

# {2-Ethoxy-6-[2-(piperidinium-1-yl)ethyl- iminomethyl]phenolato}diiodidozinc(II)

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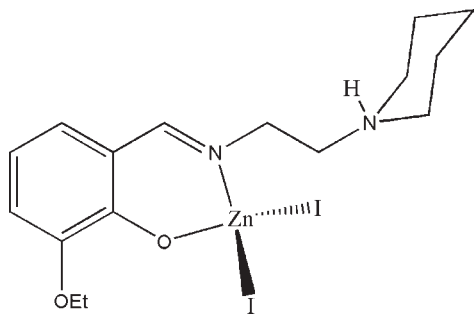
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 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.008$  Å;  $R$  factor = 0.028;  $wR$  factor = 0.068; data-to-parameter ratio = 20.9.

The title Schiff base complex,  $[\text{ZnI}_2(\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_2)]$ , is a mononuclear zinc(II) compound. The Zn atom is four-coordinated in a distorted tetrahedral geometry by one phenolate O atom and one imine N atom of the Schiff base ligand and by two iodide ions. In the crystal structure, molecules are linked through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains running along the  $a$  axis.

## Related literature

For background to the applications of Schiff bases, see: Averseng *et al.* (2001); Patra *et al.* (2002); Chen *et al.* (2003); Ruck & Jacobsen (2002). For the structures of related Schiff base zinc complexes, see: Wei *et al.* (2007); Zhu, Yang *et al.* (2009); Zhu, Yin, Li *et al.* (2009); Zhu, Yin, Yang *et al.* (2009).



## Experimental

### Crystal data

 $[\text{ZnI}_2(\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_2)]$ 
 $M_r = 595.54$ 

 Orthorhombic,  $Pna2_1$ 
 $a = 13.5934$  (10) Å

 $b = 10.2381$  (8) Å

 $c = 14.7871$  (11) Å

 $V = 2057.9$  (3) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 4.20$  mm<sup>-1</sup>
 $T = 298$  K

 $0.18 \times 0.17 \times 0.17$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer

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

 $T_{\min} = 0.518$ ,  $T_{\max} = 0.535$ 

11751 measured reflections

4438 independent reflections

 3763 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.029$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.028$ 
 $wR(F^2) = 0.068$ 
 $S = 1.02$ 

4438 reflections

212 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.58$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.88$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

2111 Friedel pairs

Flack parameter: 0.02 (2)

**Table 1**

Selected bond lengths (Å).

Zn1—O1	1.950 (2)	Zn1—I1	2.5448 (9)
Zn1—N1	2.021 (3)	Zn1—I2	2.5651 (9)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.90 (4)	2.61 (5)	3.237 (5)	127 (4)
$\text{N2}-\text{H2}\cdots\text{O1}^i$	0.90 (4)	2.01 (5)	2.867 (5)	159 (5)

 Symmetry code: (i)  $x + \frac{1}{2}, -y + \frac{3}{2}, z$ .

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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2684).

## References

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**supplementary materials**

*Acta Cryst.* (2009). E65, m1611 [ doi:10.1107/S1600536809048491 ]

## {2-Ethoxy-6-[2-(piperidinium-1-yl)ethyliminomethyl]phenolato}diiodidozinc(II)

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### Comment

Schiff bases are versatile ligands for the preparation in the preparation of metal complexes (Averseng *et al.*, 2001; Patra *et al.*, 2002; Chen *et al.*, 2003; Ruck & Jacobsen, 2002). In this paper, the new zinc(II) title complex with the Schiff base ligand 2-ethoxy-6-[(2-piperidin-1-ylethylimino)methyl]phenol is reported.

In the title complex, Fig. 1, the Zn atom is four-coordinated by one phenolate O and one imine N atoms of the Schiff base ligand, and by two iodide atoms, forming a tetrahedral coordination. The coordinate bond lengths (Table 1) and angles are comparable to those of similar zinc complexes (Wei *et al.*, 2007; Zhu, Yang *et al.*, 2009; Zhu, Yin, Li *et al.*, 2009; Zhu, Yin, Yang *et al.*, 2009).

In the crystal structure, molecules are linked through intermolecular N—H...O hydrogen bonds (Table 2), forming chains running along the *a* axis (Fig. 2).

### Experimental

2-Ethoxysalicylaldehyde (1.0 mmol, 166 mg), 2-piperidin-1-ylethylamine (1.0 mmol, 128 mg), and ZnI<sub>2</sub> (1.0 mmol, 319 mg) were mixed in a methanol solution (50 ml). The mixture was stirred at reflux for 30 min to give a colourless solution. The solution was left in air for a few days, yielding colourless block-shaped crystals.

### Refinement

The H2 atom was located from a difference Fourier map and refined isotropically, with  $U_{\text{iso}}$  restrained to 0.08 Å<sup>2</sup>. Other H atoms were constrained to ideal geometries, with  $d(\text{C}-\text{H}) = 0.93\text{--}0.97$  Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ,  $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ .

### Figures

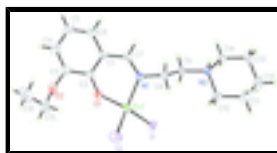


Fig. 1. The molecular structure of the title compound, with 30% displacement ellipsoids for non-hydrogen atoms.

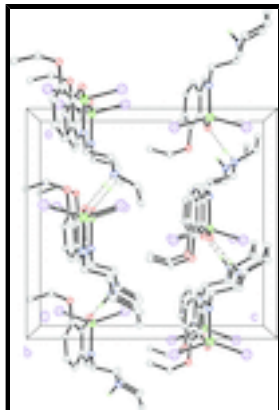


Fig. 2. The molecular packing of the title compound, viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

**{2-Ethoxy-6-[2-(piperidinium-1-yl)ethyliminomethyl]phenolato}diiodidozinc(II)**

*Crystal data*

[ZnI<sub>2</sub>(C<sub>16</sub>H<sub>24</sub>N<sub>2</sub>O<sub>2</sub>)]

*M<sub>r</sub>* = 595.54

Orthorhombic, *Pna*2<sub>1</sub>

Hall symbol: P 2c -2n

*a* = 13.5934 (10) Å

*b* = 10.2381 (8) Å

*c* = 14.7871 (11) Å

*V* = 2057.9 (3) Å<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 1144

*D<sub>x</sub>* = 1.922 Mg m<sup>-3</sup>

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 4921 reflections

θ = 2.4–27.9°

μ = 4.20 mm<sup>-1</sup>

*T* = 298 K

Block, colourless

0.18 × 0.17 × 0.17 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

*T* = 298 K

ω scans

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

*T*<sub>min</sub> = 0.518, *T*<sub>max</sub> = 0.535

11751 measured reflections

4438 independent reflections

3763 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.029

θ<sub>max</sub> = 27.0°

θ<sub>min</sub> = 2.4°

*h* = -16→17

*k* = -9→13

*l* = -18→18

*Refinement*

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.028

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0339P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.068$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.02$	$\Delta\rho_{\max} = 0.58 \text{ e } \text{Å}^{-3}$
4438 reflections	$\Delta\rho_{\min} = -0.88 \text{ e } \text{Å}^{-3}$
212 parameters	Extinction correction: none
2 restraints	Absolute structure: Flack (1983), 2111 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.02 (2)
Secondary atom site location: difference Fourier map	

### 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 ( $\text{Å}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	-0.02523 (3)	0.78151 (4)	0.73753 (7)	0.03660 (10)
I1	-0.07635 (3)	0.89935 (4)	0.881593 (19)	0.05811 (13)
I2	-0.07707 (3)	0.89530 (5)	0.590518 (18)	0.06408 (15)
N1	0.1209 (2)	0.7453 (3)	0.7350 (4)	0.0383 (6)
N2	0.2829 (3)	0.9941 (4)	0.8503 (2)	0.0380 (8)
O1	-0.06258 (15)	0.5977 (2)	0.7346 (4)	0.0386 (5)
O2	-0.1483 (3)	0.3910 (3)	0.6564 (2)	0.0567 (9)
C1	0.1005 (3)	0.5138 (4)	0.7039 (3)	0.0405 (10)
C2	-0.0040 (3)	0.5075 (4)	0.7033 (3)	0.0373 (9)
C3	-0.0463 (4)	0.3937 (4)	0.6658 (4)	0.0491 (11)
C4	0.0100 (5)	0.2871 (5)	0.6416 (4)	0.0660 (15)
H4	-0.0202	0.2112	0.6210	0.079*
C5	0.1109 (5)	0.2932 (5)	0.6478 (4)	0.0677 (16)
H5	0.1485	0.2207	0.6322	0.081*
C6	0.1556 (4)	0.4029 (5)	0.6762 (3)	0.0549 (13)
H6	0.2240	0.4063	0.6778	0.066*
C7	0.1560 (3)	0.6312 (4)	0.7217 (3)	0.0415 (12)
H7	0.2241	0.6229	0.7238	0.050*
C8	0.1907 (3)	0.8546 (4)	0.7423 (5)	0.0457 (9)
H8A	0.1612	0.9329	0.7174	0.055*
H8B	0.2495	0.8349	0.7078	0.055*
C9	0.2174 (3)	0.8774 (4)	0.8385 (3)	0.0417 (10)
H9A	0.2506	0.8007	0.8619	0.050*
H9B	0.1578	0.8901	0.8735	0.050*

## supplementary materials

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C10	0.2269 (4)	1.1197 (4)	0.8443 (4)	0.0549 (12)
H10A	0.1750	1.1199	0.8893	0.066*
H10B	0.1965	1.1265	0.7851	0.066*
C11	0.2937 (5)	1.2366 (5)	0.8595 (4)	0.0739 (17)
H11A	0.3412	1.2418	0.8106	0.089*
H11B	0.2547	1.3159	0.8586	0.089*
C12	0.3470 (5)	1.2274 (6)	0.9474 (4)	0.0751 (17)
H12A	0.3002	1.2320	0.9968	0.090*
H12B	0.3921	1.3003	0.9532	0.090*
C13	0.4043 (5)	1.0991 (6)	0.9531 (5)	0.0674 (16)
H13A	0.4553	1.0984	0.9073	0.081*
H13B	0.4357	1.0927	1.0118	0.081*
C14	0.3384 (4)	0.9850 (5)	0.9397 (3)	0.0504 (12)
H14A	0.2916	0.9807	0.9891	0.060*
H14B	0.3772	0.9055	0.9405	0.060*
C15	-0.1804 (5)	0.4628 (8)	0.5756 (4)	0.083 (2)
H15A	-0.2514	0.4725	0.5771	0.099*
H15B	-0.1516	0.5495	0.5764	0.099*
C16	-0.1523 (7)	0.3969 (10)	0.4922 (6)	0.126 (4)
H16A	-0.0819	0.3911	0.4889	0.189*
H16B	-0.1764	0.4456	0.4413	0.189*
H16C	-0.1800	0.3107	0.4914	0.189*
H2	0.333 (3)	0.987 (6)	0.811 (3)	0.080*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Zn1	0.02854 (19)	0.0357 (2)	0.0455 (2)	-0.00197 (15)	0.0007 (4)	-0.0052 (4)
I1	0.0524 (3)	0.0591 (2)	0.0628 (3)	-0.0002 (2)	0.0065 (2)	-0.0281 (2)
I2	0.0628 (4)	0.0766 (3)	0.0528 (3)	0.0076 (2)	0.0024 (2)	0.0169 (2)
N1	0.0261 (13)	0.0474 (16)	0.0414 (15)	-0.0071 (11)	-0.003 (3)	0.002 (3)
N2	0.0350 (19)	0.0452 (19)	0.0338 (18)	-0.0077 (16)	-0.0043 (15)	-0.0033 (15)
O1	0.0316 (12)	0.0381 (13)	0.0460 (13)	-0.0049 (9)	0.007 (2)	-0.005 (2)
O2	0.059 (2)	0.053 (2)	0.058 (2)	-0.0245 (15)	0.0061 (18)	-0.0095 (16)
C1	0.040 (2)	0.045 (2)	0.037 (2)	0.0128 (19)	-0.0005 (16)	0.0015 (17)
C2	0.042 (2)	0.034 (2)	0.036 (2)	0.0008 (18)	0.0030 (16)	0.0012 (15)
C3	0.062 (3)	0.039 (3)	0.046 (3)	-0.006 (2)	0.007 (2)	-0.004 (2)
C4	0.096 (5)	0.038 (3)	0.064 (4)	-0.002 (3)	0.009 (3)	-0.006 (2)
C5	0.092 (5)	0.045 (3)	0.067 (4)	0.028 (3)	0.006 (3)	-0.009 (3)
C6	0.058 (3)	0.060 (3)	0.047 (3)	0.025 (2)	0.002 (2)	0.000 (2)
C7	0.0279 (18)	0.061 (2)	0.036 (3)	0.0033 (16)	-0.0049 (18)	-0.004 (2)
C8	0.0343 (18)	0.055 (2)	0.048 (2)	-0.0151 (16)	-0.001 (3)	0.001 (4)
C9	0.037 (2)	0.044 (2)	0.045 (2)	-0.0062 (19)	-0.0032 (19)	0.0025 (18)
C10	0.067 (3)	0.049 (3)	0.049 (3)	0.008 (2)	-0.011 (3)	-0.002 (2)
C11	0.118 (5)	0.046 (3)	0.058 (3)	-0.005 (3)	-0.015 (3)	-0.002 (2)
C12	0.110 (5)	0.059 (3)	0.056 (3)	-0.020 (3)	-0.014 (3)	-0.007 (3)
C13	0.058 (4)	0.084 (4)	0.060 (4)	-0.019 (3)	-0.014 (3)	-0.012 (3)
C14	0.047 (3)	0.059 (3)	0.046 (3)	0.002 (2)	-0.017 (2)	0.000 (2)

C15	0.050 (4)	0.122 (6)	0.076 (4)	-0.021 (4)	0.002 (3)	-0.030 (4)
C16	0.073 (5)	0.214 (12)	0.091 (5)	0.007 (6)	-0.018 (5)	-0.051 (6)

*Geometric parameters (Å, °)*

Zn1—O1	1.950 (2)	C8—H8A	0.9700
Zn1—N1	2.021 (3)	C8—H8B	0.9700
Zn1—I1	2.5448 (9)	C9—H9A	0.9700
Zn1—I2	2.5651 (9)	C9—H9B	0.9700
N1—C7	1.277 (5)	C10—C11	1.520 (7)
N1—C8	1.471 (4)	C10—H10A	0.9700
N2—C10	1.498 (6)	C10—H10B	0.9700
N2—C9	1.500 (5)	C11—C12	1.491 (8)
N2—C14	1.524 (5)	C11—H11A	0.9700
N2—H2	0.90 (4)	C11—H11B	0.9700
O1—C2	1.304 (5)	C12—C13	1.529 (8)
O2—C3	1.394 (6)	C12—H12A	0.9700
O2—C15	1.470 (8)	C12—H12B	0.9700
C1—C6	1.421 (6)	C13—C14	1.485 (7)
C1—C2	1.421 (6)	C13—H13A	0.9700
C1—C7	1.444 (6)	C13—H13B	0.9700
C2—C3	1.412 (6)	C14—H14A	0.9700
C3—C4	1.381 (7)	C14—H14B	0.9700
C4—C5	1.376 (9)	C15—C16	1.457 (9)
C4—H4	0.9300	C15—H15A	0.9700
C5—C6	1.345 (7)	C15—H15B	0.9700
C5—H5	0.9300	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—H7	0.9300	C16—H16C	0.9600
C8—C9	1.486 (8)		
O1—Zn1—N1	94.50 (10)	N2—C9—H9A	109.1
O1—Zn1—I1	113.89 (16)	C8—C9—H9B	109.1
N1—Zn1—I1	111.78 (16)	N2—C9—H9B	109.1
O1—Zn1—I2	110.37 (17)	H9A—C9—H9B	107.8
N1—Zn1—I2	109.73 (17)	N2—C10—C11	111.3 (4)
I1—Zn1—I2	114.776 (17)	N2—C10—H10A	109.4
C7—N1—C8	117.8 (3)	C11—C10—H10A	109.4
C7—N1—Zn1	122.5 (3)	N2—C10—H10B	109.4
C8—N1—Zn1	119.5 (2)	C11—C10—H10B	109.4
C10—N2—C9	112.0 (3)	H10A—C10—H10B	108.0
C10—N2—C14	110.8 (4)	C12—C11—C10	111.7 (5)
C9—N2—C14	110.3 (3)	C12—C11—H11A	109.3
C10—N2—H2	115 (4)	C10—C11—H11A	109.3
C9—N2—H2	108 (4)	C12—C11—H11B	109.3
C14—N2—H2	101 (4)	C10—C11—H11B	109.3
C2—O1—Zn1	122.1 (2)	H11A—C11—H11B	107.9
C3—O2—C15	111.5 (4)	C11—C12—C13	110.5 (5)
C6—C1—C2	119.3 (4)	C11—C12—H12A	109.6
C6—C1—C7	116.2 (4)	C13—C12—H12A	109.6

## supplementary materials

C2—C1—C7	124.1 (4)	C11—C12—H12B	109.6
O1—C2—C3	118.4 (4)	C13—C12—H12B	109.6
O1—C2—C1	125.1 (4)	H12A—C12—H12B	108.1
C3—C2—C1	116.5 (4)	C14—C13—C12	111.2 (5)
C4—C3—O2	120.7 (4)	C14—C13—H13A	109.4
C4—C3—C2	121.9 (5)	C12—C13—H13A	109.4
O2—C3—C2	117.4 (4)	C14—C13—H13B	109.4
C5—C4—C3	120.0 (5)	C12—C13—H13B	109.4
C5—C4—H4	120.0	H13A—C13—H13B	108.0
C3—C4—H4	120.0	C13—C14—N2	111.5 (4)
C6—C5—C4	120.6 (5)	C13—C14—H14A	109.3
C6—C5—H5	119.7	N2—C14—H14A	109.3
C4—C5—H5	119.7	C13—C14—H14B	109.3
C5—C6—C1	121.3 (5)	N2—C14—H14B	109.3
C5—C6—H6	119.4	H14A—C14—H14B	108.0
C1—C6—H6	119.4	C16—C15—O2	112.3 (7)
N1—C7—C1	126.5 (4)	C16—C15—H15A	109.1
N1—C7—H7	116.8	O2—C15—H15A	109.1
C1—C7—H7	116.8	C16—C15—H15B	109.1
N1—C8—C9	110.4 (5)	O2—C15—H15B	109.1
N1—C8—H8A	109.6	H15A—C15—H15B	107.9
C9—C8—H8A	109.6	C15—C16—H16A	109.5
N1—C8—H8B	109.6	C15—C16—H16B	109.5
C9—C8—H8B	109.6	H16A—C16—H16B	109.5
H8A—C8—H8B	108.1	C15—C16—H16C	109.5
C8—C9—N2	112.5 (3)	H16A—C16—H16C	109.5
C8—C9—H9A	109.1	H16B—C16—H16C	109.5

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H2\cdots O2^i$	0.90 (4)	2.61 (5)	3.237 (5)	127 (4)
$N2-H2\cdots O1^i$	0.90 (4)	2.01 (5)	2.867 (5)	159 (5)

Symmetry codes: (i)  $x+1/2, -y+3/2, z$ .

Fig. 1

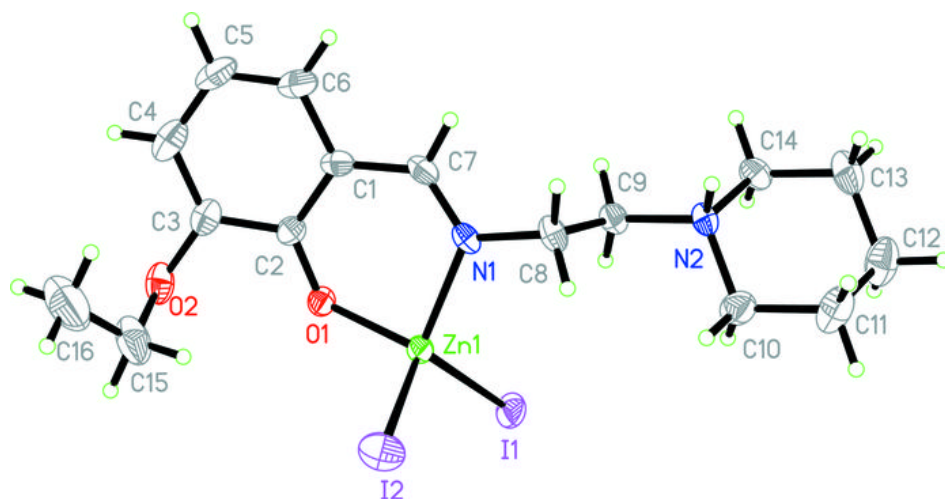


Fig. 2

