels mm^{-1}

$T = 150(2)$ K

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1998)

$T_{min} = 0.565$, $T_{max} = 0.588$

8225 measured reflections

1706 independent reflections

1575 reflections with $I > 2\sigma(I)$

$R_{int} = 0.023$

$\theta_{max} = 27.5°$

$\theta_{min} = 2.8°$

$h = -11 \rightarrow 11$

$k = -10 \rightarrow 10$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.069$

$S = 1.12$

1706 reflections

124 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.3534P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.40$ e Å^{-3}

$\Delta\rho_{min} = -0.40$ e Å^{-3}

Extinction correction: none

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	0.5000	0.5000	1.0000	0.01651 (11)
N1	0.66631 (13)	0.66168 (17)	0.96316 (12)	0.0159 (3)
C1	0.77906 (16)	0.59130 (19)	0.90988 (14)	0.0147 (3)
C2	0.89688 (16)	0.6908 (2)	0.88011 (14)	0.0156 (3)
C3	0.88952 (17)	0.8644 (2)	0.90586 (15)	0.0189 (3)
H3	0.9665	0.9356	0.8855	0.023*
C4	0.77323 (18)	0.9340 (2)	0.96005 (16)	0.0206 (3)
H4	0.7688	1.0519	0.9771	0.025*
C5	0.66301 (17)	0.8268 (2)	0.98885 (15)	0.0187 (3)
H5	0.5828	0.8721	1.0280	0.022*
C6	0.75688 (16)	0.39930 (19)	0.89076 (14)	0.0158 (3)
C7	1.03467 (16)	0.6349 (2)	0.82239 (15)	0.0179 (3)
O1	0.63949 (11)	0.33806 (14)	0.91765 (11)	0.0181 (2)
O2	0.85601 (12)	0.31061 (14)	0.85009 (11)	0.0203 (2)
O3	1.13066 (12)	0.74039 (15)	0.81237 (12)	0.0218 (3)
O4	1.05136 (14)	0.48120 (15)	0.78668 (14)	0.0261 (3)
H4A	0.9861	0.4162	0.8134	0.031*
O5	0.60695 (12)	0.44082 (15)	1.19303 (11)	0.0201 (2)
H5A	0.6325	0.5429	1.2381	0.024*
H5B	0.6920	0.3741	1.1955	0.024*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01293 (15)	0.01436 (16)	0.02298 (16)	−0.00098 (8)	0.00569 (10)	−0.00054 (9)
N1	0.0137 (6)	0.0160 (6)	0.0182 (6)	0.0004 (5)	0.0023 (5)	0.0006 (5)
C1	0.0146 (7)	0.0143 (8)	0.0150 (7)	0.0005 (5)	0.0007 (5)	0.0006 (5)
C2	0.0137 (7)	0.0184 (8)	0.0148 (7)	0.0000 (6)	0.0017 (5)	0.0008 (6)
C3	0.0172 (7)	0.0186 (8)	0.0208 (7)	−0.0038 (6)	0.0022 (6)	0.0011 (6)
C4	0.0224 (8)	0.0144 (8)	0.0251 (8)	−0.0011 (6)	0.0027 (6)	−0.0009 (6)
C5	0.0173 (7)	0.0169 (8)	0.0224 (7)	0.0019 (6)	0.0045 (6)	−0.0002 (6)
C6	0.0163 (7)	0.0151 (8)	0.0159 (7)	−0.0005 (6)	0.0017 (6)	0.0010 (5)

C7	0.0149 (7)	0.0211 (8)	0.0179 (7)	0.0003 (6)	0.0021 (6)	0.0031 (6)
O1	0.0151 (5)	0.0158 (5)	0.0243 (5)	−0.0010 (4)	0.0054 (4)	−0.0009 (4)
O2	0.0171 (5)	0.0159 (5)	0.0290 (6)	0.0014 (4)	0.0073 (4)	−0.0010 (5)
O3	0.0149 (5)	0.0225 (6)	0.0286 (6)	−0.0013 (4)	0.0047 (4)	0.0033 (5)
O4	0.0192 (6)	0.0209 (6)	0.0405 (8)	−0.0022 (4)	0.0140 (5)	−0.0025 (5)
O5	0.0167 (5)	0.0196 (6)	0.0243 (6)	0.0018 (4)	0.0030 (4)	−0.0011 (5)

Geometric parameters (Å, °)

Zn1—O1	2.0602 (12)	C3—C4	1.377 (2)
Zn1—O1^{i}	2.0602 (12)	C3—H3	0.9500
Zn1—N1^{i}	2.0691 (14)	C4—C5	1.384 (2)
Zn1—N1	2.0691 (14)	C4—H4	0.9500
Zn1—O5	2.1858 (13)	C5—H5	0.9500
Zn1—O5^{i}	2.1858 (13)	C6—O1	1.2489 (19)
N1—C5	1.334 (2)	C6—O2	1.2604 (19)
N1—C1	1.3478 (19)	C7—O3	1.2333 (19)
C1—C2	1.406 (2)	C7—O4	1.283 (2)
C1—C6	1.543 (2)	O4—H4A	0.8600
C2—C3	1.401 (2)	O5—H5A	0.9500
C2—C7	1.527 (2)	O5—H5B	0.9500
O1—Zn1—O1^{i}	180.0	C3—C2—C7	114.50 (13)
O1—Zn1—N1^{i}	101.61 (6)	C1—C2—C7	128.45 (14)
O1^{i}—Zn1—N1^{i}	78.39 (5)	C4—C3—C2	121.50 (15)
O1—Zn1—N1	78.39 (6)	C4—C3—H3	119.2
O1^{i}—Zn1—N1	101.61 (6)	C2—C3—H3	119.2
N1^{i}—Zn1—N1	180.0	C3—C4—C5	117.84 (15)
O1—Zn1—O5	89.34 (5)	C3—C4—H4	121.1
O1^{i}—Zn1—O5	90.66 (5)	C5—C4—H4	121.1
N1^{i}—Zn1—O5	89.25 (5)	N1—C5—C4	121.79 (14)
N1—Zn1—O5	90.75 (5)	N1—C5—H5	119.1
O1—Zn1—O5^{i}	90.66 (5)	C4—C5—H5	119.1
O1^{i}—Zn1—O5^{i}	89.34 (5)	O1—C6—O2	122.63 (14)
N1^{i}—Zn1—O5^{i}	90.75 (5)	O1—C6—C1	117.58 (13)
N1—Zn1—O5^{i}	89.25 (5)	O2—C6—C1	119.78 (13)
O5—Zn1—O5^{i}	180.0	O3—C7—O4	120.60 (15)
C5—N1—C1	121.20 (13)	O3—C7—C2	118.49 (14)
C5—N1—Zn1	122.53 (10)	O4—C7—C2	120.90 (14)
C1—N1—Zn1	116.26 (10)	C6—O1—Zn1	116.54 (10)
N1—C1—C2	120.60 (14)	C7—O4—H4A	111.6
N1—C1—C6	111.00 (12)	Zn1—O5—H5A	109.4
C2—C1—C6	128.40 (13)	Zn1—O5—H5B	116.8
C3—C2—C1	117.05 (14)	H5A—O5—H5B	106.8

Symmetry codes: (i) $-x+1, -y+1, -z+2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5A···O3^{ii}	0.95	1.88	2.8035 (19)	164
O5—H5B···O3^{iii}	0.95	1.89	2.8358 (18)	174
O5—H5B···O4^{iii}	0.95	2.64	3.226 (2)	121
O4—H4A···O2	0.86	1.55	2.4017 (17)	173

Symmetry codes: (ii) $x-1/2, -y+3/2, z+1/2$; (iii) $-x+2, -y+1, -z+2$.

Fig. 1

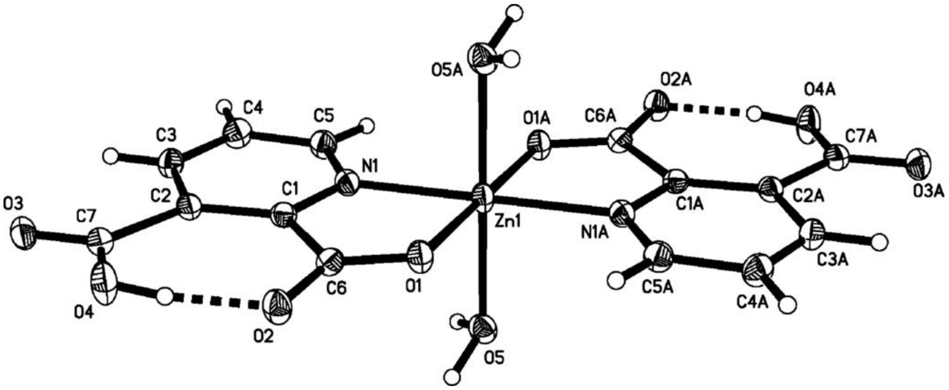
O5A
O2A
O4A
O1A
C6A
C7A
C4
C5
C3
N1
C1A
C2A
O3A
C1
C2
Zn1
N1A
O3
C7
C3A
C6
O1
C5A
C4A
O4
O2
O5

Fig. 2