

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Diaquabis(picolinato *N*-oxide- κ^2O,O')-zinc(II)

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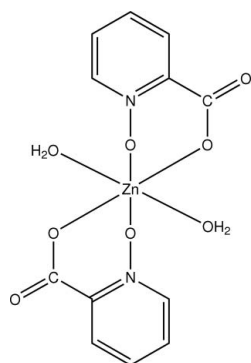
Received 31 August 2007; accepted 5 September 2007

 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(C-C) = 0.007$ Å; R factor = 0.046; wR factor = 0.149; data-to-parameter ratio = 11.0.

In the title compound, $[Zn(C_6H_4NO_3)_2(H_2O)_2]$, the Zn atom is located on a centre of inversion and shows a distorted octahedral coordination geometry. Two aqua ligands occupy the axial positions and four O atoms of the two chelating picolinic acid *N*-oxide ligands are located in the equatorial plane. Intermolecular hydrogen bonds between aqua ligands and organic ligands link molecules into a two-dimensional arrangement.

Related literature

For related literature, see: Bayot *et al.* (2006); Ciurtin *et al.* (2003); Lawrence *et al.* (1999); Meinrath *et al.* (2006); Shan *et al.* (2002); Steiner (2002); Yang *et al.* (2004); Zafar *et al.* (2000).



Experimental

Crystal data

$[Zn(C_6H_4NO_3)_2(H_2O)_2]$
 $M_r = 377.63$
 Monoclinic, $P2_1/c$
 $a = 6.6837$ (5) Å
 $b = 15.7376$ (13) Å
 $c = 6.9935$ (6) Å
 $\beta = 115.3700$ (10)°

$V = 664.67$ (9) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.90$ mm⁻¹
 $T = 298$ (2) K
 $0.21 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan *CrystalClear* (Rigaku, 2005)
 $T_{min} = 0.674$, $T_{max} = 0.733$

3515 measured reflections
 1170 independent reflections
 1033 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.149$
 $S = 1.15$
 1170 reflections

106 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.56$ e Å⁻³
 $\Delta\rho_{min} = -0.90$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Zn1—O3	2.049 (3)	Zn1—O1W	2.143 (3)
Zn1—O2	2.059 (3)		
O3—Zn1—O2	86.46 (11)	O2—Zn1—O1W ⁱ	88.93 (12)
O3—Zn1—O1W ⁱ	89.79 (11)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1WB \cdots O1 ⁱⁱ	0.90	1.98	2.753 (4)	143
O1W—H1WA \cdots O1 ⁱⁱⁱ	0.90	2.20	2.742 (4)	118

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the National Natural Science Foundation of China (project No. 20671019) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2040).

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supporting information

Acta Cryst. (2008). E64, m131 [https://doi.org/10.1107/S1600536807043681]

Diaquabis(picolinato *N*-oxide- κ^2 O,*O'*)zinc(II)**Xiu-Bing Li, Run-Ling Shang and Bai-Wang Sun****S1. Comment**

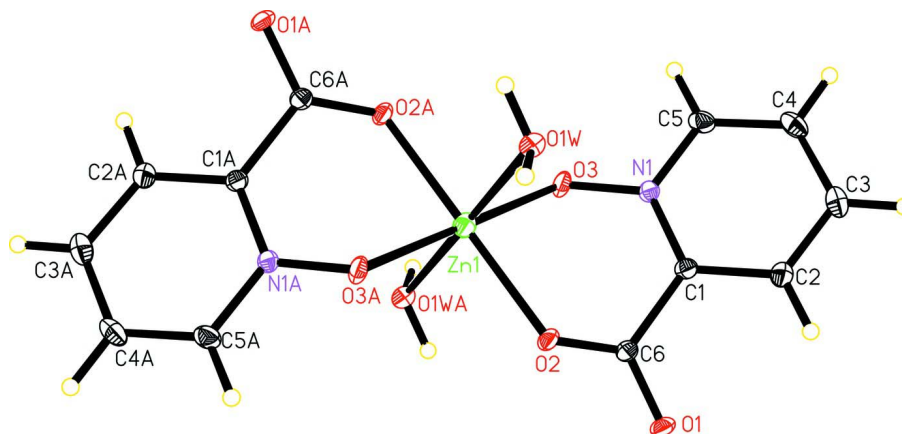
In the past decade, much attention has been paid to the design and synthesis of self-assembling systems with organic ligands containing N and O donors (Bayot *et al.*, 2006; Ciurtin *et al.*, 2003; Steiner, 2002; Zafar *et al.*, 2000). Picolinic acid *N*-oxide (PANO) is one such ligand and several crystal structures of complexes containing the PANO ligand have been reported (Yang *et al.*, 2004; Shan *et al.*, 2002; Lawrence *et al.*, 1999; Meinrath *et al.*, 2006). We report here the synthesis and crystal structure of the title complex, (I) (Fig. 1). In (I), the Zn atom is located on a crystallographic inversion centre and adopts a distorted octahedral coordination geometry. The coordination environment is defined by two pyridine *N*-oxide oxygen donors and two oxygen donors from the carboxylate groups located in the equatorial plane and two aqua O-atom donors located in the axial positions (Fig. 1). Selected bond lengths and angles are shown in Table 1. Intermolecular O1W—H1WA \cdots O1, O1W—H1WB \cdots O1 hydrogen bonds between water molecules and carboxylate groups connect the molecules of (I) into a two-dimensional network (Table 2 and Fig. 2).

S2. Experimental

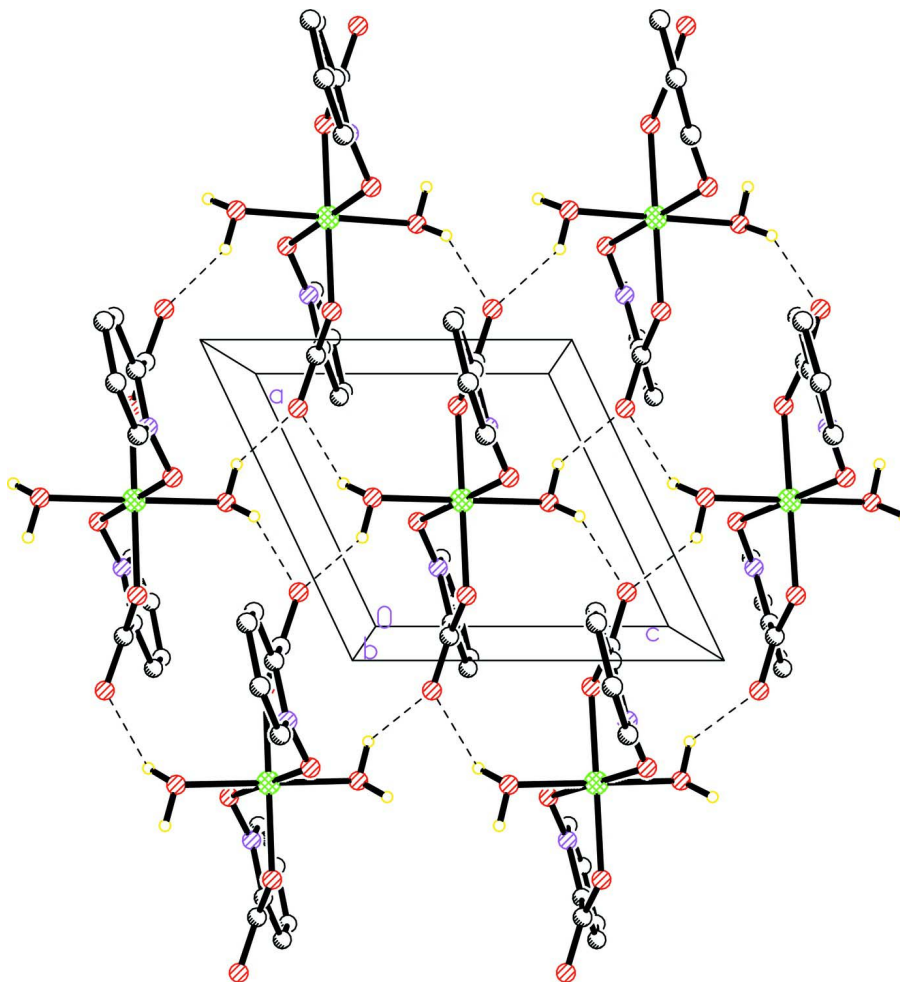
All chemicals were obtained from commercial sources and used without further purification. The title compound was prepared by the direct reaction of Zn(OOCCH₃)₂·2H₂O (22.1 mg, 0.1 mmol) and picolinic acid *N*-oxide (13.9 mg, 0.1 mmol) in water solution. Colourless block-shaped single crystals were obtained by slow evaporation at room temperature for about three weeks.

S3. Refinement

Positional parameters of all H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry operation (A): $[1 - x, 1 - y, 1 - z]$.

**Figure 2**

A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Diaquabis(picolinato N-oxide- κ^2O, O')zinc(II)

Crystal data

[Zn(C₆H₄NO₃)₂(H₂O)₂] $M_r = 377.63$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 6.6837$ (5) Å $b = 15.7376$ (13) Å $c = 6.9935$ (6) Å $\beta = 115.370$ (1)° $V = 664.67$ (9) Å³ $Z = 2$ $F(000) = 384$ $D_x = 1.887$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

 $\theta = 7.5$ – 15° $\mu = 1.90$ mm⁻¹ $T = 298$ K

Block, colourless

 $0.21 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10 pixels mm⁻¹ φ and ω scans

Absorption correction: multi-scan

CrystalClear (Rigaku, 2005) $T_{\min} = 0.674$, $T_{\max} = 0.733$

3515 measured reflections

1170 independent reflections

1033 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.019$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.6^\circ$ $h = -7 \rightarrow 7$ $k = -18 \rightarrow 17$ $l = -6 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.149$ $S = 1.15$

1170 reflections

106 parameters

0 restraints

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

 $w = 1/[\sigma^2(F_o^2) + (0.0918P)^2 + 1.2258P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.56$ e Å⁻³ $\Delta\rho_{\min} = -0.90$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.5000	0.5000	0.5000	0.0252 (3)
O1W	0.4917 (5)	0.43468 (18)	0.7653 (4)	0.0222 (7)
H1WA	0.6291	0.4170	0.8515	0.033*
H1WB	0.4423	0.4702	0.8365	0.033*

O1	-0.1745 (5)	0.49427 (16)	0.1316 (6)	0.0229 (7)
O2	0.1644 (5)	0.52224 (19)	0.3760 (5)	0.0218 (7)
O3	0.4215 (4)	0.38945 (17)	0.3288 (5)	0.0217 (7)
N1	0.2521 (5)	0.3424 (2)	0.3225 (5)	0.0168 (7)
C1	0.0530 (6)	0.3771 (2)	0.2833 (6)	0.0175 (8)
C2	-0.1198 (7)	0.3240 (3)	0.2650 (7)	0.0232 (9)
H2A	-0.2610	0.3483	0.2384	0.028*
C3	-0.0923 (8)	0.2365 (3)	0.2826 (7)	0.0287 (10)
H3A	-0.2135	0.1999	0.2667	0.034*
C4	0.1137 (8)	0.2034 (3)	0.3249 (7)	0.0273 (10)
H4A	0.1386	0.1432	0.3403	0.033*
C5	0.2825 (7)	0.2572 (3)	0.3436 (7)	0.0234 (9)
H5A	0.4259	0.2341	0.3737	0.028*
C6	0.0146 (6)	0.4725 (3)	0.2616 (6)	0.0177 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0186 (5)	0.0236 (5)	0.0293 (5)	-0.0011 (2)	0.0064 (4)	-0.0029 (3)
O1W	0.0178 (14)	0.0239 (15)	0.0222 (14)	0.0006 (11)	0.0059 (11)	0.0006 (11)
O1	0.0128 (16)	0.0208 (16)	0.0256 (17)	0.0033 (10)	-0.0008 (13)	-0.0021 (11)
O2	0.0118 (14)	0.0176 (13)	0.0289 (16)	-0.0007 (11)	0.0020 (12)	-0.0062 (13)
O3	0.0131 (13)	0.0210 (14)	0.0313 (16)	-0.0047 (11)	0.0099 (12)	-0.0094 (12)
N1	0.0162 (16)	0.0150 (16)	0.0172 (16)	-0.0009 (12)	0.0051 (13)	-0.0036 (13)
C1	0.0165 (19)	0.0163 (19)	0.0172 (19)	0.0014 (15)	0.0048 (16)	-0.0027 (15)
C2	0.019 (2)	0.021 (2)	0.028 (2)	-0.0020 (16)	0.0087 (18)	-0.0012 (17)
C3	0.032 (3)	0.023 (2)	0.032 (2)	-0.0086 (18)	0.015 (2)	-0.0015 (18)
C4	0.039 (3)	0.014 (2)	0.029 (2)	0.0003 (17)	0.014 (2)	-0.0004 (17)
C5	0.027 (2)	0.0164 (19)	0.025 (2)	0.0044 (16)	0.0089 (19)	-0.0026 (16)
C6	0.0146 (19)	0.0186 (19)	0.022 (2)	0.0019 (16)	0.0101 (16)	0.0006 (16)

Geometric parameters (Å, °)

Zn1—O3 ⁱ	2.049 (3)	N1—C1	1.354 (5)
Zn1—O3	2.049 (3)	N1—C5	1.356 (5)
Zn1—O2 ⁱ	2.059 (3)	C1—C2	1.386 (6)
Zn1—O2	2.059 (3)	C1—C6	1.520 (5)
Zn1—O1W ⁱ	2.143 (3)	C2—C3	1.389 (6)
Zn1—O1W	2.143 (3)	C2—H2A	0.9600
O1W—H1WA	0.9000	C3—C4	1.381 (7)
O1W—H1WB	0.9001	C3—H3A	0.9601
O1—C6	1.247 (5)	C4—C5	1.371 (6)
O2—C6	1.253 (5)	C4—H4A	0.9597
O3—N1	1.338 (4)	C5—H5A	0.9600
O3 ⁱ —Zn1—O3	180.00 (8)	O3—N1—C5	117.3 (3)
O3 ⁱ —Zn1—O2 ⁱ	86.46 (11)	C1—N1—C5	120.6 (3)
O3—Zn1—O2 ⁱ	93.54 (11)	N1—C1—C2	118.9 (4)

O3 ⁱ —Zn1—O2	93.54 (11)	N1—C1—C6	121.8 (3)
O3—Zn1—O2	86.46 (11)	C2—C1—C6	119.2 (3)
O2 ⁱ —Zn1—O2	180.0	C3—C2—C1	121.1 (4)
O3 ⁱ —Zn1—O1W ⁱ	90.21 (11)	C3—C2—H2A	119.6
O3—Zn1—O1W ⁱ	89.79 (11)	C1—C2—H2A	119.3
O2 ⁱ —Zn1—O1W ⁱ	91.07 (12)	C2—C3—C4	118.3 (4)
O2—Zn1—O1W ⁱ	88.93 (12)	C2—C3—H3A	120.8
O3 ⁱ —Zn1—O1W	89.79 (11)	C4—C3—H3A	120.9
O3—Zn1—O1W	90.21 (11)	C5—C4—C3	119.5 (4)
O2 ⁱ —Zn1—O1W	88.93 (12)	C5—C4—H4A	120.2
O2—Zn1—O1W	91.07 (12)	C3—C4—H4A	120.3
O1W ⁱ —Zn1—O1W	180.00 (13)	N1—C5—C4	121.5 (4)
Zn1—O1W—H1WA	109.3	N1—C5—H5A	119.2
Zn1—O1W—H1WB	109.5	C4—C5—H5A	119.4
H1WA—O1W—H1WB	109.5	O2—C6—O1	125.2 (4)
C6—O2—Zn1	126.3 (3)	O2—C6—C1	119.9 (4)
N1—O3—Zn1	119.4 (2)	O1—C6—C1	114.8 (3)
O3—N1—C1	121.9 (3)		
O3 ⁱ —Zn1—O2—C6	-175.0 (3)	C2—C1—C2—C3	0 (100)
O3—Zn1—O2—C6	5.0 (3)	C6—C1—C2—C3	179.7 (4)
O1W ⁱ —Zn1—O2—C6	94.8 (3)	C1—C2—C3—C2	0 (2)
O1W—Zn1—O2—C6	-85.2 (3)	C2—C2—C3—C4	0.0 (7)
O2 ⁱ —Zn1—O3—N1	137.7 (3)	C1—C2—C3—C4	1.8 (7)
O2—Zn1—O3—N1	-42.3 (3)	C2—C3—C4—C5	-1.4 (7)
O1W ⁱ —Zn1—O3—N1	-131.2 (3)	C2—C3—C4—C5	-1.4 (7)
O1W—Zn1—O3—N1	48.8 (3)	O3—N1—C5—C4	-175.5 (4)
Zn1—O3—N1—C1	47.5 (4)	C1—N1—C5—C4	0.4 (6)
Zn1—O3—N1—C5	-136.6 (3)	C3—C4—C5—N1	0.3 (6)
O3—N1—C1—C2	175.7 (3)	Zn1—O2—C6—O1	-154.2 (3)
C5—N1—C1—C2	0.0 (6)	Zn1—O2—C6—C1	28.9 (5)
O3—N1—C1—C2	175.7 (3)	N1—C1—C6—O2	-36.9 (6)
C5—N1—C1—C2	0.0 (6)	C2—C1—C6—O2	142.3 (4)
O3—N1—C1—C6	-5.1 (5)	C2—C1—C6—O2	142.3 (4)
C5—N1—C1—C6	179.2 (4)	N1—C1—C6—O1	145.9 (4)
N1—C1—C2—C2	0.0 (2)	C2—C1—C6—O1	-34.9 (5)
C6—C1—C2—C2	0.00 (6)	C2—C1—C6—O1	-34.9 (5)
N1—C1—C2—C3	-1.1 (6)		

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

Hydrogen-bond geometry (Å, °)

<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>
O1W—H1WB \cdots O1 ⁱⁱ	0.90	1.98	2.753 (4)	143
O1W—H1WA \cdots O1 ⁱⁱⁱ	0.90	2.20	2.742 (4)	118

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