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Bis(glycolato-κ^2O,O')(1,10-phenanthroline-κ^2N,N')-zinc(II) dihydrate

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Key indicators

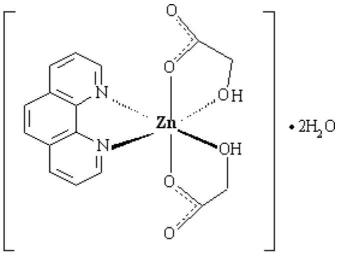
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.087
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Zn atom in the title mononuclear complex, $[Zn(C_2H_3O_3)_2(C_{12}H_8N_2)]\cdot 2H_2O$, exists in an octahedral coordination environment defined by two hydroxy O atoms, two carboxylate O atoms from different glycolate (hydroxyacetate) ligands and two N atoms from one phenanthroline (phen) ligand. The Zn atom occupies a special position with twofold symmetry. A layer structure is formed *via* O—H···O hydrogen bonds involving the water molecules.

Comment

Glycolic acid (hydroxyacetic acid) is a biologically active compound and has versatile binding modes. A number of structures of metal complexes containing the glycolate ligand have been reported (Venema *et al.*, 1990; Prout *et al.*, 1993; Svancarek *et al.*, 2000; Melikyan *et al.*, 2000). In the structures of these complexes, the glycolate ligand coordinates to the metal ions through the hydroxy and carboxy groups with a five-membered chelating mode, and the minority of hydroxyl groups of the glycolate are deprotonated (Dengel *et al.*, 1987; Lanfranchi *et al.*, 1993). However, Zn^{II} complexes with glycolic acid are less well documented (Fischinger & Webb, 1969). In order to explore further the coordination behavior and solid-state structure of Zn^{II} with the glycolate ligand, we have synthesized the title complex, (I), by the reaction of zinc acetate dihydrate, glycolic acid and 1,10-phenanthroline (phen), and its crystal structure is reported here.



(I)

As shown in Fig. 1, (I) has a mononuclear structure with Zn^{II} coordinated to one phen and two glycolate ligands. The Zn atom lies on a twofold axis and its octahedral coordination is made up of two hydroxy O atoms, two carboxy O atoms and two phen N atoms. The Zn—O3(hydroxy) distance [2.157 (2) Å] is longer that of Zn—O1(carboxy) [2.042 (2) Å], and the Zn—N distance is 2.132 (2) Å. The five-membered ring consisting of atoms Zn^{II}, O1, C7, C8 and O3 is essentially planar, with an r.m.s. deviation of 0.006 (3) Å. The dihedral angle between the five-membered ring and the phen ligand is

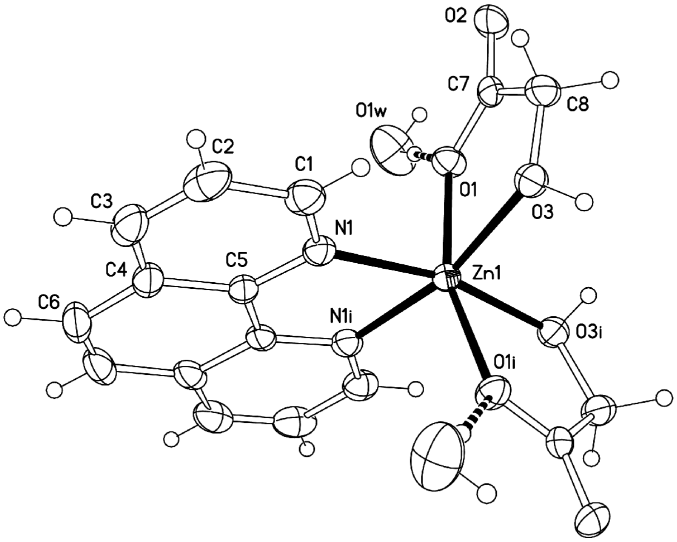


Figure 1
View of the title compound, with 30% probability ellipsoids for the non-H atoms [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$].

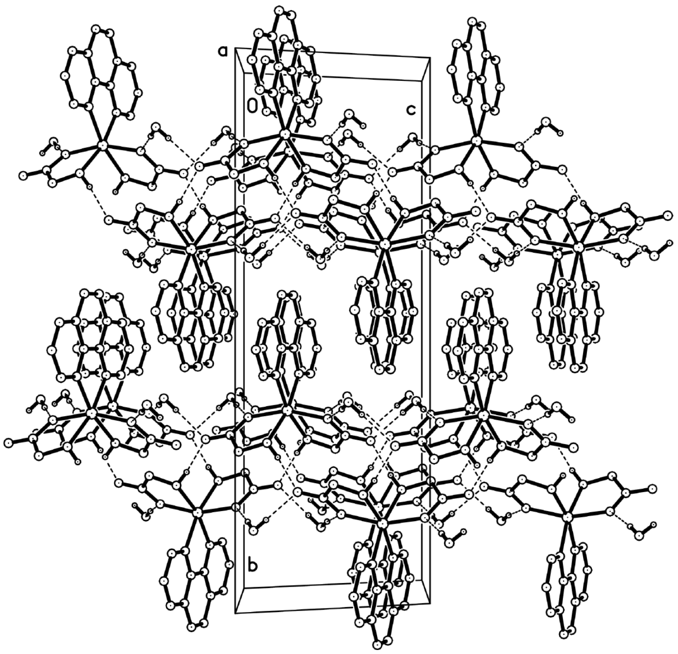


Figure 2
Packing diagram of the complex, viewed perpendicular to the bc plane. All C—H H atoms have been omitted for clarity.

81.5 (4)°. The C7—O1 and C7—O2 bond lengths [1.257 (3) and 1.242 (3) Å, respectively] are nearly equivalent, indicating the extent of delocalization in the carboxylate group. Hydrogen bonds are formed between water molecules, glycolate hydroxy O atoms and glycolate carboxy O atoms, giving rise to an O—H···O hydrogen-bonded chain along the crystallographic c axis (see Table 2 for hydrogen-bonding geometries). Furthermore, there are π–π stacking interactions between adjacent phen ligands [centroid–centroid distance = 3.553 (3) Å], resulting in an extended layer structure parallel to the bc plane.

Experimental

1,10-Phenanthroline (1.35 g, 7.5 mmol) was dissolved in water–ethanol (1:1, 50 ml) and glycolic acid (1.14 g, 15 mmol) and zinc acetate dihydrate (3.00 g, 15 mmol) were added. The pH was adjusted to 6 with 0.2 M NaOH solution. The reaction mixture was then stirred at room temperature for 3 h and filtered. Colorless single crystals were obtained from the filtered solution over several days. Analysis calculated for $C_{16}H_{18}N_2O_8Zn$: C 44.51, H 4.20, N 6.50%; found: C 44.38, H 4.12, N 6.32%.

Crystal data

$[Zn(C_2H_3O_3)_2(C_{12}H_8N_2)]\cdot 2H_2O$
$M_r = 431.71$
Monoclinic, $C2/c$
$a = 8.2827\ (9)$ Å
$b = 24.480\ (4)$ Å
$c = 9.1094\ (8)$ Å
$\beta = 109.588\ (4)°$
$V = 1740.1\ (4)$ Å^3
$Z = 4$
$D_x = 1.648$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6065 reflections
$\theta = 3.6$–$27.4°$
$\mu = 1.46$ mm^{-1}
$T = 293\ (2)$ K
Prism, colorless
0.39 × 0.25 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.593$, $T_{\max} = 0.771$
8212 measured reflections
1999 independent reflections
1625 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -31 \rightarrow 31$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.087$
$S = 1.07$
1999 reflections
126 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.5291P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.132 (2)	O1—C7	1.257 (3)
Zn1—O1	2.042 (2)	O2—C7	1.242 (3)
Zn1—O3	2.157 (2)		
N1—Zn1—N1^{i}	78.1 (1)	O1—Zn1—O3	77.06 (7)
N1—Zn1—O3^{i}	167.08 (7)	O1—Zn1—O3^{i}	90.27 (7)
N1—Zn1—O3	92.02 (8)	O1^{i}—Zn1—N1^{i}	98.95 (7)
O1^{i}—Zn1—N1	96.10 (7)	O3^{i}—Zn1—O3	98.9 (1)
O1—Zn1—O1^{i}	160.6 (1)	N1^{i}—Zn1—O3	167.08 (7)

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···O1	0.85	2.01	2.857 (3)	171
O1W—H2W···O2^{ii}	0.85	2.14	2.989 (4)	179
O3—H10···O2^{iii}	0.84 (3)	1.81 (3)	2.639 (3)	169 (3)

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

H atoms bonded to carbon were placed in calculated positions, with C—H = 0.93 or 0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C), and were

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included in the refinement in the riding-model approximation. Water H atoms were located in difference Fourier maps and then included in fixed positions, with O—H = 0.85 Å and U_{iso}(H) = 1.5U_{eq}(O). The H atom of the carboxy group was located from a difference Fourier and refined, with O—H = 0.84 (3) Å and U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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