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

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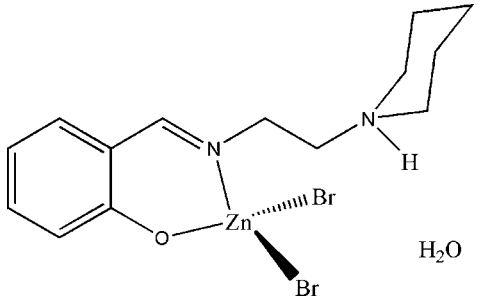
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.038; wR factor = 0.091; data-to-parameter ratio = 20.0.

The asymmetric unit of the title compound, $[ZnBr_2(C_{14}H_{20}N_2O)]\cdot H_2O$, consists of a mononuclear Schiff base zinc(II) complex molecule and a solvent water molecule. The Zn^{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 Br^- anions. In the crystal structure, molecules are linked through intermolecular O—H···Br and O—H···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

$[ZnBr_2(C_{14}H_{20}N_2O)]\cdot H_2O$
M_r = 475.53
Triclinic, $P\overline{1}$
a = 9.2997 (18) Å
b = 10.1776 (17) Å
c = 11.1667 (18) Å
α = 71.510 (2)°
β = 71.215 (2)°
γ = 67.571 (2)°
V = 901.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.80 mm^{-1}
T = 298 (2) K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.390, T_{max} = 0.422
(expected range = 0.326–0.352)
5468 measured reflections
3983 independent reflections
2891 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.90 (4)	1.89 (4)	2.777 (4)	169 (4)
O2—H2A···Br2^{i}	0.85 (4)	2.57 (4)	3.399 (3)	165 (4)
O2—H2B···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).

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supporting information

Acta Cryst. (2008). E**64**, m859–m860 [doi:10.1107/S1600536808015730]

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

Yi-Jun Wei, Feng-Wu Wang and Qi-Yong Zhu

S1. 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^{II} atom in (I) is formed by the imine N and phenolate O atoms of the Schiff base ligand and by two terminal Br^- 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).

S2. 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 fitered. Yellow block-shaped single crystals suitable for X-ray diffraction formed from the filtrate after one day.

S3. 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_{iso}(H) values set at 1.2U_{eq}(C).

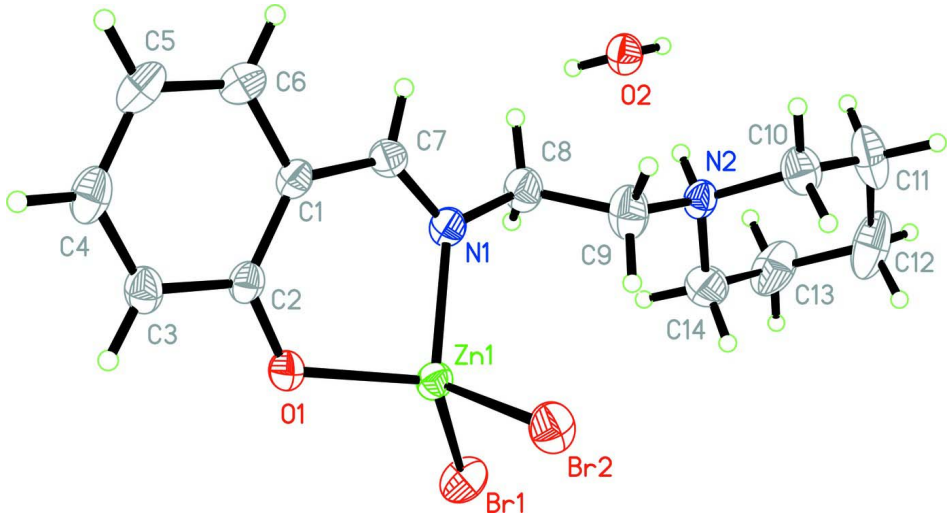

Figure 1

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

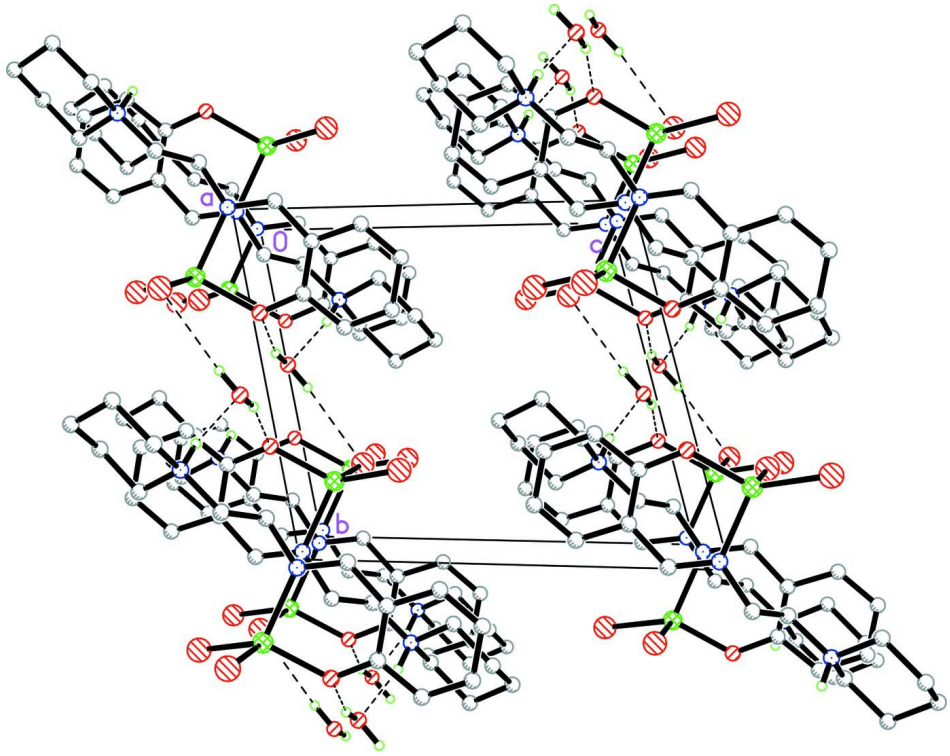

Figure 2

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

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Crystal data

$[ZnBr_2(C_{14}H_{20}N_2O)]\cdot H_2O$
$M_r = 475.53$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.2997\ (18)$ Å
$b = 10.1776\ (17)$ Å

c = 11.1667 (18) Å
α = 71.510 (2)°
β = 71.215 (2)°
γ = 67.571 (2)°
V = 901.8 (3) Å^3
Z = 2
F(000) = 472
D_x = 1.751 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1797 reflections
θ = 2.2–25.4°
μ = 5.80 mm^{-1}
T = 298 K
Block, yellow
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω 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_{int} = 0.016
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −12→9
k = −13→12
l = −14→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
3983 reflections
199 parameters
4 restraints
Primary atom site location: structure-invariant direct methods

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{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*
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 (Å²)

	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 (Å, º)

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
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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.90 (4)	1.89 (4)	2.777 (4)	169 (4)
O2—H2*A*···Br2^{i}	0.85 (4)	2.57 (4)	3.399 (3)	165 (4)
O2—H2*B*···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*.