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# Bis{5-methoxy-2-[(1*H*-pyrrol-2-yl)methyliminomethyl]phenolato}zinc(II)

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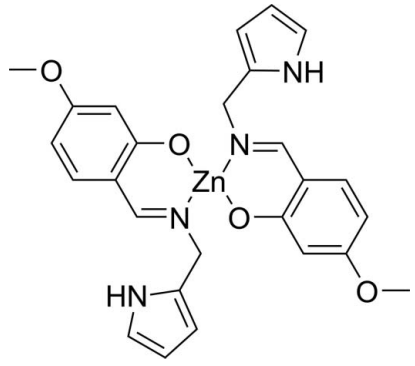
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.058; $wR$ factor = 0.154; data-to-parameter ratio = 18.0.

The title compound, $[Zn(C_{13}H_{13}N_2O_2)_2]$, contains a Zn(II) centre, located on a twofold rotation axis, that is coordinated by two O atoms and two N atoms from two salicylal Schiff base molecules. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds.

## Related literature

For the importance of zinc derivatives in biological processes, see: Chen *et al.* (2007); Xiao & Xiao (2008); Xiao *et al.* (2007, 2008). For related structures, see: You & Zhu (2006); Zhu *et al.* (2004); Qiu *et al.* (2004).




## Experimental

### Crystal data

$[Zn(C_{13}H_{13}N_2O_2)_2]$ $M_r$ = 523.88
Monoclinic, $C2/c$
$a$ = 27.210 (4) Å
$b$ = 5.2239 (9) Å
$c$ = 24.335 (3) Å
$\beta$ = 137.179 (9)°
$V$ = 2351.1 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.09 mm$^{-1}$
$T$ = 298 K
0.30 × 0.30 × 0.20 mm

### Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.736, $T_{max}$ = 0.812
8100 measured reflections
2881 independent reflections
2429 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.154
$S$ = 1.08
2881 reflections
160 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.57 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C9—H9$A$···O1$^{i}$ | 0.97 | 2.32 | 3.291 (4) | 177 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2551).

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# supplementary materials

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# Bis{5-methoxy-2-[(1*H*-pyrrol-2-yl)methyliminomethyl]phenolato}zinc(II)

**Y.-J. Cai, P. Huang, J. Li and Q. Wang**

## Comment

Zinc derivatives are particularly interesting owing to their essential importance in several biological processes (Xiao *et al.*, 2008; Xiao & Xiao, 2008; Xiao *et al.*, 2007; Chen *et al.*, 2007). We have reported the structures of a few zinc(II) complexes (You & Zhu, 2006; Qiu *et al.*, 2004; Zhu *et al.*, 2004). As an extension of our work on the structural characterization of zinc compounds, we report the crystal structure of the title compound, (I), which has been determined in an attempt to understand the structural behaviour of nitrogen containing ligands.

The present X-ray single-crystal diffraction study reveals that compound (I), Bis(4-methoxysalicylidene(1*H*-pyrrol-2-yl)methanaminato)zinc(II), consists of a Zn(II) atom and two bidentate salicylal Schiff base ligands. As shown in Fig. 1, The central Zn atom exhibits 4-coordination by two N atoms from imine moieties and two O-anions from salicylal groups, forming a slightly distorted tetrahedron. The distortion arises from the difference between O—Zn and Zn—N (Table 1). The O1—C1 distance (1.308 (3) Å) and C2—C8 (1.425 (4) Å) are shorter than classical single C—O and C—C bonds, respectively. This suggests that electron significantly delocalized over the O1—C1—C2—C8—N1 group and the same to the O1A—C1A—C2A—C8A—N1A group. Zn1, N1, C8 and O1 almost stand in the plane of ring C1 to C6, and makes dihedral angle of 85.569 (45) ° with corresponding plane of the other half molecules.

The hydrogen-bonding interactions occur between the C—H in the $CH_2$ group as donors and O in the salicylal moiety as acceptors (Table 1). These intermolecular hydrogen bonds construct an infinite ribbon. Interactions between the ribbons are van der Waals forces.

## Experimental

0.5 mmol of zinc oxide, 1 mmol of 4-methoxysalicylaldehyde and 1 mmol of (1*H*-pyrrol-2-yl)methanamine were dissolved in 10 ml methanol. After 3 ml ammonia was added, the result solution was then heated to 423 K for 12 h. The reactor was cooled to room temperature at a rate of 10 K $h^{-1}$. The mixture was filtered and held at room temperature for 12 d. Colorless block crystals were isolated in 41% yield.

## Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms. $U_{iso}$(H) values were set at 1.2 times $U_{eq}$(C, N) for aromatic C and N in pyrrole ring groups and 1.5 times $U_{eq}$(C) for $CH_3$ groups.

## Figures

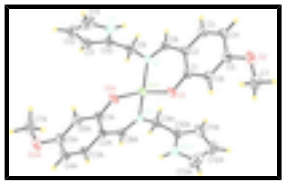


Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

## Bis{5-methoxy-2-[(1*H*-pyrrol-2-yl)methyliminomethyl]phenolato}zinc(II)

*Crystal data*

| | |
|---|---|
| $[Zn(C_{13}H_{13}N_2O_2)_2]$ | $F(000) = 1088$ |
| $M_r = 523.88$ | $D_x = 1.480$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -C 2yc | Cell parameters from 2301 reflections |
| $a = 27.210$ (4) Å | $\theta = 2.5$–$26.1°$ |
| $b = 5.2239$ (9) Å | $\mu = 1.09$ mm$^{-1}$ |
| $c = 24.335$ (3) Å | $T = 298$ K |
| $\beta = 137.179$ (9)° | Block, colorless |
| $V = 2351.1$ (6) Å$^3$ | 0.30 × 0.30 × 0.20 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX area-detector diffractometer | 2881 independent reflections |
| Radiation source: fine-focus sealed tube | 2429 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.028$ |
| φ and ω scans | $\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $h = -36 \rightarrow 35$ |
| $T_{min} = 0.736$, $T_{max} = 0.812$ | $k = -6 \rightarrow 6$ |
| 8100 measured reflections | $l = -26 \rightarrow 32$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.058$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.154$ | H-atom parameters constrained |
| $S = 1.08$ | $w = 1/[\sigma^2(F_o^2) + (0.0814P)^2 + 2.3252P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2881 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 160 parameters | $\Delta\rho_{max} = 0.72$ e Å$^{-3}$ |

0 restraints $\Delta\rho_{min} = -0.57$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.93603 (16) | 0.2495 (6) | 0.29548 (18) | 0.0401 (6) |
| C2 | 0.87592 (16) | 0.4163 (6) | 0.24129 (18) | 0.0398 (6) |
| C3 | 0.81985 (18) | 0.3941 (7) | 0.2349 (2) | 0.0503 (8) |
| H3 | 0.7802 | 0.5026 | 0.1996 | 0.060* |
| C4 | 0.82168 (18) | 0.2211 (7) | 0.2782 (2) | 0.0550 (8) |
| H4 | 0.7838 | 0.2105 | 0.2720 | 0.066* |
| C5 | 0.8813 (2) | 0.0602 (7) | 0.3317 (2) | 0.0490 (8) |
| C6 | 0.93736 (19) | 0.0707 (6) | 0.3401 (2) | 0.0462 (7) |
| H6 | 0.9763 | −0.0409 | 0.3754 | 0.055* |
| C7 | 0.9380 (2) | −0.2809 (8) | 0.4279 (2) | 0.0673 (10) |
| H7A | 0.9385 | −0.3953 | 0.3973 | 0.101* |
| H7B | 0.9317 | −0.3774 | 0.4559 | 0.101* |
| H7C | 0.9830 | −0.1896 | 0.4663 | 0.101* |
| C8 | 0.86588 (16) | 0.6031 (6) | 0.19107 (18) | 0.0418 (7) |
| H8 | 0.8240 | 0.7009 | 0.1592 | 0.050* |
| C9 | 0.88198 (18) | 0.8391 (7) | 0.1226 (2) | 0.0507 (8) |
| H9A | 0.9208 | 0.9554 | 0.1449 | 0.061* |
| H9B | 0.8430 | 0.9389 | 0.1065 | 0.061* |
| C10 | 0.85569 (18) | 0.7069 (7) | 0.0508 (2) | 0.0523 (8) |
| C11 | 0.8621 (2) | 0.7591 (13) | 0.0026 (3) | 0.0938 (18) |
| H11 | 0.8869 | 0.8941 | 0.0066 | 0.113* |
| C12 | 0.8210 (3) | 0.5539 (14) | −0.0582 (3) | 0.103 (2) |
| H12 | 0.8155 | 0.5318 | −0.1004 | 0.123* |
| C13 | 0.7934 (3) | 0.4069 (13) | −0.0421 (3) | 0.103 (2) |
| H13 | 0.7646 | 0.2637 | −0.0717 | 0.124* |
| N1 | 0.90797 (13) | 0.6538 (5) | 0.18427 (15) | 0.0398 (5) |
| N2 | 0.81349 (18) | 0.4967 (6) | 0.02362 (19) | 0.0571 (8) |
| H2 | 0.8016 | 0.4320 | 0.0451 | 0.069* |
| O1 | 0.99094 (12) | 0.2514 (5) | 0.30668 (15) | 0.0552 (6) |
| O2 | 0.88016 (16) | −0.1038 (6) | 0.37455 (17) | 0.0663 (7) |
| Zn1 | 1.0000 | 0.46424 (10) | 0.2500 | 0.0430 (2) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0412 (15) | 0.0449 (16) | 0.0473 (16) | −0.0004 (12) | 0.0366 (14) | −0.0027 (13) |
| C2 | 0.0376 (14) | 0.0469 (17) | 0.0450 (16) | −0.0015 (12) | 0.0334 (14) | −0.0038 (13) |
| C3 | 0.0426 (16) | 0.065 (2) | 0.0567 (19) | 0.0051 (15) | 0.0406 (16) | 0.0019 (16) |
| C4 | 0.0501 (18) | 0.073 (2) | 0.065 (2) | −0.0037 (17) | 0.0497 (18) | −0.0036 (18) |
| C5 | 0.058 (2) | 0.0520 (18) | 0.0553 (19) | −0.0101 (15) | 0.0475 (18) | −0.0071 (15) |
| C6 | 0.0513 (18) | 0.0469 (17) | 0.0528 (18) | 0.0015 (14) | 0.0421 (16) | 0.0016 (14) |
| C7 | 0.086 (3) | 0.060 (2) | 0.067 (2) | −0.011 (2) | 0.059 (2) | 0.0029 (19) |
| C8 | 0.0363 (14) | 0.0465 (16) | 0.0448 (16) | 0.0037 (12) | 0.0304 (14) | −0.0004 (13) |
| C9 | 0.0515 (18) | 0.0459 (18) | 0.060 (2) | −0.0014 (14) | 0.0424 (17) | 0.0043 (15) |
| C10 | 0.0449 (17) | 0.067 (2) | 0.0499 (18) | 0.0130 (15) | 0.0363 (16) | 0.0157 (16) |
| C11 | 0.067 (3) | 0.156 (5) | 0.081 (3) | 0.038 (3) | 0.061 (3) | 0.049 (3) |
| C12 | 0.081 (4) | 0.168 (6) | 0.059 (3) | 0.047 (4) | 0.051 (3) | 0.013 (3) |
| C13 | 0.091 (4) | 0.114 (5) | 0.060 (3) | 0.025 (3) | 0.042 (3) | −0.013 (3) |
| N1 | 0.0355 (12) | 0.0458 (13) | 0.0423 (13) | −0.0016 (10) | 0.0298 (11) | −0.0022 (11) |
| N2 | 0.0605 (19) | 0.0552 (18) | 0.0486 (16) | −0.0063 (13) | 0.0378 (16) | −0.0074 (13) |
| O1 | 0.0481 (12) | 0.0674 (15) | 0.0712 (15) | 0.0150 (11) | 0.0504 (13) | 0.0210 (12) |
| O2 | 0.0821 (18) | 0.0695 (17) | 0.0832 (19) | −0.0025 (15) | 0.0719 (17) | 0.0089 (15) |
| Zn1 | 0.0374 (3) | 0.0513 (3) | 0.0530 (3) | 0.000 | 0.0372 (3) | 0.000 |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.308 (3) | C9—N1 | 1.460 (4) |
| C1—C6 | 1.412 (4) | C9—C10 | 1.487 (5) |
| C1—C2 | 1.417 (4) | C9—H9A | 0.9700 |
| C2—C3 | 1.421 (4) | C9—H9B | 0.9700 |
| C2—C8 | 1.425 (4) | C10—C11 | 1.333 (5) |
| C3—C4 | 1.360 (5) | C10—N2 | 1.359 (5) |
| C3—H3 | 0.9300 | C11—C12 | 1.471 (8) |
| C4—C5 | 1.391 (5) | C11—H11 | 0.9300 |
| C4—H4 | 0.9300 | C12—C13 | 1.318 (9) |
| C5—O2 | 1.367 (4) | C12—H12 | 0.9300 |
| C5—C6 | 1.384 (5) | C13—N2 | 1.340 (6) |
| C6—H6 | 0.9300 | C13—H13 | 0.9300 |
| C7—O2 | 1.422 (5) | N1—Zn1 | 1.983 (2) |
| C7—H7A | 0.9600 | N2—H2 | 0.8600 |
| C7—H7B | 0.9600 | O1—Zn1 | 1.923 (2) |
| C7—H7C | 0.9600 | Zn1—O1$^{i}$ | 1.923 (2) |
| C8—N1 | 1.299 (4) | Zn1—N1$^{i}$ | 1.983 (2) |
| C8—H8 | 0.9300 | | |
| O1—C1—C6 | 117.6 (3) | N1—C9—H9B | 109.5 |
| O1—C1—C2 | 123.5 (3) | C10—C9—H9B | 109.5 |
| C6—C1—C2 | 118.9 (3) | H9A—C9—H9B | 108.1 |
| C1—C2—C3 | 117.7 (3) | C11—C10—N2 | 110.3 (4) |
| C1—C2—C8 | 125.7 (3) | C11—C10—C9 | 132.7 (4) |

| | | | |
|---|---|---|---|
| C3—C2—C8 | 116.6 (3) | N2—C10—C9 | 116.9 (3) |
| C4—C3—C2 | 122.9 (3) | C10—C11—C12 | 104.0 (5) |
| C4—C3—H3 | 118.6 | C10—C11—H11 | 128.0 |
| C2—C3—H3 | 118.6 | C12—C11—H11 | 128.0 |
| C3—C4—C5 | 118.8 (3) | C13—C12—C11 | 107.7 (5) |
| C3—C4—H4 | 120.6 | C13—C12—H12 | 126.1 |
| C5—C4—H4 | 120.6 | C11—C12—H12 | 126.1 |
| O2—C5—C6 | 123.5 (3) | C12—C13—N2 | 109.2 (6) |
| O2—C5—C4 | 115.5 (3) | C12—C13—H13 | 125.4 |
| C6—C5—C4 | 121.1 (3) | N2—C13—H13 | 125.4 |
| C5—C6—C1 | 120.6 (3) | C8—N1—C9 | 117.3 (3) |
| C5—C6—H6 | 119.7 | C8—N1—Zn1 | 120.3 (2) |
| C1—C6—H6 | 119.7 | C9—N1—Zn1 | 122.2 (2) |
| O2—C7—H7A | 109.5 | C13—N2—C10 | 108.8 (5) |
| O2—C7—H7B | 109.5 | C13—N2—H2 | 125.6 |
| H7A—C7—H7B | 109.5 | C10—N2—H2 | 125.6 |
| O2—C7—H7C | 109.5 | C1—O1—Zn1 | 125.7 (2) |
| H7A—C7—H7C | 109.5 | C5—O2—C7 | 118.3 (3) |
| H7B—C7—H7C | 109.5 | O1—Zn1—O1$^{i}$ | 109.36 (15) |
| N1—C8—C2 | 128.1 (3) | O1—Zn1—N1$^{i}$ | 117.41 (10) |
| N1—C8—H8 | 116.0 | O1$^{i}$—Zn1—N1$^{i}$ | 96.71 (10) |
| C2—C8—H8 | 116.0 | O1—Zn1—N1 | 96.71 (10) |
| N1—C9—C10 | 110.7 (3) | O1$^{i}$—Zn1—N1 | 117.41 (10) |
| N1—C9—H9A | 109.5 | N1$^{i}$—Zn1—N1 | 120.10 (15) |
| C10—C9—H9A | 109.5 | | |
| O1—C1—C2—C3 | −180.0 (3) | C2—C8—N1—C9 | 174.4 (3) |
| C6—C1—C2—C3 | 0.1 (4) | C2—C8—N1—Zn1 | −1.1 (4) |
| O1—C1—C2—C8 | 1.2 (5) | C10—C9—N1—C8 | −102.9 (3) |
| C6—C1—C2—C8 | −178.8 (3) | C10—C9—N1—Zn1 | 72.5 (3) |
| C1—C2—C3—C4 | −0.1 (5) | C12—C13—N2—C10 | −0.4 (6) |
| C8—C2—C3—C4 | 178.8 (3) | C11—C10—N2—C13 | 1.4 (5) |
| C2—C3—C4—C5 | 0.7 (5) | C9—C10—N2—C13 | 178.5 (4) |
| C3—C4—C5—O2 | 178.3 (3) | C6—C1—O1—Zn1 | 177.0 (2) |
| C3—C4—C5—C6 | −1.3 (5) | C2—C1—O1—Zn1 | −3.0 (5) |
| O2—C5—C6—C1 | −178.3 (3) | C6—C5—O2—C7 | −3.0 (5) |
| C4—C5—C6—C1 | 1.3 (5) | C4—C5—O2—C7 | 177.4 (3) |
| O1—C1—C6—C5 | 179.4 (3) | C1—O1—Zn1—O1$^{i}$ | −119.8 (3) |
| C2—C1—C6—C5 | −0.7 (5) | C1—O1—Zn1—N1$^{i}$ | 131.4 (3) |
| C1—C2—C8—N1 | 1.1 (5) | C1—O1—Zn1—N1 | 2.4 (3) |
| C3—C2—C8—N1 | −177.8 (3) | C8—N1—Zn1—O1 | −0.4 (3) |
| N1—C9—C10—C11 | −139.4 (4) | C9—N1—Zn1—O1 | −175.6 (2) |
| N1—C9—C10—N2 | 44.4 (4) | C8—N1—Zn1—O1$^{i}$ | 115.5 (2) |
| N2—C10—C11—C12 | −1.7 (4) | C9—N1—Zn1—O1$^{i}$ | −59.7 (3) |
| C9—C10—C11—C12 | −178.1 (4) | C8—N1—Zn1—N1$^{i}$ | −127.6 (3) |
| C10—C11—C12—C13 | 1.4 (6) | C9—N1—Zn1—N1$^{i}$ | 57.2 (2) |
| C11—C12—C13—N2 | −0.6 (6) | | |

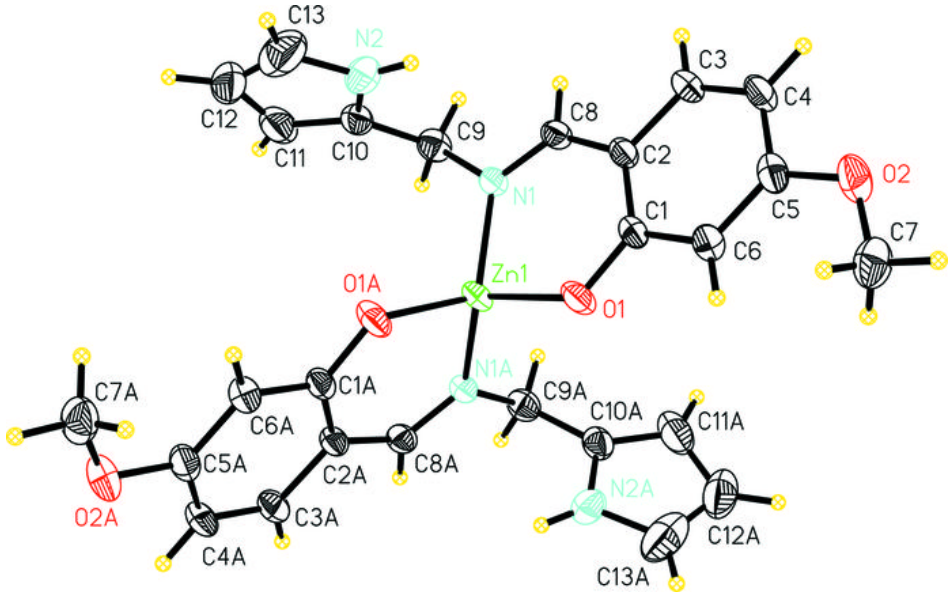
Symmetry codes: (i) −*x*+2, *y*, −*z*+1/2.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9A···O1$^{ii}$ | 0.97 | 2.32 | 3.291 (4) | 177 |

Symmetry codes: (ii) −*x*+2, *y*+1, −*z*+1/2.

**Fig. 1**


C13
N2
C12
C11
C10
C9
C8
C3
C4
C2
N1
C1
C5
O2
C7
C6
Zn1
O1A
O1
N1A
C9A
C10A
C11A
C7A
C6A
C1A
C5A
C2A
C8A
N2A
C12A
O2A
C3A
C4A
C13A