Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[μ-2-methoxy-*N*′-(2-oxidobenzoyl)benzohydrazidato(3–)]dipyridinetrizinc(II)

Zong-Xue Yu[a] and Yu-Xi Sun[b]*

[a]Department of Chemical Engineering, Chongqing Three Gorges College, Wanzhou 404000, People's Republic of China, and [b]Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: yuxisun@163.com

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
R factor = 0.050
wR factor = 0.132
Data-to-parameter ratio = 15.0

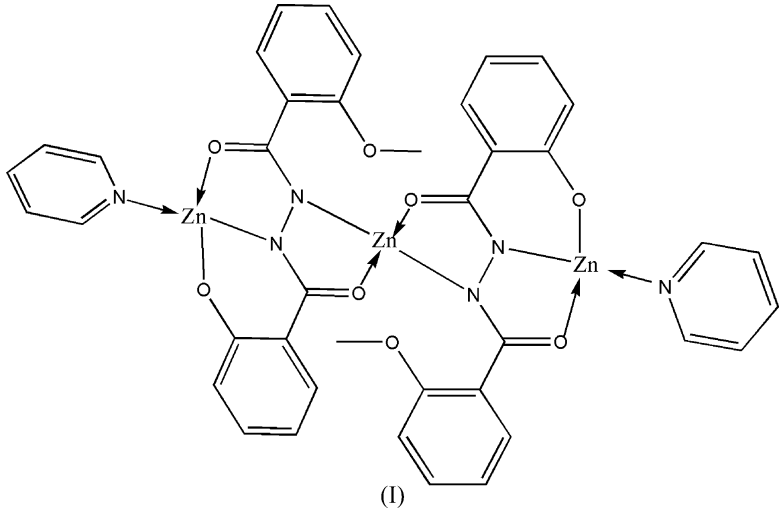
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 November 2006
Accepted 21 November 2006

In the title trinuclear zinc(II) complex, $[Zn_3(C_{15}H_{11}N_2O_4)_2(C_5H_5N)_2]$, the central Zn^{II} ion has a distorted square-planar coordination involving two O and two N atoms from two bridging *N*′-(2-hydroxybenzoyl)-2-methoxybenzohydrazide ligands. The coordination around the terminal Zn^{II} ions is square planar, involving two O atoms and one N atom of the bridging ligand and one N atom from a pyridine molecule.

Comment

Transition metal compounds are present in the active sites of several important classes of metalloproteins. The study of Schiff base compounds is of great interest in various fields of chemistry (Downing & Urbach, 1969; Ganeshpure *et al.*, 1996; Bosnich, 1968; Costes *et al.*, 1995; Duda *et al.*, 2003). The crystal structures of zinc(II) complexes have been widely studied (Howard *et al.*, 2006; Granifo *et al.*, 2006; Tong, 2005; You, 2005). As an extension of work on the structural characterization of complexes, the preparation and crystal structure of the title trinuclear zinc(II) complex, (I), is reported here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected bond distances and angles are given in Table 1. Each Zn^{II} atom has a square-planar geometry. The central Zn2 atom is coordinated by two O and two N atoms from two bridging trianionic *N*′-(2-hydroxybenzoyl)-2-methoxybenzohydrazide ligands, whereas the other two Zn^{II} atoms, Zn1 and Zn3, are coordinated by two O atoma and one N atom from one bridging ligand and the N atom of a pyridine molecule. The two bridging trianionic ligands therefore act as quinquedentate ligands through one phenolate O atom, two keto O atoms and two N atoms, and two pyridine molecules act as

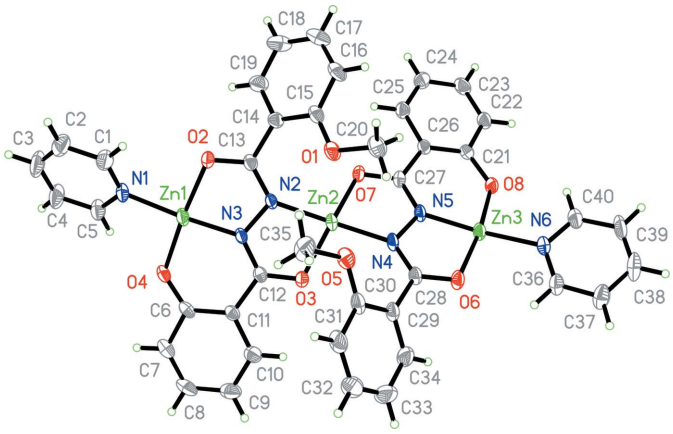


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii.

monodentate ligands through the N atom, forming a trinuclear zinc(II) complex.

The *trans* angle N2—Zn2—N4 at the central Zn2 is close to 180°; however, the other *trans* angle O3—Zn2—O7 deviates significantly from the ideal 180°. The four *trans* angles at Zn1 and Zn3 are close to 180°, ranging from 162.99 (13) to 174.51 (11)°. All the other angles subtended at the three Zn^{II} atoms are close to 90°, indicating that they are in a distorted square-planar configuration. The dihedral angle between the C6–C11 and C14–C19 benzene rings is 27.5 (3)°, and that between the C21–C26 and C29–C34 benzene rings is 32.3 (3)°.

As illustrated in Fig. 2, a weak C5–H5···O3^{i} interaction [H5···O3^{i} = 2.43 Å, C5···O3^{i} = 3.307 (5) Å and C5—H5···O3^{i} = 157°; symmetry code: (i) $1-x, 1-y, 1-z$] is observed in the crystal structure of (I). The unit cell contains two solvent-accessible voids, each with a volume of 43 Å^3, but no solvent molecules were found in the crystal structure.

Experimental

All chemicals were obtained from commercial sources and used without purification. 2-Hydroxybenzoyl chloride (31.3 mg, 0.2 mmol) and 2-methoxybenzoyl chloride (34.1 mg, 0.2 mmol) were dissolved in dry methanol (50 ml). The mixture was stirred while hydrazine monohydrate (0.2 mmol, about 10.2 mg) was added dropwise over 30 min at room temperature to give a clear yellow solution. To this solution was added a methanol solution (25 ml) of $Zn(OAc)_2$ (0.4 mmol, 73.6 mg) and pyridine (0.4 mmol, 31.7 mg), with stirring. The resulting solution was allowed to stand in air for 27 d, after which time pale-green plate-shaped crystals of (I) formed at the bottom of the vessel on slow evaporation of the methanol.

Crystal data

$[Zn_3(C_{15}H_{11}N_2O_4)_2(C_5H_5N)_2]$
M_r = 920.83
Monoclinic, $P2_1/c$
a = 13.0201 (10) Å
b = 18.4015 (14) Å
c = 16.6352 (12) Å
β = 110.088 (1)°
V = 3743.2 (5) Å^3
Z = 4
D_x = 1.634 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.97 mm^{-1}
T = 295 (2) K
Plate, pale green
0.33 × 0.18 × 0.08 mm

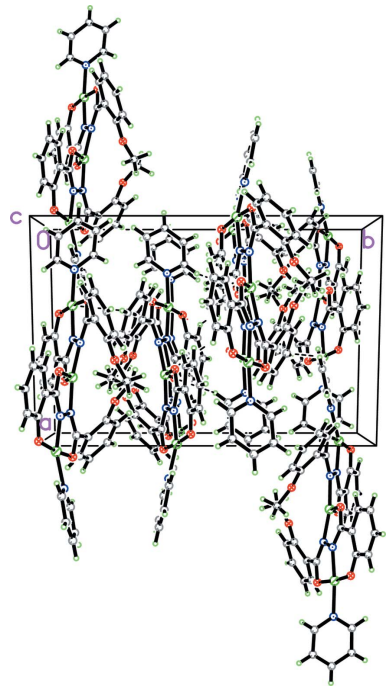


Figure 2
The packing of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.663, T_{max} = 0.858
30154 measured reflections
7738 independent reflections
5721 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.132
S = 1.02
7738 reflections
516 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 2.3964P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O4	1.880 (3)	Zn2—O3	1.951 (2)
Zn1—N3	1.888 (3)	Zn2—O7	1.955 (2)
Zn1—O2	1.950 (3)	Zn3—N5	1.904 (3)
Zn1—N1	1.990 (3)	Zn3—O8	1.915 (2)
Zn2—N2	1.934 (3)	Zn3—O6	1.974 (3)
Zn2—N4	1.936 (3)	Zn3—N6	2.008 (3)
O4—Zn1—N3	93.23 (12)	N2—Zn2—O7	102.20 (11)
O4—Zn1—O2	174.51 (11)	N4—Zn2—O7	81.56 (11)
N3—Zn1—O2	81.45 (11)	O3—Zn2—O7	148.65 (12)
O4—Zn1—N1	91.73 (13)	N5—Zn3—O8	91.91 (11)
N3—Zn1—N1	174.25 (13)	N5—Zn3—O6	80.68 (11)
O2—Zn1—N1	93.51 (12)	O8—Zn3—O6	172.11 (10)
N2—Zn2—N4	170.49 (13)	N5—Zn3—N6	162.99 (13)
N2—Zn2—O3	81.76 (11)	O8—Zn3—N6	94.69 (12)
N4—Zn2—O3	99.64 (12)	O6—Zn3—N6	93.17 (12)

All H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTPlus* (Bruker, 2002); data reduction: *SAINTPlus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

The authors thank Chongqing Three Gorges College for a research grant and Qufu Normal University for a technology grant.

References

Bosnich, B. (1968). *J. Am. Chem. Soc.* **90**, 627–632.
Bruker (2002). *SMART* (Version 5.628), *SAINT-Plus* (Version 6.22) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Costes, J. P., Dominiguez-Vera, J. M. & Laurent, J. P. (1995). *Polyhedron*, **14**, 2179–2187.
Downing, R. S. & Urbach, F. L. (1969). *J. Am. Chem. Soc.* **91**, 5977–5983.
Duda, D., Govindasamy, L., Agbandje-McKenna, M., Tu, C., Silverman, D. N. & McKenna, R. (2003). *Acta Cryst.* D**59**, 93–104.
Ganeshpure, P. A., Tembe, G. L. & Satish, S. (1996). *J. Mol. Catal.* **A113**, L423–L425.
Granifo, J., Garland, M. T. & Baggio, R. (2006). *Acta Cryst.* C**62**, m56–m59.
Howard, R. H., Bochmann, M. & Wright, J. A. (2006). *Acta Cryst.* C**62**, m293–m296.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Tong, Y.-P. (2005). *Acta Cryst.* E**61**, m1771–m1773.
You, Z.-L. (2005). *Acta Cryst.* C**61**, m383–m385.