

Diaquabis(5-methylpyridine-2-carboxylato- κ^2N,O)zinc(II)

Lian-Cai Du

College of Bioengineering, Weifang University, Weifang 261061, People's Republic of China

Correspondence e-mail: taixishi@lzu.edu.cn

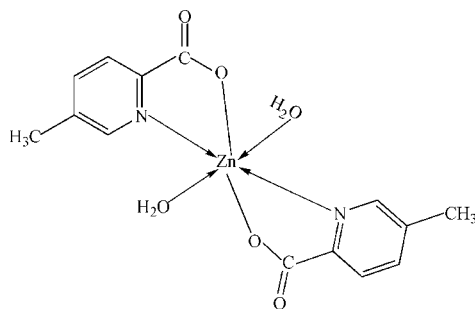
Received 13 December 2008; accepted 14 December 2008

 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(C-C) = 0.005$ Å; R factor = 0.034; wR factor = 0.097; data-to-parameter ratio = 11.8.

In the title compound, $[Zn(C_7H_6NO_2)_2(H_2O)_2]$, the Zn atom (site symmetry $\bar{1}$) adopts a distorted *trans*- ZnN_2O_4 octahedral coordination arising from two *N,O*-bidentate 5-methylpyridine-2-carboxylate ligands and two water molecules. In the crystal structure, molecules form a layered network linked by $O-H\cdots O$ hydrogen bonds.

Related literature

For background, see: Hagrman *et al.* (1998); Ranford *et al.* (1998).



Experimental

Crystal data

 $[Zn(C_7H_6NO_2)_2(H_2O)_2]$
 $M_r = 373.66$

 Triclinic, $P\bar{1}$
 $a = 5.1703$ (6) Å

 $b = 6.4620$ (10) Å

 $c = 12.2781$ (14) Å

 $\alpha = 104.678$ (2)°

 $\beta = 90.646$ (1)°

 $\gamma = 109.493$ (2)°

 $V = 372.01$ (8) Å³
 $Z = 1$

 Mo $K\alpha$ radiation

 $\mu = 1.68$ mm⁻¹
 $T = 298$ (2) K

 $0.49 \times 0.46 \times 0.27$ mm

Data collection

Bruker SMART CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2000)

 $T_{\min} = 0.493$, $T_{\max} = 0.659$

1917 measured reflections

1275 independent reflections

 1260 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.097$
 $S = 1.15$

1275 reflections

108 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.63$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³
Table 1

Selected bond lengths (Å).

Zn1—O1	2.104 (2)	Zn1—N1	2.116 (2)
Zn1—O3	2.134 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O2 ⁱ	0.85	1.88	2.693 (4)	160
O3—H3B \cdots O1 ⁱⁱ	0.85	1.94	2.757 (3)	160

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The author thanks the Science and Technology Foundation of Weifang (2008–19) for a research grant.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2880).

References

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supplementary materials

Acta Cryst. (2009). E65, m106 [doi:10.1107/S1600536808042530]

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Comment

As part of our efforts to achieve supramolecular transition metal complexes by self-assembly (Ranford, *et al.*, 1998; Hargman, *et al.*, 1998), we now report on the synthesis and crystal structure of the title compound, (I), (Fig. 1).

The Zn^{II} centre in (I) is six-coordinate with two O donors of H₂O, and two N,O-bidentate ligands (Table 1). In the crystal packing, the molecules form a layers linked by O—H...O hydrogen bonds (Table 2).

Experimental

A solution of 1.0 mmol 5-methylpyridine-2-carboxylic acid and 1.0 mmol NaOH in 5 ml 95% ethanol was added to a solution of 0.5 mmol Zn(CH₃COO)₂·4H₂O in 5 ml ethanol at room temperature. The mixture was refluxed for 2 h with stirring, then the resulting precipitate was filtered, washed, and dried *in vacuo* over P₄O₁₀ for 48 h. Colourless blocks of (I) were obtained by slowly evaporating from methanol at room temperature.

Refinement

The H atoms were geometrically placed (C—H = 0.93–0.96 Å, O—H = 0.85 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, O) or 1.5U_{eq}(methyl C).

Figures

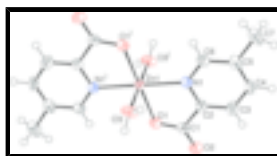


Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids for the non-hydrogen atoms. Symmetry code: (i) 1–x, 1–y, 1–z.

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Crystal data

[Zn(C₇H₆NO₂)₂(H₂O)₂]

M_r = 373.66

Triclinic, *PT*

Hall symbol: -P 1

a = 5.1703 (6) Å

Z = 1

*F*₀₀₀ = 192

D_x = 1.668 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 1975 reflections

supplementary materials

$b = 6.4620 (10) \text{ \AA}$	$\theta = 3.4\text{--}27.9^\circ$
$c = 12.2781 (14) \text{ \AA}$	$\mu = 1.68 \text{ mm}^{-1}$
$\alpha = 104.678 (2)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 90.646 (1)^\circ$	Block, colourless
$\gamma = 109.493 (2)^\circ$	$0.49 \times 0.46 \times 0.27 \text{ mm}$
$V = 372.01 (8) \text{ \AA}^3$	

Data collection

Bruker SMART CCD diffractometer	1275 independent reflections
Radiation source: fine-focus sealed tube	1260 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.013$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
ω scans	$\theta_{\text{min}} = 1.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -6 \rightarrow 3$
$T_{\text{min}} = 0.493$, $T_{\text{max}} = 0.659$	$k = -6 \rightarrow 7$
1917 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.034$	$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 0.3083P]$
$wR(F^2) = 0.097$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1275 reflections	$\Delta\rho_{\text{max}} = 0.63 \text{ e \AA}^{-3}$
108 parameters	$\Delta\rho_{\text{min}} = -0.60 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.094 (11)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.5000	0.5000	0.5000	0.0269 (2)
N1	0.4060 (5)	0.5257 (4)	0.6691 (2)	0.0265 (5)
C4	0.3260 (7)	0.5138 (6)	0.8903 (2)	0.0351 (7)
H4	0.2979	0.5079	0.9643	0.042*
O1	0.6819 (4)	0.2811 (3)	0.54326 (17)	0.0298 (5)
O2	0.7442 (5)	0.1369 (4)	0.6842 (2)	0.0399 (6)
O3	0.1258 (4)	0.2198 (4)	0.4350 (2)	0.0368 (5)
H3A	0.1410	0.0905	0.4068	0.044*
H3B	-0.0199	0.2048	0.4683	0.044*
C1	0.6533 (6)	0.2552 (5)	0.6414 (2)	0.0266 (6)
C2	0.4953 (6)	0.3907 (5)	0.7150 (2)	0.0269 (6)
C3	0.4520 (8)	0.3852 (6)	0.8251 (3)	0.0417 (8)
H3	0.5124	0.2887	0.8550	0.050*
C6	0.2809 (6)	0.6541 (5)	0.7341 (3)	0.0309 (6)
H6	0.2175	0.7490	0.7040	0.037*
C5	0.2430 (6)	0.6500 (6)	0.8452 (3)	0.0351 (7)
C7	0.1086 (8)	0.8011 (7)	0.9188 (3)	0.0500 (9)
H7A	-0.0093	0.7186	0.9650	0.075*
H7B	0.0010	0.8473	0.8718	0.075*
H7C	0.2483	0.9337	0.9664	0.075*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0346 (3)	0.0309 (3)	0.0229 (3)	0.0191 (2)	0.00841 (19)	0.01007 (19)
N1	0.0300 (12)	0.0281 (12)	0.0253 (12)	0.0143 (10)	0.0049 (10)	0.0083 (10)
C4	0.0477 (18)	0.0513 (19)	0.0163 (13)	0.0286 (15)	0.0102 (12)	0.0108 (13)
O1	0.0357 (11)	0.0321 (11)	0.0293 (11)	0.0209 (9)	0.0098 (8)	0.0089 (9)
O2	0.0559 (14)	0.0394 (12)	0.0369 (12)	0.0323 (11)	0.0035 (10)	0.0111 (10)
O3	0.0337 (11)	0.0298 (11)	0.0486 (13)	0.0161 (9)	0.0117 (10)	0.0067 (10)
C1	0.0274 (13)	0.0224 (13)	0.0306 (15)	0.0109 (11)	0.0018 (11)	0.0052 (11)
C2	0.0304 (14)	0.0264 (13)	0.0252 (14)	0.0119 (11)	0.0036 (11)	0.0066 (11)
C3	0.054 (2)	0.051 (2)	0.0337 (17)	0.0306 (17)	0.0084 (15)	0.0194 (15)
C6	0.0349 (15)	0.0321 (15)	0.0313 (15)	0.0196 (12)	0.0078 (12)	0.0076 (12)
C5	0.0341 (15)	0.0399 (17)	0.0289 (15)	0.0146 (13)	0.0065 (12)	0.0029 (13)
C7	0.054 (2)	0.059 (2)	0.0395 (19)	0.0315 (19)	0.0155 (16)	0.0005 (17)

Geometric parameters (\AA , $^\circ$)

Zn1—O1	2.104 (2)	O2—C1	1.232 (4)
Zn1—O3	2.134 (2)	O3—H3A	0.8499
Zn1—O1 ⁱ	2.104 (2)	O3—H3B	0.8499
Zn1—N1 ⁱ	2.116 (2)	C1—C2	1.531 (4)
Zn1—N1	2.116 (2)	C2—C3	1.380 (4)

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Zn1—O3 ⁱ	2.134 (2)	C3—H3	0.9300
N1—C6	1.334 (4)	C6—C5	1.387 (5)
N1—C2	1.343 (4)	C6—H6	0.9300
C4—C5	1.327 (5)	C5—C7	1.507 (4)
C4—C3	1.338 (5)	C7—H7A	0.9600
C4—H4	0.9300	C7—H7B	0.9600
O1—C1	1.262 (4)	C7—H7C	0.9600
O1—Zn1—O1 ⁱ	180.0	Zn1—O3—H3B	121.9
O1—Zn1—N1 ⁱ	100.78 (8)	H3A—O3—H3B	110.5
O1 ⁱ —Zn1—N1 ⁱ	79.22 (8)	O2—C1—O1	126.8 (3)
O1—Zn1—N1	79.22 (8)	O2—C1—C2	117.3 (3)
O1 ⁱ —Zn1—N1	100.78 (8)	O1—C1—C2	115.9 (2)
N1 ⁱ —Zn1—N1	180.0	N1—C2—C3	120.1 (3)
O1—Zn1—O3 ⁱ	89.38 (9)	N1—C2—C1	116.9 (2)
O1 ⁱ —Zn1—O3 ⁱ	90.62 (9)	C3—C2—C1	123.0 (3)
N1 ⁱ —Zn1—O3 ⁱ	92.23 (9)	C4—C3—C2	122.3 (3)
N1—Zn1—O3 ⁱ	87.77 (9)	C4—C3—H3	118.8
O1—Zn1—O3	90.62 (9)	C2—C3—H3	118.8
O1 ⁱ —Zn1—O3	89.38 (9)	N1—C6—C5	121.7 (3)
N1 ⁱ —Zn1—O3	87.77 (9)	N1—C6—H6	119.1
N1—Zn1—O3	92.23 (9)	C5—C6—H6	119.1
O3 ⁱ —Zn1—O3	180.0	C4—C5—C6	120.9 (3)
C6—N1—C2	117.7 (2)	C4—C5—C7	117.9 (3)
C6—N1—Zn1	130.4 (2)	C6—C5—C7	121.2 (3)
C2—N1—Zn1	111.95 (18)	C5—C7—H7A	109.5
C5—C4—C3	117.3 (3)	C5—C7—H7B	109.5
C5—C4—H4	121.3	H7A—C7—H7B	109.5
C3—C4—H4	121.3	C5—C7—H7C	109.5
C1—O1—Zn1	115.99 (17)	H7A—C7—H7C	109.5
Zn1—O3—H3A	116.6	H7B—C7—H7C	109.5
O1—Zn1—N1—C6	176.5 (3)	C6—N1—C2—C1	-176.5 (2)
O1 ⁱ —Zn1—N1—C6	-3.5 (3)	Zn1—N1—C2—C1	2.3 (3)
O3 ⁱ —Zn1—N1—C6	86.7 (3)	O2—C1—C2—N1	177.6 (3)
O3—Zn1—N1—C6	-93.3 (3)	O1—C1—C2—N1	-0.9 (4)
O1—Zn1—N1—C2	-2.21 (19)	O2—C1—C2—C3	0.0 (4)
O1 ⁱ —Zn1—N1—C2	177.79 (19)	O1—C1—C2—C3	-178.5 (3)
O3 ⁱ —Zn1—N1—C2	-92.0 (2)	C5—C4—C3—C2	-0.2 (6)
O3—Zn1—N1—C2	88.0 (2)	N1—C2—C3—C4	-1.1 (5)
N1 ⁱ —Zn1—O1—C1	-178.1 (2)	C1—C2—C3—C4	176.4 (3)
N1—Zn1—O1—C1	1.9 (2)	C2—N1—C6—C5	0.0 (4)
O3 ⁱ —Zn1—O1—C1	89.7 (2)	Zn1—N1—C6—C5	-178.6 (2)
O3—Zn1—O1—C1	-90.3 (2)	C3—C4—C5—C6	1.3 (5)
Zn1—O1—C1—O2	-179.4 (2)	C3—C4—C5—C7	-178.2 (3)
Zn1—O1—C1—C2	-1.2 (3)	N1—C6—C5—C4	-1.3 (5)
C6—N1—C2—C3	1.2 (4)	N1—C6—C5—C7	178.2 (3)

Zn1—N1—C2—C3 -180.0 (2)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3A \cdots O2 ⁱⁱ	0.85	1.88	2.693 (4)	160
O3—H3B \cdots O1 ⁱⁱⁱ	0.85	1.94	2.757 (3)	160

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

Fig. 1

