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[2-(3,5-Dibromo-2-oxidobenzylamino)-3-phenylpropanoato-κ^3N,O,O'](1,10-phenanthroline-κ^2N,N')zinc(II) dihydrate

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Key indicators

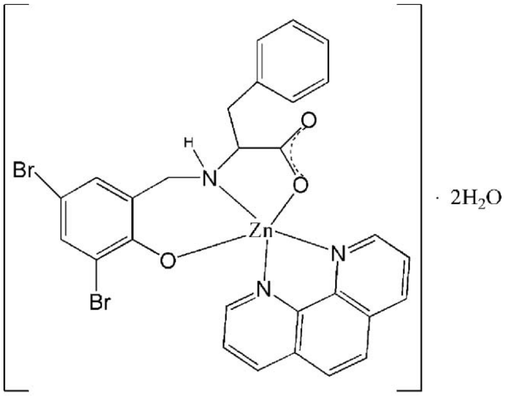
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.010 Å
R factor = 0.054
wR factor = 0.165
Data-to-parameter ratio = 18.6

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

In the title complex, $[Zn(C_{16}H_{13}Br_2NO_3)(C_{12}H_8N_2)]\cdot 2H_2O$ or $[Zn(L)(phen)]\cdot 2H_2O$, [LH_2 = 2-(3,5-dibromo-2-hydroxybenzylamino)-3-phenylpropionic acid and phen = 1,10-phenanthroline], the Zn^{II} atom is coordinated in a slightly distorted square-pyramidal geometry by two O atoms and one N atom from an L^{2-} ligand and two N atoms from a phen ligand. One of the carboxylate O atoms is in the apical position. In the crystal structure, complex molecules are linked into a chain along the c axis through N—H···O hydrogen bonds; the water molecules are connected to the chains *via* O—H···O hydrogen bonds.

Comment

Schiff base complexes containing amino acids have been studied for many years (Wang *et al.*, 1994; Casella & Gullotti, 1986; Jiang *et al.*, 2003; Zhang *et al.*, 2003, Sreenivasulu *et al.*, 2005), and have aroused increasing interest because of their antiviral, anticancer and antibacterial activities. Most model studies of metal complexes of Schiff base ligands containing salicylaldehyde and amino acids have focused on the binding mode of these ligands (Nakagima *et al.* 1989; Kettmann *et al.*, 1993). The crystal structures of the complexes obtained demonstrate that the Schiff base ligands act in a tridentate mode, coordinating through the phenolato O, imine N, and carboxylate O atoms. Our research group is interested in tridentate reduced Schiff bases; they are more flexible because the C═N bond of the Schiff base has been reduced, and this helps to overcome the ligand instability (Sreenivasulu *et al.*, 2005; Zhang *et al.*, 2006).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The Zn^{II} atom is coordinated by two O atoms and one N atom from an

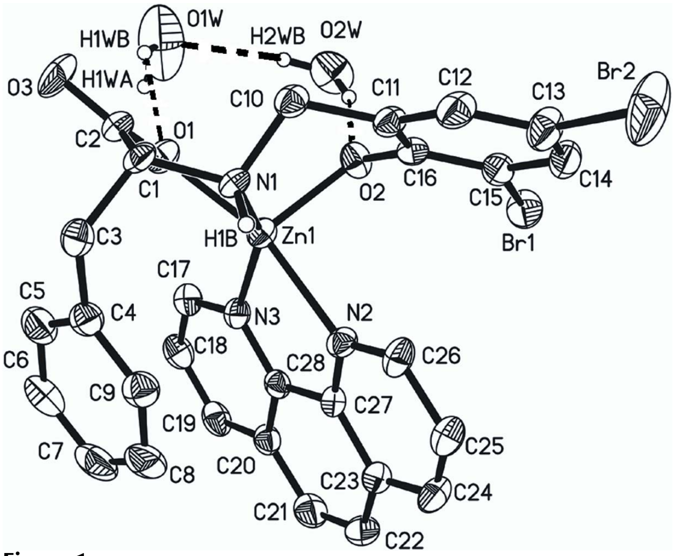


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines. Carbon-bound H atoms have been omitted.

L^{2-} ligand and two N atoms from a phen ligand with atom O1 in the apical position, giving a slightly distorted square-pyramidal geometry (LH_2 = 2-(3,5-dibromo-2-hydroxybenzylamino)-3-phenylpropionic acid and phen = 1,10-phenanthroline). The distance between the centroid of the C4–C9 phenyl ring and atom Zn1 is 3.694 (8) Å.

In the crystal structure, N—H···O hydrogen bonds involving the carboxylate O atoms link the complex molecules into a chain along the *c* axis. The water molecules are connected to the chains *via* O—H···O hydrogen bonds (Table 2).

Experimental

A solution of 2-amino-3-phenylpropionic acid (2 mmol, 0.328 g) and potassium hydroxide (2 mmol, 0.112 g) in distilled water (15 ml) was slowly added to a solution of 3,5-dibromo-2-hydroxybenzaldehyde (2 mmol, 0.560 g) in methanol (20 ml). The mixture was stirred for 30 min at room temperature; the solution was then added to solid sodium borohydride (2 mmol, 0.076 g) and stirred for 2 h until the yellow solution had become colourless. This solution was slowly added to a solution of zinc nitrate (1 mmol, 0.297 g) in distilled water (10 ml). The mixture was stirred and refluxed for 2 h at room temperature. Phen (2 mmol, 0.396 g) was added and the reaction continued for a further 2 h. The solution was filtered and the filtrate was left to stand at room temperature. Colourless plates suitable for X-ray diffration were obtained in a yield of 69% (based on zinc nitrate).

Crystal data

$[Zn(C_{16}H_{13}Br_2NO_3)(C_{12}H_8N_2)]\cdot 2H_2O$
M_r = 708.72
Monoclinic, $P2_1/c$
a = 9.5929 (11) Å
b = 25.969 (3) Å
c = 11.9430 (13) Å
β = 107.101 (2)°
V = 2843.7 (6) Å^3
Z = 4
D_x = 1.655 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.72 mm^{-1}
T = 294 (2) K
Plate, colourless
0.34 × 0.28 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.299, T_{max} = 0.690
18930 measured reflections
6547 independent reflections
2965 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.165
S = 0.97
6547 reflections
352 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 3.5289P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −1.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O2	1.968 (4)	Zn1—N2	2.201 (5)
Zn1—N3	2.049 (5)	N1—C1	1.464 (7)
Zn1—O1	2.067 (4)	N1—C10	1.492 (7)
Zn1—N1	2.085 (5)		
O2—Zn1—N3	112.36 (18)	O1—Zn1—N1	80.90 (17)
O2—Zn1—O1	100.86 (18)	O2—Zn1—N2	94.83 (18)
N3—Zn1—O1	93.93 (18)	N3—Zn1—N2	78.83 (19)
O2—Zn1—N1	98.17 (18)	O1—Zn1—N2	164.26 (18)
N3—Zn1—N1	149.45 (19)	N1—Zn1—N2	98.17 (18)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O3^i	0.91	1.97	2.865 (7)	167
O1*W*—H1*WB*···O1	0.85	2.25	2.914 (9)	136
O2*W*—H2*WB*···O1*W*	0.86	2.11	2.958 (9)	169
O2*W*—H2*WA*···O2	0.85	1.89	2.712 (6)	163

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

Water H atoms were located in a difference Fourier map and were allowed to ride on the O atom, with $U_{iso}(H) = 1.2U_{eq}(O)$. All other H atoms were positioned geometrically and refined as riding, with C–H = 0.93–0.97 Å and N–H = 0.91 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The deepest residual density hole is located 1.09 Å from a Br atom.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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