

# Dibromido[2-[2-(piperidinium-1-yl)ethyl- iminomethyl]phenolato}zinc(II) monohydrate

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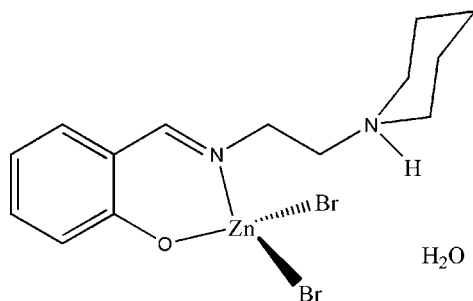
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.091; data-to-parameter ratio = 20.0.

The asymmetric unit of the title compound,  $[\text{ZnBr}_2(\text{C}_{14}\text{H}_{20}\text{N}_2\text{O})]\cdot\text{H}_2\text{O}$ , consists of a mononuclear Schiff base zinc(II) complex molecule and a solvent water molecule. The  $\text{Zn}^{\text{II}}$  atom is four-coordinated in an approximately tetrahedral geometry, binding to the imine N and phenolate O atoms of the neutral zwitterionic Schiff base ligand and to two terminal  $\text{Br}^-$  anions. In the crystal structure, molecules are linked through intermolecular  $\text{O}-\text{H}\cdots\text{Br}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains running along the  $b$  axis.

## Related literature

For the background to Schiff base zinc(II) complexes, see: Bhosekar *et al.* (2006); Chisholm *et al.* (2001); Jian *et al.* (2004); Lacroix *et al.* (1996); Tatar *et al.* (2002). For related structures, see: Ma, Gu *et al.* (2006); Ma, Lv *et al.* (2006); Peng & Hou (2006); Peng *et al.* (2006); Wei *et al.* (2007); Zhang *et al.* (2008); Zhu *et al.* (2007).



## Experimental

### Crystal data

$[\text{ZnBr}_2(\text{C}_{14}\text{H}_{20}\text{N}_2\text{O})]\cdot\text{H}_2\text{O}$   
 $M_r = 475.53$   
 Triclinic,  $P\bar{1}$   
 $a = 9.2997$  (18) Å

$b = 10.1776$  (17) Å  
 $c = 11.1667$  (18) Å  
 $\alpha = 71.510$  (2)°  
 $\beta = 71.215$  (2)°

$\gamma = 67.571$  (2)°  
 $V = 901.8$  (3) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation

$\mu = 5.80$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.20 \times 0.20 \times 0.18$  mm

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.390$ ,  $T_{\text{max}} = 0.422$   
 (expected range = 0.326–0.352)

5468 measured reflections  
 3983 independent reflections  
 2891 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.091$   
 $S = 1.02$   
 3983 reflections  
 199 parameters  
 4 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.72$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.55$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

Zn1—O1	1.936 (2)	Zn1—Br1	2.3417 (7)
Zn1—N1	2.024 (3)	Zn1—Br2	2.3991 (7)
O1—Zn1—N1	93.91 (11)	O1—Zn1—Br2	109.78 (8)
O1—Zn1—Br1	116.12 (8)	N1—Zn1—Br2	108.84 (9)
N1—Zn1—Br1	113.04 (8)	Br1—Zn1—Br2	113.42 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}$	0.90 (4)	1.89 (4)	2.777 (4)	169 (4)
$\text{O2}-\text{H2A}\cdots\text{Br2}^i$	0.85 (4)	2.57 (4)	3.399 (3)	165 (4)
$\text{O2}-\text{H2B}\cdots\text{O1}^{ii}$	0.86 (3)	1.93 (4)	2.762 (4)	165 (5)

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2508).

## References

- Bhosekar, G., Jess, I. & Näther, C. (2006). *Acta Cryst.* **E62**, m2073–m2074.  
 Bruker (2002). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Chisholm, M. H., Gallucci, J. C. & Zhen, H. (2001). *Inorg. Chem.* **40**, 5051–5054.  
 Jian, F., Li, C., Sun, P. & Xiao, H. (2004). *Acta Cryst.* **E60**, m1811–m1812.  
 Lacroix, P. G., Di Bella, S. & Ledoux, I. (1996). *Chem. Mater.* **8**, 541–545.

- Ma, J.-Y., Gu, S.-H., Guo, J.-W., Lv, B.-L. & Yin, W.-P. (2006). *Acta Cryst.* **E62**, m1437–m1438.
- Ma, J.-Y., Lv, B.-L., Gu, S.-H., Guo, J.-W. & Yin, W.-P. (2006). *Acta Cryst.* **E62**, m1322–m1323.
- Peng, S.-J. & Hou, H.-Y. (2006). *Acta Cryst.* **E62**, m2947–m2949.
- Peng, S.-J., Zhou, C.-S. & Yang, T. (2006). *Acta Cryst.* **E62**, m1413–m1415.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tatar, L., Atakol, O. & Ülkü, D. (2002). *Acta Cryst.* **E58**, m83–m85.
- Wei, Y.-J., Wang, F.-W. & Zhu, Q.-Y. (2007). *Acta Cryst.* **E63**, m654–m655.
- Zhang, D.-F., Zhou, M.-H. & Yuan, C.-J. (2008). *Acta Cryst.* **E64**, m825–m826.
- Zhu, Q.-Y., Wei, Y.-J. & Wang, F.-W. (2007). *Acta Cryst.* **E63**, m1431–m1432.

**supplementary materials**

*Acta Cryst.* (2008). E64, m859-m860 [ doi:10.1107/S1600536808015730 ]

## Dibromido{2-[2-(piperidinium-1-yl)ethyliminomethyl]phenolato}zinc(II) monohydrate

Y.-J. Wei, F.-W. Wang and Q.-Y. Zhu

### Comment

Zinc(II) complexes derived from Schiff base ligands have been studied extensively due to their interesting structures and wide applications (Lacroix *et al.*, 1996; Chisholm *et al.*, 2001; Jian *et al.*, 2004; Tatar *et al.*, 2002; Bhosekar *et al.*, 2006). Recently, we have reported two Schiff base zinc(II) complexes with bromide ligands (Wei *et al.*, 2007; Zhu *et al.*, 2007). As a continuation of our work on the structures of such complexes, we report herein the crystal structure of the new title complex, (I), which is isostructural with the zinc(II) complex with chloride ligands (Zhang *et al.*, 2008).

The tetrahedral coordination sphere of Zn<sup>II</sup> atom in (I) is formed by the imine N and phenolate O atoms of the Schiff base ligand and by two terminal Br<sup>-</sup> anions (Fig. 1). The coordinate bond distances (Table 1) are typical and comparable with the values in other similar zinc(II) complexes (Peng & Hou, 2006; Peng *et al.*, 2006; Ma, Gu *et al.*, 2006; Ma, Lv *et al.*, 2006). The O1—Zn1—N1 and O1—Zn1—Br1 bond angles deviate most from ideal tetrahedral geometry with values of 93.91 (11) and 116.12 (8)°, respectively. The other angles in the coordination sphere are in the range 108.84 (9)–113.42 (2)° (Table 1).

In the crystal structure of (I), molecules are linked through intermolecular O—H...Br and O—H...O hydrogen bonds (Table 2), forming chains running along the *b* axis (Fig. 2).

### Experimental

Compound (I) was obtained by stirring of salicylaldehyde (0.1 mmol, 12.2 mg), 2-piperidin-1-ylethylamine (0.1 mmol, 12.8 mg), and zinc(II) bromide (0.1 mmol, 22.5 mg) in methanol (20 ml) for 30 min at room temperature. The reaction mixture was filtered. Yellow block-shaped single crystals suitable for X-ray diffraction formed from the filtrate after one day.

### Refinement

Atoms H2, H2A and H2B were located in a difference Fourier map and refined isotropically, with the N—H, O—H, and H...H distances restrained to 0.90 (1), 0.85 (1), and 1.37 (2) Å, respectively. Other H atom positions were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, with  $U_{\text{iso}}(\text{H})$  values set at  $1.2U_{\text{eq}}(\text{C})$ .

### Figures

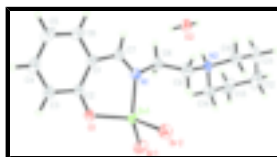


Fig. 1. The molecular structure of (I), shown with 30% probability displacement ellipsoids.

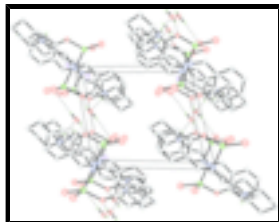


Fig. 2. Molecular packing of (I), viewed along the  $a$  axis. Intermolecular hydrogen bonds are shown as dashed lines.

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### Crystal data

$[\text{ZnBr}_2(\text{C}_{14}\text{H}_{20}\text{N}_2\text{O})]\cdot\text{H}_2\text{O}$

$M_r = 475.53$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 9.2997$  (18) Å

$b = 10.1776$  (17) Å

$c = 11.1667$  (18) Å

$\alpha = 71.510$  (2)°

$\beta = 71.215$  (2)°

$\gamma = 67.571$  (2)°

$V = 901.8$  (3) Å<sup>3</sup>

$Z = 2$

$F_{000} = 472$

$D_x = 1.751$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1797 reflections

$\theta = 2.2\text{--}25.4^\circ$

$\mu = 5.80$  mm<sup>-1</sup>

$T = 298$  (2) K

Block, yellow

$0.20 \times 0.20 \times 0.18$  mm

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\omega$  scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.390$ ,  $T_{\max} = 0.422$

5468 measured reflections

3983 independent reflections

2891 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -12 \rightarrow 9$

$k = -13 \rightarrow 12$

$l = -14 \rightarrow 14$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.091$

$S = 1.03$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 0.3508P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

3983 reflections  $\Delta\rho_{\max} = 0.72 \text{ e } \text{\AA}^{-3}$   
 199 parameters  $\Delta\rho_{\min} = -0.55 \text{ e } \text{\AA}^{-3}$   
 4 restraints Extinction correction: none  
 Primary atom site location: structure-invariant direct methods

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.82333 (5)	-0.20793 (4)	0.11443 (4)	0.03909 (13)
Br1	0.93736 (6)	-0.24561 (5)	0.28619 (4)	0.06059 (15)
Br2	0.56849 (5)	-0.24885 (5)	0.18395 (4)	0.05682 (14)
O1	0.9582 (3)	-0.3089 (3)	-0.0226 (2)	0.0451 (6)
O2	0.7190 (3)	0.4568 (3)	0.0481 (3)	0.0515 (7)
N1	0.8013 (4)	-0.0074 (3)	-0.0039 (3)	0.0406 (7)
N2	0.5644 (4)	0.2574 (3)	0.2120 (3)	0.0420 (7)
C1	0.8738 (4)	-0.1069 (4)	-0.1951 (3)	0.0378 (8)
C2	0.9415 (4)	-0.2555 (4)	-0.1434 (3)	0.0379 (8)
C3	0.9993 (5)	-0.3518 (4)	-0.2277 (4)	0.0451 (9)
H3	1.0451	-0.4508	-0.1960	0.054*
C4	0.9897 (5)	-0.3031 (5)	-0.3550 (4)	0.0555 (11)
H4	1.0283	-0.3696	-0.4077	0.067*
C5	0.9234 (6)	-0.1561 (5)	-0.4069 (4)	0.0594 (12)
H5	0.9176	-0.1236	-0.4935	0.071*
C6	0.8668 (5)	-0.0600 (5)	-0.3273 (4)	0.0513 (10)
H6	0.8225	0.0387	-0.3611	0.062*
C7	0.8178 (4)	0.0091 (4)	-0.1258 (4)	0.0409 (8)
H7	0.7915	0.1043	-0.1745	0.049*
C8	0.7581 (5)	0.1241 (4)	0.0443 (4)	0.0552 (11)
H8A	0.7560	0.2080	-0.0279	0.066*
H8B	0.8374	0.1140	0.0884	0.066*
C9	0.5982 (5)	0.1467 (5)	0.1357 (5)	0.0633 (12)
H9A	0.5928	0.0547	0.1952	0.076*
H9B	0.5166	0.1790	0.0870	0.076*
C10	0.3889 (5)	0.3298 (5)	0.2454 (5)	0.0594 (11)
H10A	0.3359	0.2573	0.2962	0.071*

## supplementary materials

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H10B	0.3502	0.3761	0.1664	0.071*
C11	0.3486 (6)	0.4435 (6)	0.3223 (6)	0.0836 (17)
H11A	0.3915	0.5216	0.2677	0.100*
H11B	0.2334	0.4848	0.3476	0.100*
C12	0.4153 (7)	0.3795 (7)	0.4409 (6)	0.094 (2)
H12A	0.3925	0.4556	0.4853	0.113*
H12B	0.3651	0.3077	0.4997	0.113*
C13	0.5908 (6)	0.3095 (7)	0.4042 (5)	0.0835 (17)
H13A	0.6325	0.2653	0.4819	0.100*
H13B	0.6414	0.3830	0.3510	0.100*
C14	0.6302 (6)	0.1951 (5)	0.3306 (5)	0.0701 (13)
H14A	0.7453	0.1528	0.3059	0.084*
H14B	0.5862	0.1179	0.3862	0.084*
H2B	0.8210 (13)	0.423 (5)	0.029 (5)	0.080*
H2A	0.699 (4)	0.524 (4)	0.086 (4)	0.080*
H2	0.611 (5)	0.327 (4)	0.168 (4)	0.080*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Zn1	0.0453 (3)	0.0381 (2)	0.0358 (2)	-0.01543 (19)	-0.00516 (18)	-0.01193 (18)
Br1	0.0684 (3)	0.0803 (3)	0.0459 (2)	-0.0337 (3)	-0.0178 (2)	-0.0131 (2)
Br2	0.0462 (3)	0.0559 (3)	0.0720 (3)	-0.0217 (2)	-0.0069 (2)	-0.0184 (2)
O1	0.0509 (16)	0.0409 (14)	0.0377 (14)	-0.0066 (12)	-0.0070 (12)	-0.0138 (11)
O2	0.0503 (17)	0.0479 (16)	0.0572 (18)	-0.0181 (14)	-0.0049 (14)	-0.0170 (13)
N1	0.0497 (19)	0.0346 (16)	0.0412 (17)	-0.0198 (14)	-0.0023 (14)	-0.0134 (13)
N2	0.0438 (19)	0.0411 (18)	0.0415 (17)	-0.0163 (15)	-0.0001 (14)	-0.0157 (14)
C1	0.041 (2)	0.041 (2)	0.0329 (18)	-0.0188 (17)	-0.0024 (15)	-0.0099 (15)
C2	0.033 (2)	0.044 (2)	0.0387 (19)	-0.0167 (16)	-0.0012 (15)	-0.0128 (16)
C3	0.046 (2)	0.044 (2)	0.049 (2)	-0.0169 (18)	-0.0028 (18)	-0.0200 (18)
C4	0.059 (3)	0.071 (3)	0.049 (2)	-0.030 (2)	0.001 (2)	-0.031 (2)
C5	0.071 (3)	0.081 (3)	0.034 (2)	-0.040 (3)	-0.002 (2)	-0.013 (2)
C6	0.057 (3)	0.054 (2)	0.042 (2)	-0.023 (2)	-0.0084 (19)	-0.0033 (18)
C7	0.039 (2)	0.0348 (19)	0.047 (2)	-0.0154 (16)	-0.0043 (17)	-0.0070 (16)
C8	0.064 (3)	0.043 (2)	0.059 (3)	-0.022 (2)	0.001 (2)	-0.0222 (19)
C9	0.061 (3)	0.063 (3)	0.076 (3)	-0.026 (2)	0.001 (2)	-0.037 (2)
C10	0.046 (3)	0.062 (3)	0.074 (3)	-0.018 (2)	-0.011 (2)	-0.022 (2)
C11	0.040 (3)	0.071 (3)	0.140 (5)	-0.011 (2)	0.002 (3)	-0.056 (4)
C12	0.083 (4)	0.141 (5)	0.084 (4)	-0.056 (4)	0.023 (3)	-0.073 (4)
C13	0.077 (4)	0.140 (5)	0.050 (3)	-0.045 (4)	-0.013 (3)	-0.030 (3)
C14	0.055 (3)	0.074 (3)	0.065 (3)	-0.011 (3)	-0.019 (2)	-0.002 (3)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Zn1—O1	1.936 (2)	C5—H5	0.9300
Zn1—N1	2.024 (3)	C6—H6	0.9300
Zn1—Br1	2.3417 (7)	C7—H7	0.9300
Zn1—Br2	2.3991 (7)	C8—C9	1.488 (6)
O1—C2	1.321 (4)	C8—H8A	0.9700

O2—H2B	0.86 (3)	C8—H8B	0.9700
O2—H2A	0.85 (4)	C9—H9A	0.9700
N1—C7	1.282 (5)	C9—H9B	0.9700
N1—C8	1.467 (4)	C10—C11	1.519 (6)
N2—C10	1.487 (5)	C10—H10A	0.9700
N2—C14	1.491 (5)	C10—H10B	0.9700
N2—C9	1.503 (5)	C11—C12	1.496 (8)
N2—H2	0.90 (4)	C11—H11A	0.9700
C1—C2	1.404 (5)	C11—H11B	0.9700
C1—C6	1.415 (5)	C12—C13	1.485 (7)
C1—C7	1.454 (5)	C12—H12A	0.9700
C2—C3	1.411 (5)	C12—H12B	0.9700
C3—C4	1.370 (6)	C13—C14	1.502 (7)
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.389 (6)	C13—H13B	0.9700
C4—H4	0.9300	C14—H14A	0.9700
C5—C6	1.371 (6)	C14—H14B	0.9700
O1—Zn1—N1	93.91 (11)	C9—C8—H8A	109.7
O1—Zn1—Br1	116.12 (8)	N1—C8—H8B	109.7
N1—Zn1—Br1	113.04 (8)	C9—C8—H8B	109.7
O1—Zn1—Br2	109.78 (8)	H8A—C8—H8B	108.2
N1—Zn1—Br2	108.84 (9)	C8—C9—N2	112.2 (3)
Br1—Zn1—Br2	113.42 (2)	C8—C9—H9A	109.2
C2—O1—Zn1	121.6 (2)	N2—C9—H9A	109.2
H2B—O2—H2A	105 (2)	C8—C9—H9B	109.2
C7—N1—C8	117.3 (3)	N2—C9—H9B	109.2
C7—N1—Zn1	119.8 (2)	H9A—C9—H9B	107.9
C8—N1—Zn1	122.8 (2)	N2—C10—C11	110.8 (3)
C10—N2—C14	111.0 (3)	N2—C10—H10A	109.5
C10—N2—C9	108.7 (3)	C11—C10—H10A	109.5
C14—N2—C9	113.6 (3)	N2—C10—H10B	109.5
C10—N2—H2	108 (3)	C11—C10—H10B	109.5
C14—N2—H2	102 (3)	H10A—C10—H10B	108.1
C9—N2—H2	113 (3)	C12—C11—C10	111.6 (4)
C2—C1—C6	119.5 (3)	C12—C11—H11A	109.3
C2—C1—C7	125.2 (3)	C10—C11—H11A	109.3
C6—C1—C7	115.1 (3)	C12—C11—H11B	109.3
O1—C2—C1	123.9 (3)	C10—C11—H11B	109.3
O1—C2—C3	118.6 (3)	H11A—C11—H11B	108.0
C1—C2—C3	117.4 (3)	C13—C12—C11	110.0 (4)
C4—C3—C2	121.6 (4)	C13—C12—H12A	109.7
C4—C3—H3	119.2	C11—C12—H12A	109.7
C2—C3—H3	119.2	C13—C12—H12B	109.7
C3—C4—C5	121.2 (4)	C11—C12—H12B	109.7
C3—C4—H4	119.4	H12A—C12—H12B	108.2
C5—C4—H4	119.4	C12—C13—C14	110.9 (4)
C6—C5—C4	118.4 (4)	C12—C13—H13A	109.5
C6—C5—H5	120.8	C14—C13—H13A	109.5
C4—C5—H5	120.8	C12—C13—H13B	109.5



## supplementary materials

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C5—C6—C1	121.8 (4)	C14—C13—H13B	109.5
C5—C6—H6	119.1	H13A—C13—H13B	108.0
C1—C6—H6	119.1	N2—C14—C13	111.4 (4)
N1—C7—C1	126.2 (3)	N2—C14—H14A	109.4
N1—C7—H7	116.9	C13—C14—H14A	109.4
C1—C7—H7	116.9	N2—C14—H14B	109.4
N1—C8—C9	109.9 (3)	C13—C14—H14B	109.4
N1—C8—H8A	109.7	H14A—C14—H14B	108.0

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 $\cdots$ O2	0.90 (4)	1.89 (4)	2.777 (4)	169 (4)
O2—H2A $\cdots$ Br2 <sup>i</sup>	0.85 (4)	2.57 (4)	3.399 (3)	165 (4)
O2—H2B $\cdots$ O1 <sup>ii</sup>	0.86 (3)	1.93 (4)	2.762 (4)	165 (5)

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

Fig. 1

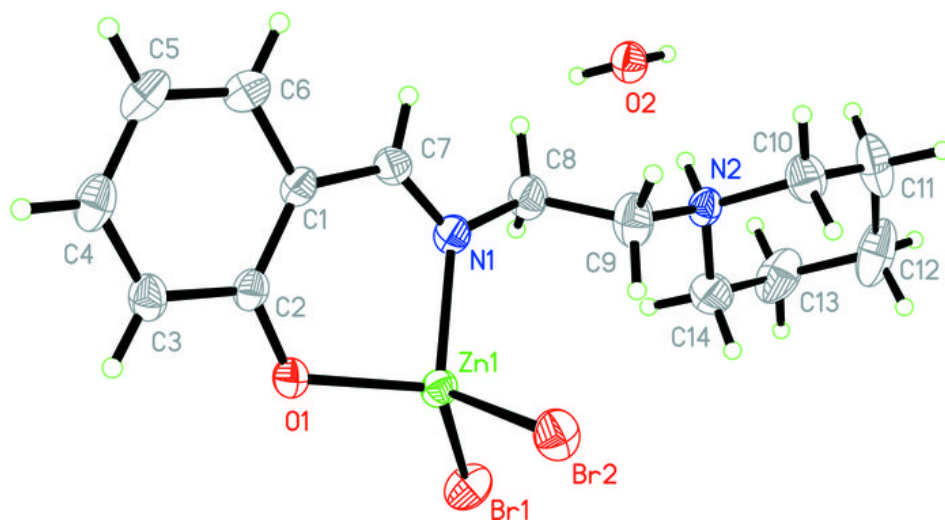


Fig. 2

