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Tetrakis{μ-1-[2-(2-oxidoethylamino)ethyliminomethyl]-2-naphtholato}tetrazinc(**II**)

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The title compound, $[Zn_4(C_{15}H_{16}N_2O_2)_4]$, is a novel tetranuclear zinc(II) complex with crystallographic fourfold inversion–rotation symmetry. Each Zn^{II} atom has a distorted square-pyramidal coordination geometry. In the basal plane, the Zn atom is coordinated by two N atoms and two O atoms of one Schiff base ligand. The apical position is occupied by an O atom of another Schiff base ligand. The four Zn atoms in the complex are linked through four bridging O atoms, forming an eight-membered ring.

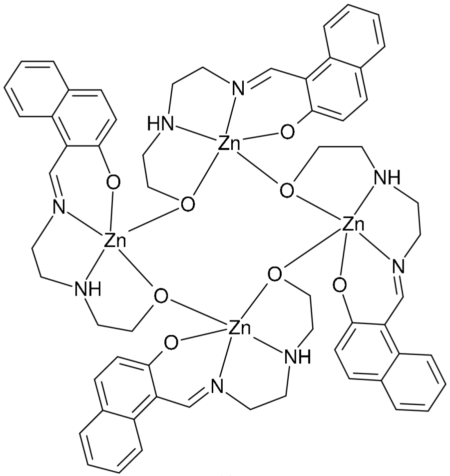
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.104
Data-to-parameter ratio = 16.8

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

Comment

Transition metal compounds containing Schiff base ligands have been of great interest for many years (Asada *et al.*, 2003; Zhu *et al.*, 2000; Yamada, 1999; Archer & Wang, 1990). These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures (Costamagna *et al.*, 1992; Bhatia *et al.*, 1981). Zinc, an important transition metal in biology, functions as the active site of hydrolytic enzymes, where it is in a hard-donor coordination environment of nitrogen and oxygen (Sanmartín *et al.*, 2000). Zinc has long been recognized as an important cofactor in biological molecules, either as a structural template in protein folding or as a Lewis acid catalyst that can readily adopt four-, five- or six-coordination (Vallee & Auld, 1993). Recently, we have reported a few Schiff base zinc(II) complexes (You *et al.*, 2003; You *et al.*, 2004). As an extension of our work on the structural characterization of Schiff base zinc complexes, the title tetranuclear zinc(II) complex, (I), is reported here.



(I)

The title compound, (I), is a tetranuclear zinc(II) complex, as shown in Fig. 1. The asymmetric unit of (I) consists of one-

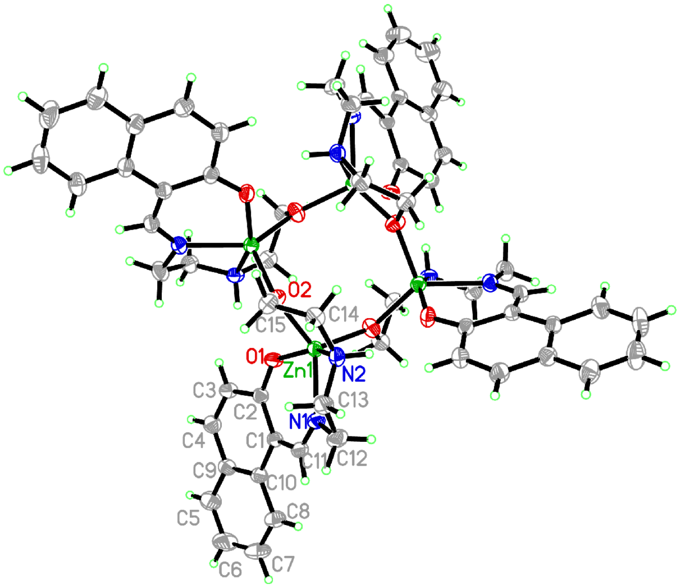


Figure 1
The structure of (I), showing the atom-numbering scheme of the asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level.

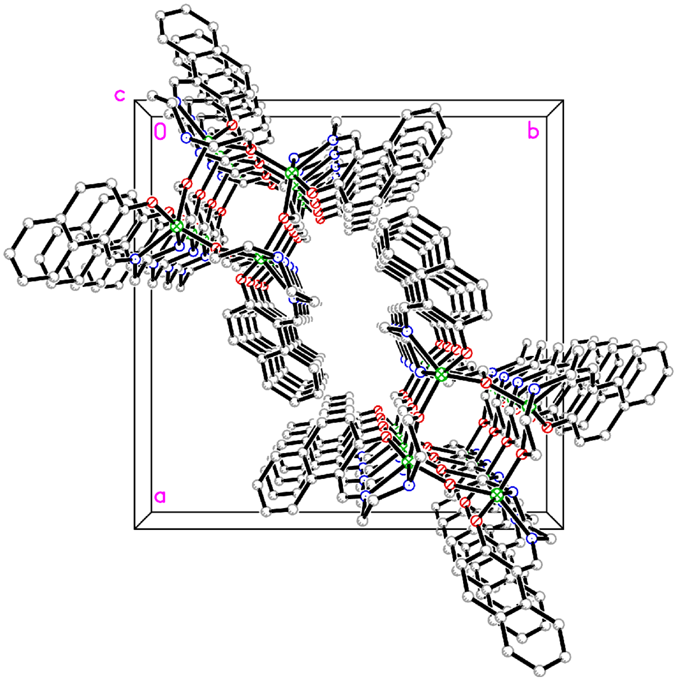


Figure 2
The crystal packing of (I), viewed along the *c* axis.

fourth of the title complex, with three-fourths generated by fourfold improper rotation ($\bar{4}$) symmetry. Each Zn atom has a square-pyramidal coordination polyhedron, coordinated by two N atoms and three O atoms of two Schiff base ligands. The significant distortion of the square pyramid is revealed by the bond angles between the apical and basal donor atoms (Table 1). The bond angles between the donor atoms in the basal positions show less distortion. The apical bond of the square pyramid is a little shorter than the basal bonds (Table 1). The Zn1—O2 bond length is a little longer than that of Zn1—O2^{i} [symmetry code: (i) $\frac{1}{2} - y$, x, $\frac{3}{2} - z$], which is probably caused by the strain created by the five-membered chelate ring Zn1/N2/C14/C15/O2.

The bond angles N1—Zn1—N2 and N2—Zn1—O2 deviate from 90° by 10.34 (8) and 8.22 (7)°, respectively. This is due to the strain created by the five-membered chelate rings, *viz*. Zn1/N1/C12/C13/N2 and Zn1/N2/C14/C15/O2. The conformation of the six-membered ring containing the metal, azomethine N atom, phenolate O atom and three C atoms (C11, C1 and C2) is a boat. The distances of the two *para*-positioned boat atoms, Zn1 and C1, from the mean plane of the other four atoms are 0.128 (2) and 0.107 (2) Å, respectively. Atoms C12 and C13 deviate by 0.222 (2) and −0.471 (2) Å, respectively, from the plane defined by atoms N1, Zn1 and N2. Atoms C14 and C15 deviate by −0.167 (2) and 0.456 (2) Å, respectively, from the plane defined by atoms N2, Zn1 and O2.

The four Zn atoms are linked together by four bridging O atoms to form an eight-membered ring. The r.m.s. deviation of the four Zn atoms from their mean plane is 0.134 (2) Å. Each adjacent Zn···Zn distance is 3.568 (2) Å.

An intramolecular N—H···O hydrogen bond is observed in the complex (Table 2). A view of the molecular packing in the crystal structure is shown in Fig. 2.

Experimental

N-(2-Hydroxyethyl)ethylenediamine (0.2 mmol, 20.8 mg) and salicylaldehyde (0.2 mmol, 24.4 mg) were dissolved in methanol (5 ml). The mixture was stirred for 10 min at room temperature to give a clear yellow solution. To the solution was added zinc oxide powder (0.2 mmol, 16.0 mg), and the mixture was stirred for another 10 min. The mixture was then transferred to a stainless steel bomb, which was sealed, heated at 423 K for 12 h, and cooled gradually to room temperature. Colourless block-shaped crystals were formed.

Crystal data

$[\mathrm{Zn(C_{15}H_{16}N_2O_2)_4}]$
$M_r = 1286.67$
Tetragonal, $P4_2/n$
$a = 18.230\ (3)$ Å
$c = 8.531\ (2)$ Å
$V = 2835.1\ (9)$ Å^3
$Z = 2$
$D_x = 1.507$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2123 reflections
$\theta = 2.3$–25.0°
$\mu = 1.73$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
0.23 × 0.21 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.691$, $T_{\max} = 0.819$
12763 measured reflections
3045 independent reflections
2242 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.086$
$\theta_{\max} = 27.0°$
$h = -15 \rightarrow 22$
$k = -22 \rightarrow 23$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.104$
$S = 0.97$
3045 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.55$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O2^{i}	1.9467 (17)	Zn1—N1	2.101 (2)
Zn1—O2	2.0065 (17)	Zn1—N2	2.176 (2)
Zn1—O1	2.0351 (17)		
O2^{i}—Zn1—O2	114.87 (11)	O1—Zn1—N1	84.64 (8)
O2^{i}—Zn1—O1	104.88 (7)	O2^{i}—Zn1—N2	100.54 (8)
O2—Zn1—O1	94.31 (7)	O2—Zn1—N2	81.78 (7)
O2^{i}—Zn1—N1	109.52 (9)	O1—Zn1—N2	153.42 (7)
O2—Zn1—N1	134.20 (8)	N1—Zn1—N2	79.66 (8)

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O1^{i}	0.91	2.03	2.849 (3)	150

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

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H = 0.93 or 0.97 Å and N—H = 0.90 Å, and with $U_{\rm iso}$(H) values fixed at 0.08 Å^2.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998; data reduction: *SAINT* ; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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