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Dibromo{2,4-dichloro-6-[2-(diethylamino)-ethyliminomethyl]phenolato}zinc(II)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.018 Å
R factor = 0.066
wR factor = 0.130
Data-to-parameter ratio = 19.8

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

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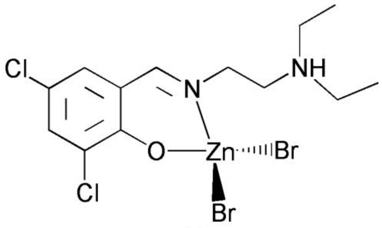
In the title mononuclear zinc(II) complex, [ZnBr$_2$(C$_{13}$H$_{18}$Cl$_2$N$_2$O)], there are two independent complex molecules in the asymmetric unit. Each Zn^{II} atom is four-coordinated by the imine N and phenolate O atoms of the Schiff base ligand, and by two Br atoms, forming a tetrahedral geometry. In the crystal structure, the molecules are linked through intermolecular N—H···O and C—H···Br hydrogen bonds, forming a three-dimensional network.

Comment

Zinc is the second most abundant transition metal in biology, and functions as the active site of hydrolytic enzymes, such as carboxypeptidase and carbonic anhydrase, where it is in a hard donor coordination of nitrogen and oxygen (Lipscomb & Sträter, 1996; Bertini *et al.*, 1994). Zinc has long been recognized as an important co-factor in biological molecules, either as a structural template in protein folding or as a Lewis acid catalyst that can readily adopt four-, five- or six-coordination (Harrison *et al.*, 2006; Tirosh *et al.*, 2005; Musie *et al.*, 2004; Vallee & Auld, 1993). Recent reports have suggested that zinc is able to play a catalytic role in the activation of thiols as nucleophiles at physiological pH (Wilker & Lippard, 1997; Myers *et al.*, 1993). We have reported the structures of a few transition metal complexes (Peng *et al.*, 2005; Peng, Yang & Zhou, 2006; Peng, Zhou & Yang, 2006). As a continuation of this work, we report here a new zinc(II) complex, (I), derived from 2,4-dichloro-6-[2-(diethylamino)ethyliminomethyl]-phenol.



(I)

The Zn atom in each of the two symmetry-independent complex molecules in (I) shows a tetrahedral geometry and is four-coordinated by the imine N and phenolate O atoms of the Schiff base ligand, and by two Br atoms (Fig. 1). As expected, the C8/C9/N2/C10–C13 unit adopts a zigzag geometry to minimize steric effects. Each amine N atom in the complex is protonated and does not coordinate to the metal ion. The Zn—O and Zn—N bond lengths (Table 1) are comparable to the values in other similar complexes (Peng, Zhou & Yang, 2006; Qiu, 2006; Tatar, Atakol & Arici, 2002; Tatar, Atakol & Ülkü, 2002; Ülkü *et al.*, 2000). The angles subtended at atom

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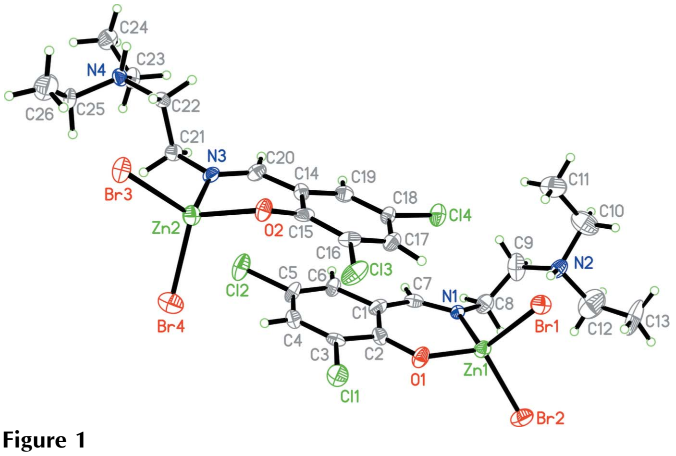

Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level.

Zn1 range from 96.8 (3) to 118.8 (2)°, and the angles subtended at Zn2 range from 93.5 (3) to 115.1 (2)° (Table 1), indicating distorted tetrahedral coordination for the metal ions.

In the crystal structure, the molecules are linked through intermolecular N—H···O and C—H···Br hydrogen bonds (Table 2), forming a three-dimensional network, as shown in Fig. 2.

Experimental

3,5-Dichlorosalicylaldehyde (0.5 mmol, 96.3 mg) and *N*,*N*-diethylethane-1,2-diamine (0.5 mmol, 58.2 mg) were stirred into 30 ml of methanol. After 1 h, $ZnBr_2$ (0.3 mmol, 67.5 mg) in methanol (10 ml) was added, and the stirring continued for a further 1 h. The filtrate was kept at room temperature for about two weeks, depositing yellow needle-like crystals of (I).

Crystal data

$[ZnBr_2(C_{13}H_{18}Cl_2N_2O)]$
$M_r = 514.38$
Monoclinic, *Pc*
$a = 7.687$ (1) Å
$b = 19.102$ (3) Å
$c = 13.841$ (2) Å
$\beta = 118.452$ (3)°
$V = 1786.9$ (4) Å^3
$Z = 4$
$D_x = 1.912$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 6.14$ mm^{-1}
$T = 298$ (2) K
Block cut from needle, light yellow
$0.12 \times 0.07 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.515$, $T_{max} = 0.695$
15186 measured reflections
7708 independent reflections
4594 reflections with $I > 2\sigma(I)$
$R_{int} = 0.075$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.130$
$S = 0.94$
7708 reflections
389 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}
Absolute structure: Flack (1983), 3653 Friedel pairs.
Flack parameter: −0.007 (13)

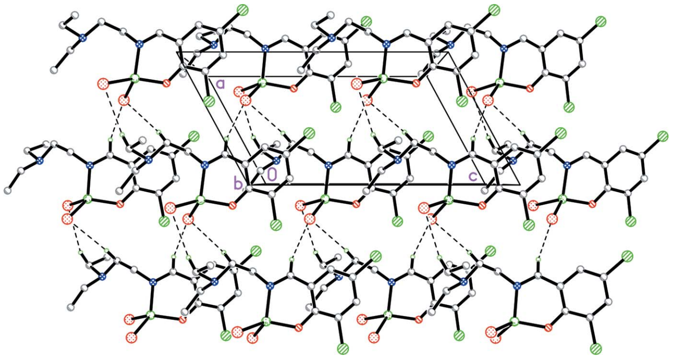

Figure 2
The crystal packing of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	1.913 (7)	Zn2—O2	1.972 (7)
Zn1—N1	2.009 (8)	Zn2—N3	2.017 (8)
Zn1—Br2	2.3616 (18)	Zn2—Br4	2.3575 (18)
Zn1—Br1	2.3982 (17)	Zn2—Br3	2.3649 (17)
O1—Zn1—N1	96.8 (3)	O2—Zn2—N3	93.5 (3)
O1—Zn1—Br2	118.8 (2)	O2—Zn2—Br4	110.6 (2)
N1—Zn1—Br2	107.9 (2)	N3—Zn2—Br4	114.3 (2)
O1—Zn1—Br1	111.0 (2)	O2—Zn2—Br3	115.1 (2)
N1—Zn1—Br1	114.2 (2)	N3—Zn2—Br3	109.6 (2)
Br2—Zn1—Br1	107.99 (7)	Br4—Zn2—Br3	112.53 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O2^{i}	0.90 (9)	2.20 (8)	2.960 (11)	142 (11)
N2—H2···Br1	0.90 (12)	2.48 (13)	3.373 (9)	170 (11)
C7—H7···Br2^{ii}	0.93	2.85	3.735 (8)	159
C23—H23*B*···Br3^{iii}	0.97	2.92	3.611 (8)	129
C25—H25*B*···Br3^{i}	0.97	2.79	3.613 (8)	143
C26—H26*A*···Br3	0.96	2.80	3.764 (17)	177

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

Atoms H2 and H4*A* were located in a difference Fourier map and were refined isotropically, with N—H distances restrained to 0.90 (1) Å. All other H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Bertini, I., Gray, H. B., Lippard, S. J. & Valentine, J. S. (1994). *Bioinorganic Chemistry*. Mills Valley, CA: University Science Books.

Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01). *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Harrison, W. T. A., Ramadevi, P. & Kumaresan, S. (2006). *Acta Cryst.* E**62**, m513–m515.

Lipscomb, W. N. & Sträter, N. (1996). *Chem. Rev.* **96**, 2375–2434.

Musie, G. T., Li, X. & Powell, D. R. (2004). *Acta Cryst.* E**60**, m471–m472.

Myers, L. C., Terranova, M. P., Frentz, A. E., Wagner, G. & Verdine, G. L. (1993). *Science*, **261**, 1164–1167.

Peng, S.-J., Yang, D.-W., Xiao, Z.-L., Wu, D.-X. & Cao, Z. (2005). *Z. Kristallogr. New Cryst. Struct.* **220**, 633–634.

Peng, S.-J., Yang, T. & Zhou, C.-S. (2006). *Acta Cryst.* E**62**, m235–m236.

Peng, S.-J., Zhou, C.-S. & Yang, T. (2006). *Acta Cryst.* E**62**, m1147–m1149.

Qiu, X.-Y. (2006). *Acta Cryst.* E**62**, m717–m718.

Tatar, L., Atakol, O. & Arici, C. (2002). *Acta Cryst.* E**58**, m154–m156.

Tatar, L., Atakol, O. & Ülkü, D. (2002). *Acta Cryst.* E**58**, m83–m85.

Tirosh, E., Maman, R. & Goldberg, I. (2005). *Acta Cryst.* E**61**, m751–m754.

Ülkü, D., Arici, C., Tahir, M. N. & Atakol, O. (2000). *Acta Cryst.* C**56**, 1298–1299.

Vallee, B. L. & Auld, D. S. (1993). *Acc. Chem. Res.* **26**, 543–551.

Wilker, J. J. & Lippard, S. J. (1997). *Inorg. Chem.* **36**, 969–978.