

Diaquabis(1,10-phenanthroline- κ^2N,N')-
zinc(II) pamoate monohydrateAi-Qing Ma,^{a*} Zhen-Bin Jia^a and
Guo-Ping Wang^b^aSchool of Basic Course, Guangdong Medical College, Zhanjiang 524023, People's Republic of China, and ^bDepartment of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of ChinaCorrespondence e-mail:
maq197511@yahoo.com.cn

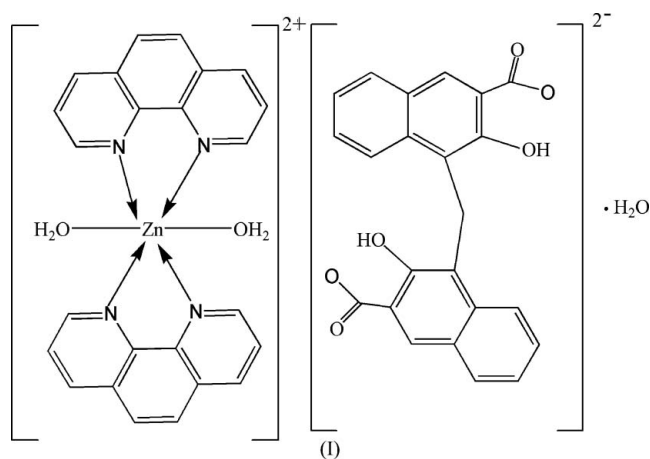
Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.084
 wR factor = 0.160
Data-to-parameter ratio = 11.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $[\text{Zn}(\text{C}_{12}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_2](\text{C}_{23}\text{H}_{14}\text{O}_6)\cdot\text{H}_2\text{O}$, the Zn^{II} ion lies on a twofold axis and exhibits an approximately octahedral configuration. The pamoate dianion [or 4,4-methylenebis(3-hydroxy-2-naphthoate)], also lying on a twofold axis, is uncoordinated and balances the charge. In the crystal structure, $\text{O}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds link the mononuclear ions into a two-dimensional network.

Comment

Pamoic acid is a well known aromatic dicarboxylic acid and has been used as a means of masking unpleasant tastes and for obtaining prolonged therapeutic action by forming slightly soluble salts with certain drugs (Dryden & Ridley, 1999; Jorgensen, 1998; Morovjan *et al.*, 1998). However, in the construction of metal-organic coordination polymers, pamoic acid as a ligand is comparable with other aromatic dicarboxylic acids, such as benzene-1,4-dicarboxylic acid and 5-hydroxy-isophthalic acid (Hong & You, 2004; Li *et al.*, 2004; Plater *et al.*, 2001; Xiao *et al.*, 2005; Xiao & Zhu, 2003; Zhu *et al.*, 2004), and the complexes are very limited. We report here the crystal structure of the title compound $[\text{Zn}(\text{phen})_2(\text{H}_2\text{O})_2](\text{pa})\cdot(\text{H}_2\text{O})$, (I), where phen is 1,10-phenanthroline and pa is the pamoate dianion.

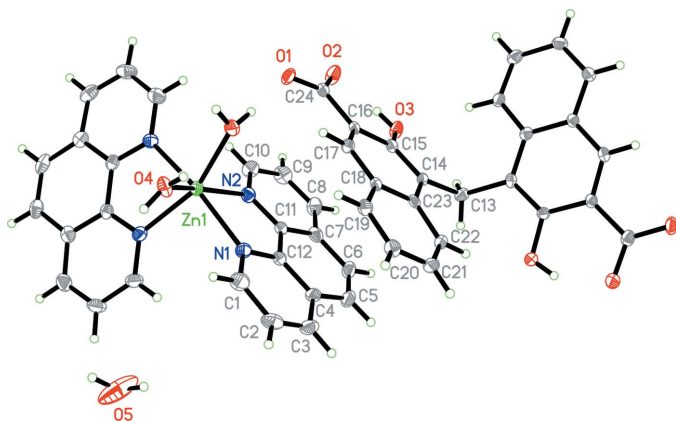


The title compound, (I), consists of a $[\text{Zn}(\text{phen})_2(\text{H}_2\text{O})_2]^+$ cation, a pamoate dianion and one uncoordinated water molecule (Fig. 1). The coordination geometry of the Zn^{II} atom is best described as distorted octahedral, comprising four N atoms of two 1,10-phenanthroline molecules and two O atoms from two coordinated water molecules; geometric parameters are given in Table 1. The Zn^{II} ion lies on a crystallographic twofold axis. The pamoate dianion, which also lies on a crystallographic twofold axis, does not coordinate to the Zn^{II} atom

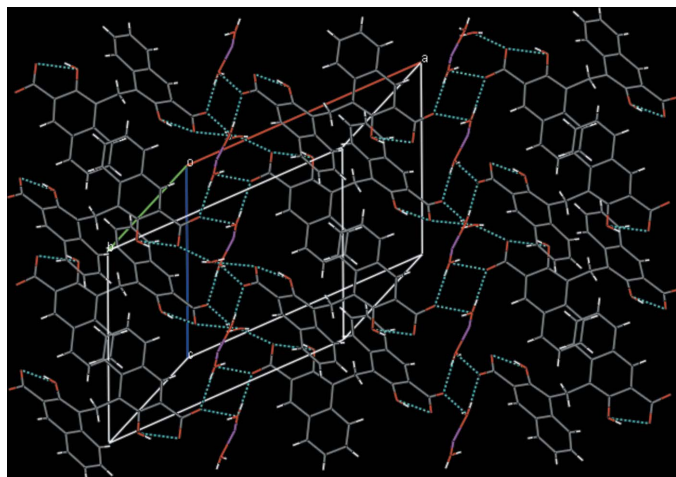
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Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering scheme. Unlabelled atoms are related to labelled atoms by the symmetry operation $(1 - x, y, \frac{1}{2} - z)$ in the cation and $(2 - x, y, \frac{1}{2} - z)$ in the anion.


Figure 2

The two-dimensional network formed by hydrogen-bonding interactions (dashed lines). For clarity, the 1,10-phenanthroline ligands have been omitted.

but balances the charge. The dihedral angle between the two phen ligands is $73.0(1)^\circ$, while the dihedral angle between the two pamoate ring systems is $74.7(2)^\circ$.

The stability of the solid-state structure of (I) is enhanced significantly by hydrogen-bonding interactions (Table 2). An intramolecular hydrogen bond exists between atom O3 and the uncoordinated carboxylate atom O2. Two intermolecular hydrogen bonds are formed between atom O4 of the coordinated water and the uncoordinated carboxylate atom O1. An additional intermolecular hydrogen bond is formed between atom O5 of the uncoordinated water molecule and the uncoordinated carboxylate atom O2. All these intermolecular hydrogen bonds link neighbouring mononuclear ions into a two-dimensional network (Fig. 2).

Experimental

An aqueous solution (10 ml) containing $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.30 mmol, 0.084 g) was added slowly to a solution (10 ml) of *N,N*-dimethyl-

formamide containing 1,10-phenanthroline (0.30 mmol, 0.054 g) and pamoic acid (0.30 mmol, 0.110 g). Yellow crystals suitable for X-ray analysis were obtained on allowing the solution to stand at room temperature for 14 d.

Crystal data

$[\text{Zn}(\text{C}_{12}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_2] \cdot (\text{C}_{23}\text{H}_{14}\text{O}_6) \cdot \text{H}_2\text{O}$
 $M_r = 866.17$
 Monoclinic, $C2/c$
 $a = 15.6006(11) \text{ \AA}$
 $b = 22.8892(17) \text{ \AA}$
 $c = 11.5193(8) \text{ \AA}$
 $\beta = 115.704(2)^\circ$
 $V = 3706.3(5) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.552 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 1053 reflections
 $\theta = 2.6\text{--}23.5^\circ$
 $\mu = 0.73 \text{ mm}^{-1}$
 $T = 298(2) \text{ K}$
 Prism, yellow
 $0.28 \times 0.11 \times 0.06 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.821, T_{\max} = 0.952$
 9911 measured reflections

3375 independent reflections
 2823 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$
 $\theta_{\text{max}} = 25.3^\circ$
 $h = -13 \rightarrow 18$
 $k = -27 \rightarrow 21$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.084$
 $wR(F^2) = 0.160$
 $S = 1.20$
 3375 reflections
 290 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 6.9583P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.35 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

Zn1—O4	2.079 (3)	Zn1—N1	2.222 (4)
Zn1—N2	2.174 (4)		
O4—Zn1—O4 ⁱ	79.5 (2)	N2—Zn1—N1 ⁱ	93.99 (14)
O4—Zn1—N2	160.12 (13)	O4—Zn1—N1	89.31 (14)
O4—Zn1—N2 ⁱ	91.57 (14)	N2—Zn1—N1	75.41 (14)
N2—Zn1—N2 ⁱ	102.0 (2)	N1 ⁱ —Zn1—N1	163.4 (2)
O4—Zn1—N1 ⁱ	103.56 (14)		

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

Table 2

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

$D\text{—}H \cdots A$	$D\text{—}H$	$H \cdots A$	$D \cdots A$	$D\text{—}H \cdots A$
O4—H4B \cdots O1 ⁱ	0.83 (2)	1.84 (2)	2.673 (4)	177 (5)
O4—H4A \cdots O1 ⁱⁱ	0.83 (2)	2.01 (3)	2.757 (5)	149 (5)
O5—H5A \cdots O2 ⁱ	0.84 (2)	2.06 (6)	2.839 (5)	154 (12)
O3—H3 \cdots O2	0.82	1.76	2.508 (4)	150

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

H atoms of the water molecules were located in a difference map and were refined isotropically, with O—H and H \cdots H distance restraints of 0.84 (1) and 1.37 (2) \AA , respectively. The remaining H atoms were positioned geometrically (C—H = 0.93 \AA or O—H = 0.82 \AA) and allowed to ride on their parent atoms. The $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O})$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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