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Dibromido{2-morpholino-*N*-[1-(2-pyridyl)ethylidene]ethanamine-κ^3N,N',N''}zinc(II)

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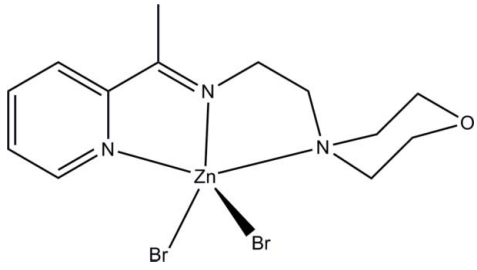
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.013 Å; R factor = 0.065; wR factor = 0.177; data-to-parameter ratio = 18.7.

In the title complex, $[ZnBr_2(C_{13}H_{19}N_3O)]$, the Zn^{II} atom is five-coordinated by the three N-donor atoms of the Schiff base ligand and by two Br atoms in a distorted square-pyramidal geometry. The morpholine ring adopts a chair conformation.

Related literature

For background to Schiff base complexes, see: Dhar & Chakravarty (2003); Das *et al.* (2006); Nayak *et al.* (2006). For the crystal structures of similar Schiff base–zinc(II) complexes, see: Wang (2010); Zhu *et al.* (2007); Li & Zhang (2004); Zhu & Yang (2008).



Experimental

Crystal data

$[ZnBr_2(C_{13}H_{19}N_3O)]$
M_r = 458.50
Monoclinic, $P2_1/n$
a = 9.831 (4) Å
b = 13.985 (6) Å
c = 12.183 (5) Å
β = 106.626 (4)°
V = 1604.9 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.51 mm^{-1}
T = 298 K
0.35 × 0.32 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.209, T_{max} = 0.230
12426 measured reflections
3411 independent reflections
2159 reflections with $I > 2\sigma(I)$
R_{int} = 0.108

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.177
S = 1.05
3411 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.35 e Å^{-3}
$\Delta\rho_{min}$ = −1.26 e Å^{-3}

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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* and *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2250).

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supporting information

Acta Cryst. (2011). E**67**, m261 [doi:10.1107/S1600536811002753]

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Yan-Wei Ding, Xiao-Ling Wang and Li-Li Ni

S1. Comment

In the last few years, considerable attention has focused on the preparation and properties of Schiff base complexes (Dhar & Chakravarty, 2003; Das *et al.*, 2006; Nayak *et al.*, 2006). Herein we report on the crystal structure of a new Schiff base zinc(II) complex.

The molecular structure of the title mononuclear zinc(II) complex is shown in Fig. 1. The Zn atom is five-coordinated by the three N-donor atoms of the Schiff base ligand, and by two Br atoms in a distorted square-pyramidal geometry. All the coordinate bond lengths are within normal values and are comparable to those in similar zinc(II) complexes with Schiff bases (Wang, 2010; Zhu *et al.*, 2007; Li & Zhang, 2004; Zhu & Yang, 2008). As expected, the morpholine ring adopts a chair conformation.

S2. Experimental

The title complex was prepared by the reaction of 2-acetylpyridine (0.20 g, 1.65 mmol), 4-(2-aminoethyl)morpholine (0.21 g, 1.65 mmol), and zinc bromide (0.37 g, 1.65 mmol) in methanol at ambient temperature. Colourless block-like single crytals were formed by slow evaporation of the solution in air.

S3. Refinement

The C-bound H-atoms were included in calculated positions and treated as riding atoms: C-H = 0.93, 0.97 and 0.96 Å for CH, CH_2 and CH_3 H-atoms, respectively, with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.2 for CH and CH_2 H-atoms, and 1.5 for CH_3 H-atoms. The highest residual density peak, 1.35 e Å^{-2}, is 0.58 Å from atom Zn1, while the deepest residual density hole, -1.26 e Å^{-2}, is 0.62 Å from atom Br1.

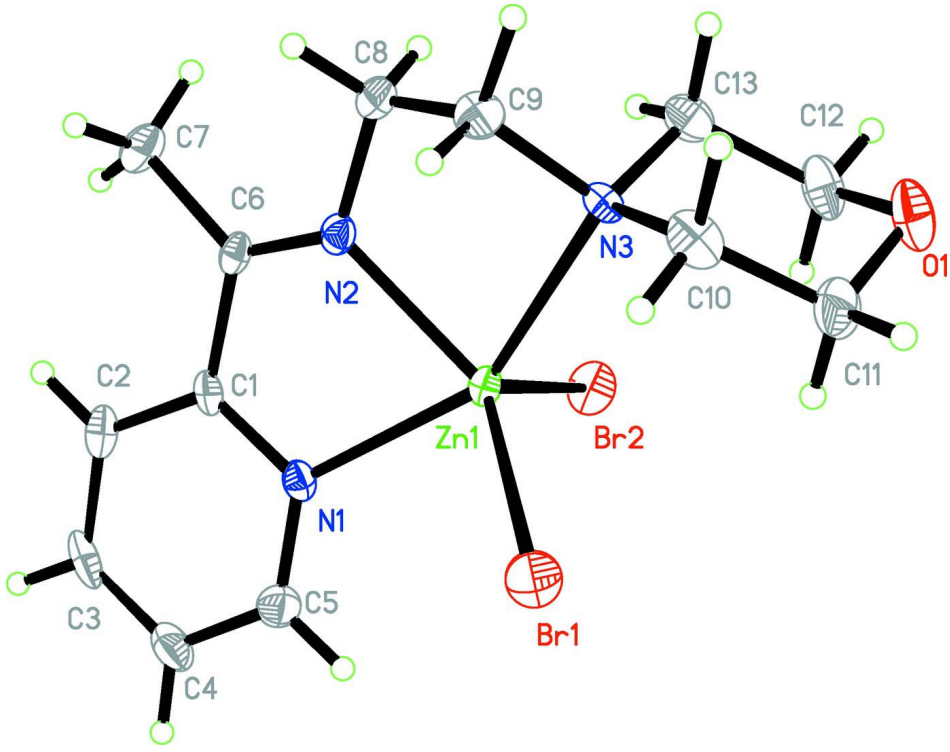

Figure 1

Molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

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Crystal data

$[ZnBr_2(C_{13}H_{19}N_3O)]$
$M_r = 458.50$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.831\ (4)$ Å
$b = 13.985\ (6)$ Å
$c = 12.183\ (5)$ Å
$\beta = 106.626\ (4)°$
$V = 1604.9\ (11)$ Å^3
$Z = 4$
$F(000) = 904$
$D_x = 1.898$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1887 reflections
$\theta = 2.3–25.1°$
$\mu = 6.51$ mm^{-1}
$T = 298$ K
Block, colourless
0.35 × 0.32 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.209$, $T_{max} = 0.230$
12426 measured reflections
3411 independent reflections
2159 reflections with $I > 2\sigma(I)$
$R_{int} = 0.108$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
$h = -12\rightarrow12$
$k = -17\rightarrow17$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.177$
$S = 1.05$
3411 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 5.9893P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.35$ e Å^{-3}
$\Delta\rho_{min} = -1.26$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.27265 (9)	0.91109 (6)	0.83908 (8)	0.0207 (3)
Br1	0.45896 (10)	0.79761 (8)	0.87089 (9)	0.0448 (3)
Br2	0.04314 (10)	0.86043 (7)	0.72941 (9)	0.0418 (3)
O1	0.3163 (7)	0.9365 (6)	0.4853 (6)	0.0490 (19)
N1	0.2385 (7)	0.8835 (5)	1.0100 (6)	0.0247 (16)
N2	0.2537 (7)	1.0444 (5)	0.9095 (6)	0.0252 (16)
N3	0.3418 (6)	1.0118 (5)	0.7105 (5)	0.0227 (15)
C1	0.1995 (7)	0.9617 (6)	1.0565 (6)	0.0210 (18)
C2	0.1623 (9)	0.9559 (7)	1.1573 (7)	0.033 (2)
H2	0.1349	1.0105	1.1889	0.040*
C3	0.1660 (11)	0.8680 (8)	1.2116 (8)	0.044 (3)
H3	0.1394	0.8630	1.2788	0.053*
C4	0.2089 (10)	0.7900 (8)	1.1649 (8)	0.042 (3)
H4	0.2155	0.7307	1.2006	0.051*
C5	0.2422 (10)	0.8003 (7)	1.0642 (8)	0.036 (2)
H5	0.2690	0.7460	1.0314	0.043*
C6	0.2071 (8)	1.0522 (6)	0.9937 (7)	0.0251 (19)
C7	0.1620 (9)	1.1447 (6)	1.0362 (8)	0.036 (2)
H7A	0.2439	1.1770	1.0840	0.054*
H7B	0.0967	1.1315	1.0797	0.054*
H7C	0.1167	1.1847	0.9721	0.054*
C8	0.2824 (10)	1.1260 (6)	0.8452 (8)	0.036 (2)
H8A	0.3204	1.1785	0.8969	0.043*
H8B	0.1954	1.1472	0.7902	0.043*
C9	0.3870 (10)	1.0959 (6)	0.7849 (8)	0.037 (2)

H9A	0.4031	1.1489	0.7388	0.045*
H9B	0.4764	1.0815	0.8414	0.045*
C10	0.4615 (9)	0.9792 (7)	0.6732 (8)	0.038 (2)
H10A	0.5339	0.9538	0.7384	0.045*
H10B	0.5020	1.0331	0.6434	0.045*
C11	0.4195 (12)	0.9037 (7)	0.5826 (9)	0.046 (3)
H11A	0.5026	0.8835	0.5610	0.055*
H11B	0.3829	0.8487	0.6135	0.055*
C12	0.1956 (11)	0.9629 (8)	0.5184 (8)	0.048 (3)
H12A	0.1594	0.9073	0.5485	0.057*
H12B	0.1223	0.9850	0.4516	0.057*
C13	0.2283 (9)	1.0409 (7)	0.6081 (8)	0.036 (2)
H13A	0.2573	1.0982	0.5759	0.043*
H13B	0.1432	1.0558	0.6298	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0158 (5)	0.0246 (5)	0.0212 (5)	−0.0006 (4)	0.0046 (4)	−0.0044 (4)
Br1	0.0322 (6)	0.0540 (7)	0.0475 (7)	0.0094 (5)	0.0102 (5)	0.0007 (5)
Br2	0.0264 (5)	0.0500 (6)	0.0486 (7)	−0.0049 (4)	0.0101 (4)	−0.0081 (5)
O1	0.038 (4)	0.083 (5)	0.030 (4)	0.004 (4)	0.016 (3)	−0.002 (4)
N1	0.022 (4)	0.032 (4)	0.019 (4)	0.004 (3)	0.004 (3)	−0.001 (3)
N2	0.024 (4)	0.027 (4)	0.022 (4)	0.000 (3)	0.001 (3)	−0.004 (3)
N3	0.018 (3)	0.028 (4)	0.020 (4)	−0.003 (3)	0.004 (3)	0.005 (3)
C1	0.007 (4)	0.037 (5)	0.015 (4)	0.000 (3)	−0.003 (3)	−0.005 (3)
C2	0.025 (5)	0.049 (6)	0.027 (5)	−0.005 (4)	0.007 (4)	−0.015 (4)
C3	0.046 (6)	0.070 (8)	0.014 (5)	−0.009 (5)	0.006 (4)	0.002 (5)
C4	0.052 (6)	0.049 (6)	0.025 (5)	−0.006 (5)	0.010 (5)	0.009 (5)
C5	0.035 (5)	0.034 (5)	0.036 (6)	0.006 (4)	0.007 (4)	0.000 (4)
C6	0.021 (4)	0.028 (4)	0.023 (5)	−0.003 (3)	0.002 (4)	−0.010 (4)
C7	0.028 (5)	0.034 (5)	0.047 (6)	0.010 (4)	0.014 (4)	−0.012 (4)
C8	0.051 (6)	0.026 (5)	0.031 (5)	−0.008 (4)	0.012 (5)	−0.006 (4)
C9	0.040 (6)	0.032 (5)	0.037 (6)	−0.023 (4)	0.006 (4)	−0.002 (4)
C10	0.020 (5)	0.055 (6)	0.041 (6)	−0.003 (4)	0.011 (4)	0.008 (5)
C11	0.051 (6)	0.055 (7)	0.041 (6)	0.007 (5)	0.027 (5)	−0.003 (5)
C12	0.043 (6)	0.072 (8)	0.023 (5)	−0.005 (5)	0.002 (5)	−0.003 (5)
C13	0.030 (5)	0.042 (6)	0.035 (5)	0.002 (4)	0.007 (4)	0.008 (4)

Geometric parameters (Å, º)

Zn1—N2	2.083 (7)	C4—H4	0.9300
Zn1—N1	2.235 (7)	C5—H5	0.9300
Zn1—N3	2.347 (6)	C6—C7	1.507 (11)
Zn1—Br1	2.3701 (15)	C7—H7A	0.9600
Zn1—Br2	2.3775 (15)	C7—H7B	0.9600
O1—C11	1.398 (12)	C7—H7C	0.9600
O1—C12	1.407 (12)	C8—C9	1.485 (13)

N1—C5	1.334 (11)	C8—H8A	0.9700
N1—C1	1.337 (10)	C8—H8B	0.9700
N2—C6	1.241 (11)	C9—H9A	0.9700
N2—C8	1.457 (11)	C9—H9B	0.9700
N3—C10	1.451 (11)	C10—C11	1.496 (13)
N3—C13	1.474 (10)	C10—H10A	0.9700
N3—C9	1.474 (10)	C10—H10B	0.9700
C1—C2	1.380 (12)	C11—H11A	0.9700
C1—C6	1.492 (11)	C11—H11B	0.9700
C2—C3	1.391 (14)	C12—C13	1.512 (13)
C2—H2	0.9300	C12—H12A	0.9700
C3—C4	1.352 (14)	C12—H12B	0.9700
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.365 (13)	C13—H13B	0.9700

N2—Zn1—N1	73.5 (3)	C6—C7—H7B	109.5
N2—Zn1—N3	79.4 (3)	H7A—C7—H7B	109.5
N1—Zn1—N3	151.0 (2)	C6—C7—H7C	109.5
N2—Zn1—Br1	133.48 (18)	H7A—C7—H7C	109.5
N1—Zn1—Br1	92.70 (17)	H7B—C7—H7C	109.5
N3—Zn1—Br1	98.80 (16)	N2—C8—C9	108.2 (7)
N2—Zn1—Br2	108.40 (19)	N2—C8—H8A	110.1
N1—Zn1—Br2	95.80 (18)	C9—C8—H8A	110.1
N3—Zn1—Br2	102.32 (16)	N2—C8—H8B	110.1
Br1—Zn1—Br2	117.20 (6)	C9—C8—H8B	110.1
C11—O1—C12	108.1 (7)	H8A—C8—H8B	108.4
C5—N1—C1	118.3 (8)	N3—C9—C8	113.6 (7)
C5—N1—Zn1	128.4 (6)	N3—C9—H9A	108.8
C1—N1—Zn1	113.1 (5)	C8—C9—H9A	108.8
C6—N2—C8	123.3 (7)	N3—C9—H9B	108.8
C6—N2—Zn1	121.2 (6)	C8—C9—H9B	108.8
C8—N2—Zn1	115.2 (5)	H9A—C9—H9B	107.7
C10—N3—C13	107.9 (7)	N3—C10—C11	112.0 (7)
C10—N3—C9	108.4 (7)	N3—C10—H10A	109.2
C13—N3—C9	108.8 (7)	C11—C10—H10A	109.2
C10—N3—Zn1	115.9 (5)	N3—C10—H10B	109.2
C13—N3—Zn1	115.9 (5)	C11—C10—H10B	109.2
C9—N3—Zn1	99.3 (5)	H10A—C10—H10B	107.9
N1—C1—C2	120.6 (8)	O1—C11—C10	112.0 (8)
N1—C1—C6	114.4 (7)	O1—C11—H11A	109.2
C2—C1—C6	124.9 (8)	C10—C11—H11A	109.2
C1—C2—C3	119.9 (9)	O1—C11—H11B	109.2
C1—C2—H2	120.1	C10—C11—H11B	109.2
C3—C2—H2	120.1	H11A—C11—H11B	107.9
C4—C3—C2	118.7 (9)	O1—C12—C13	112.0 (8)
C4—C3—H3	120.6	O1—C12—H12A	109.2
C2—C3—H3	120.6	C13—C12—H12A	109.2
C3—C4—C5	118.5 (9)	O1—C12—H12B	109.2

C3—C4—H4	120.7	C13—C12—H12B	109.2
C5—C4—H4	120.7	H12A—C12—H12B	107.9
N1—C5—C4	123.9 (9)	N3—C13—C12	111.5 (8)
N1—C5—H5	118.1	N3—C13—H13A	109.3
C4—C5—H5	118.1	C12—C13—H13A	109.3
N2—C6—C1	115.7 (7)	N3—C13—H13B	109.3
N2—C6—C7	125.1 (8)	C12—C13—H13B	109.3
C1—C6—C7	119.3 (8)	H13A—C13—H13B	108.0
C6—C7—H7A	109.5		