Bis{μ-3-methoxy-N′-[1-(2-oxidophenyl)ethylidene]-benzohydrazidato}bis[pyridinezinc(II)] pyridine solvate

Hapipah Ali,[a] Nur Ashikin Khamis,[a] M. Sukeri. M. Yusof[b] and Bohari. M. Yamin[b]*

[a]Department of Chemistry, University of Malaya, Kuala Lumpur, Malaysia, and [b]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail: bohari@pkrisc.cc.ukm.my

The title compound, [Zn$_2$(C$_{21}$H$_{14}$N$_2$O$_3$)$_2$(C$_5$H$_5$N)$_2$]·C$_5$H$_5$N, is dimeric *via* Zn—O bridging, with an average value for the Zn—O bonds of 2.027 (2) Å. The Zn···Zn separation is 3.1546 (5) Å. The molecule has a center of inversion and the coordination geometry of both Zn atoms is square pyramidal.

Comment

The title compound, (I), obtained by recrystallization of [Zn(C$_{21}$H$_{14}$N$_2$O$_3$)$_2$] from pyridine is isostructural with [Zn(C$_{13}$H$_9$N$_2$O$_2$SCl)(C$_5$H$_5$N)]$_2$ (Ali *et al.*, 2003), except for the presence of one solvated pyridine molecule (Fig. 1). The molecule is dimeric with an average Zn—O bridging distance of 2.027 (2) Å and a Zn1···Zn1^i [symmetry code: (i) $2 - x$, $2 - y$, $-z$] separation of 3.1546 (5) Å, in agreement with the same distances [2.021 (2) and 3.1004 (5) Å, respectively] in the [Zn(C$_{13}$H$_9$N$_2$O$_2$SCl)(C$_5$H$_5$N)]$_2$ complex.

(I)
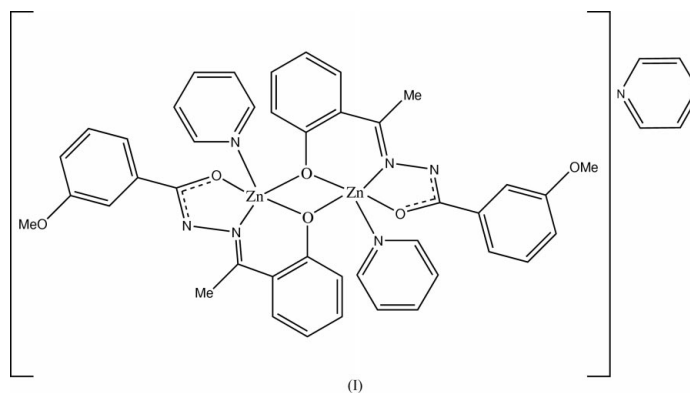
The complex in (I) has a center of inversion and the coordination geometry of both Zn atoms in the molecule is closer to square pyramidal than trigonal bipyramidal. Atoms O1, O3, O3^i and N2 occupy the basal plane [maximum displacement of 1.083 (2) Å for atom N2 from the mean plane] with atom N3 located at the apex of the pyramid. The N2—Zn1—N3 bond angle is 112.05 (8)°. The structural dimensions of the *O,N,O*-tridentate ligands (Table 1) are in normal ranges (Orpen *et al.*, 1989; Allen *et al.*, 1987) and are in agreement with other pyramidal zinc complexes. The methoxyphenyl group [O2/C1–C6; maximum deviation of 0.009 (3) Å for atom C5 from the mean plane] and the phenolate group [O3/C11–C16; maximum deviation of −0.028 (3) Å for atom C12 from the mean plane] are individually planar and make a dihedral angle of 36.85 (11)°. The coordinated pyridine ring (N3/C17—C21) makes dihedral angles with the methoxyphenyl and phenolate groups of 85.16 (13) and 80.38 (14)°, respectively. There is a weak intramolecular C—H···O interaction (Table 2) in the title complex.

Experimental

The title complex was synthesized by the template condensation of 2-hydroxyacetophenone (0.24 g, 1.8 mmol) and 3-methoxy-benzhydrazide (0.30 g, 1.8 mmol) with zinc acetate dihydrate (0.20 g, 0.9 mmol) by refluxing and stirring in ethanol for 5 h. The yellow solid was filtered off and recrystallized from pyridine.

Crystal data

$[Zn_2(C_{21}H_{14}N_2O_3)_2(C_5H_5N)_2]\cdot$ C_5H_5N
$M_r = 1011.76$
Triclinic, $P\bar{1}$
$a = 8.5896$ (8) Å
$b = 10.6112$ (10) Å
$c = 14.2017$ (13) Å
$\alpha = 71.360$ (2)°
$\beta = 73.261$ (1)°
$\gamma = 79.670$ (2)°
$V = 1169.11$ (19) Å^3

$Z = 1$
$D_x = 1.437$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5617 reflections
$\theta = 1.5$–25.0°
$\mu = 1.09$ mm^{-1}
$T = 293$ (2) K
Block, pale yellow
$0.46 \times 0.37 \times 0.20$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.635$, $T_{max} = 0.812$
7209 measured reflections

4005 independent reflections
3771 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.087$
$S = 1.04$
4005 reflections
307 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.6076P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O3	2.0125 (14)	O1—C8	1.277 (3)
Zn1—O1	2.0147 (16)	O3—C11	1.341 (2)
Zn1—N3	2.0488 (19)	N1—C8	1.314 (3)
Zn1—O3^i	2.0540 (15)	N1—N2	1.396 (3)
Zn1—N2	2.0576 (18)	N2—C9	1.290 (3)
Zn1—Zn1^i	3.1546 (5)		
O3—Zn1—O1	103.59 (6)	N3—Zn1—O3^i	104.80 (7)
O3—Zn1—N3	102.82 (7)	O3—Zn1—N2	144.21 (7)
O1—Zn1—N3	101.35 (7)	O1—Zn1—N2	77.77 (7)
O3—Zn1—O3^i	78.26 (6)	N3—Zn1—N2	112.05 (8)
O1—Zn1—O3^i	152.68 (7)	O3^i—Zn1—N2	85.17 (7)

Symmetry code: (i) $2 - x, 2 - y, -z$.

Table 2
Hydrogen-bonding geometry (Å, °).

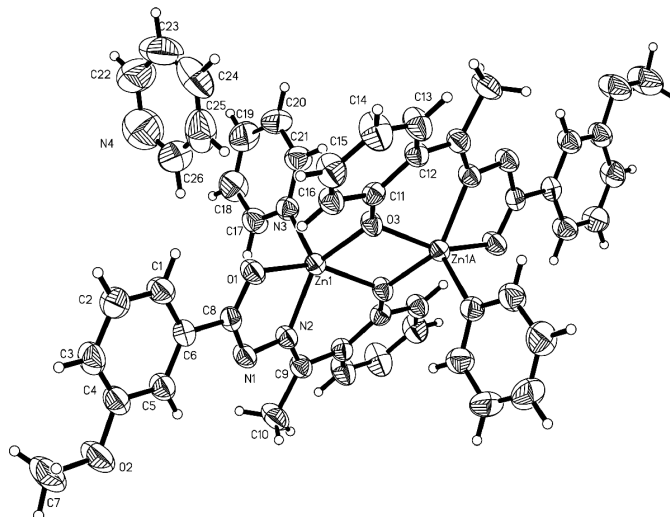| D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C16—H16A$\cdots$O1	0.93	2.30	3.133 (3)	148

Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids.

After their location in a difference map, all H atoms were positioned geometrically and allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 1997); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

References

Ali, H. M., Omar, R. S., Yusof, M. S. M. & Yamin, B. M. (2003). Acta Cryst. E**59**, m1118–m1120.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). J. Chem. Soc. Perkin Trans. 2, pp. S1–19.
Nardelli, M. (1995). J. Appl. Cryst. **28**, 659.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1989). J. Chem. Soc. Dalton Trans. pp. S1–83.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (1997). SHELXS97, SHELXL97 and SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). J. Appl. Cryst. **36**, 7–13.