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Bis[aquachlorobis(1,10-phenanthroline)-zinc(II)] benzene-1,4-dioxyacetate dihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.104
Data-to-parameter ratio = 15.8

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

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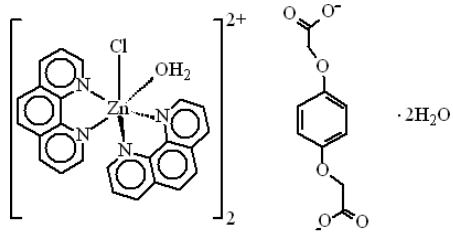
The title complex, $[ZnCl(C_{12}H_8N_2)_2(H_2O)]_2(C_{10}H_8O_6)\cdot 2H_2O$ ($C_{10}H_8O_6$ = benzene-1,4-dioxyacetate and phen = 1,10-phenanthroline) consists of two $[ZnCl(phen)_2(H_2O)]^{2+}$ cations, a benzene-1,4-dioxyacetate dianion and two water molecules. In the cation, the Zn^{II} atom is coordinated by four N atoms from two phen ligands, one Cl^- anion and one water molecule, forming a distorted octahedral coordination environment. The benzene-1,4-dioxyacetate dianion lies on an inversion center, with anions and cations forming one-dimensional chains through O—H···O hydrogen bonds.

Comment

Recently, we have reported the crystal structure of the complex bis[aquachlorobis(1,10-phenanthroline)manganese(II)] benzene-1,4-dioxyacetate dihydrate (Gao *et al.*, 2004). The title zinc(II) analog, (I), was synthesized under similar reaction conditions and is reported here. A similar structural description of the Mn complex applies to the present isomorphous complex. As shown in Fig. 1, the Zn^{II} coordination is slightly distorted octahedral, defined by four N atoms from two phen ligands, one Cl^- anion and one water molecule. The Zn—N bond lengths are somewhat different from the Mn—N analogous ones, compared with 2.242 (2)–2.338 (2) Å in the Mn complex (Gao *et al.*, 2004), ranging from 2.263 (2) to 2.130 (2) Å. The water molecules form hydrogen bonds with carboxylate O atoms of adjacent anions and chloro ligands of adjacent cations,, resulting in a one-dimensional chain (Table 2 and Fig. 2). In the crystal structure, the shortest ring-centroid to ring-centroid distances between symmetry-related phen ligands are 3.77 (4) and 3.78 (4) Å, and it is unlikely that these distances give rise to significant π–π stacking interactions.



(I)

Experimental

Benzene-1,4-dioxyacetic acid was prepared according to the method described for the synthesis of benzene-1,2-dioxyacetic acid by Mirci (1990). The title complex was prepared by the addition of phen (20 mmol) and zinc(II) acetate dihydrate (20 mmol) to an aqueous solution of benzene-1,4-dioxyacetic acid (40 mmol), and the pH was adjusted to 6 with 0.1 *M* sodium hydroxide. Colorless single crystals

were isolated from the filtered solution after several days. Analysis calculated for $C_{58}H_{48}Cl_2N_8O_{10}Zn_2$: C 62.95, H 4.37%.; found: C 63.19, H 4.49%.

Crystal data

$[ZnCl(C_{12}H_8N_2)_2(H_2O)]_2$-$(C_{10}H_8O_6)\cdot 2H_2O$	$Z = 1$
$M_r = 1218.72$	$D_x = 1.554$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.950$ (6) Å	Cell parameters from 6638 reflections
$b = 12.06$ (1) Å	$\theta = 3.3$–$27.5°$
$c = 12.81$ (1) Å	$\mu = 1.09$ mm^{-1}
$\alpha = 61.95$ (3)°	$T = 293$ (2) K
$\beta = 80.65$ (4)°	Prism, colorless
$\gamma = 73.92$ (3)°	0.37 × 0.25 × 0.18 mm
$V = 1302.6$ (17) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	5884 independent reflections
ω scans	4682 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$R_{int} = 0.034$
$T_{min} = 0.687$, $T_{max} = 0.827$	$\theta_{max} = 27.5°$
12 345 measured reflections	$h = -12 \rightarrow 11$
	$k = -15 \rightarrow 15$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.3253P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.104$	$\Delta\rho_{max} = 0.59$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.39$ e Å^{-3}
5884 reflections	
373 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

Zn1—Cl1	2.355 (2)	Zn1—N3	2.130 (2)
Zn1—N1	2.263 (2)	Zn1—N4	2.264 (2)
Zn1—N2	2.156 (2)	Zn1—O1*W*	2.138 (2)
N1—Zn1—Cl1	171.26 (5)	N3—Zn1—N4	76.07 (8)
N1—Zn1—N4	87.94 (8)	N3—Zn1—O1*W*	96.63 (8)
N2—Zn1—Cl1	97.48 (8)	N4—Zn1—Cl1	97.05 (7)
N2—Zn1—N1	75.33 (9)	O1*W*—Zn1—Cl1	92.24 (7)
N2—Zn1—N4	89.26 (8)	O1*W*—Zn1—N1	83.66 (8)
N3—Zn1—Cl1	97.56 (8)	O1*W*—Zn1—N2	95.68 (8)
N3—Zn1—N1	90.59 (9)	O1*W*—Zn1—N4	168.84 (7)
N3—Zn1—N2	160.13 (7)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O2^{i}	0.85 (4)	1.93 (3)	2.776 (3)	171 (3)
O1*W*—H1*W*2···O1	0.84 (1)	1.94 (1)	2.778 (3)	172 (3)
O2*W*—H2*W*1···Cl1^{i}	0.87 (7)	2.52 (7)	3.333 (5)	156 (7)
O2*W*—H2*W*2···O1	0.87 (7)	2.22 (4)	3.013 (5)	152 (6)

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

C-bound H atoms were placed in calculated positions [C—H = 0.93 (aromatic) or 0.97 Å (aliphatic) and $U_{iso}(H) = 1.2U_{eq}(C)$] in the riding-model approximation. The H atoms of water molecules were located from difference Fourier synthesis maps and refined with O—H and H···H distance restraints [0.85 (1) and 1.39 (1) Å]; $U_{iso}(H) = 1.5U_{eq}(O)$.

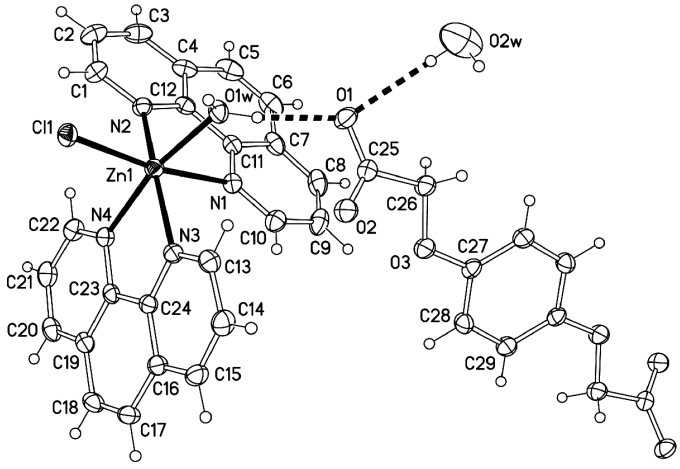


Figure 1
*ORTEP*II (Johnson, 1976) drawing of the title complex, showing 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines and the unlabeled atoms are related by the symmetry transformation $(-x, 1-y, 1-z)$.

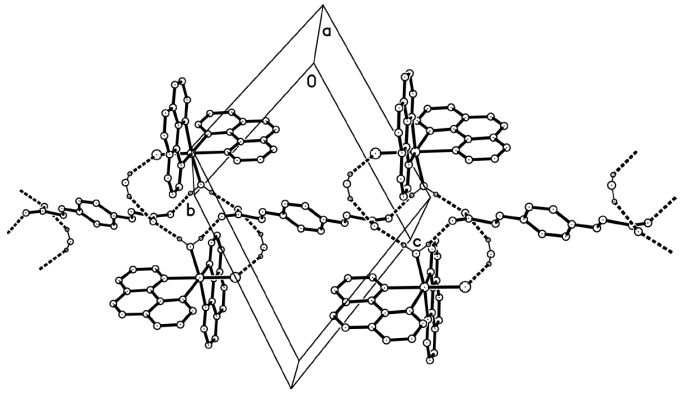


Figure 2
The hydrogen-bonded chain structure of the title complex.

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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