

Bis[μ -2-(1-butylbenzimidazol-2-yl)phenolato]- $1\kappa N^3:2\kappa O;1\kappa O:2\kappa N^3$ -bis[[2-(1-butylbenzimidazol-2-yl)phenolato- $\kappa^2 N^3,O$]zinc(II)] 2-(1-butylbenzimidazol-2-yl)phenol disolvate**Yi-Ping Tong,^a Yan-Wen Lin^b and Seik Weng Ng^{c*}**^aDepartment of Chemistry, Hanshan Normal College, 521041 Chaozhou, People's Republic of China, ^bDepartment of Biology, Hanshan Normal College, 521041 Chaozhou, People's Republic of China, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

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Key indicatorsSingle-crystal X-ray study
 $T = 295$ K
Mean $\sigma(C-C) = 0.005$ Å
Disorder in main residue
 R factor = 0.068
 wR factor = 0.196
Data-to-parameter ratio = 13.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The centrosymmetric dinuclear title compound, $[Zn_2(C_{17}H_{17}N_2O)_4] \cdot 2C_{17}H_{18}N_2O$, has one 2-(1-butylbenzimidazol-2-yl)phenolate acting as a chelating ligand and the other as a bridging ligand, conferring a tetrahedral geometry on the Zn atom. The uncoordinated 2-(1-butylbenzimidazol-2-yl)phenol molecule interacts with the O atom of the chelating anion through a hydrogen bond.

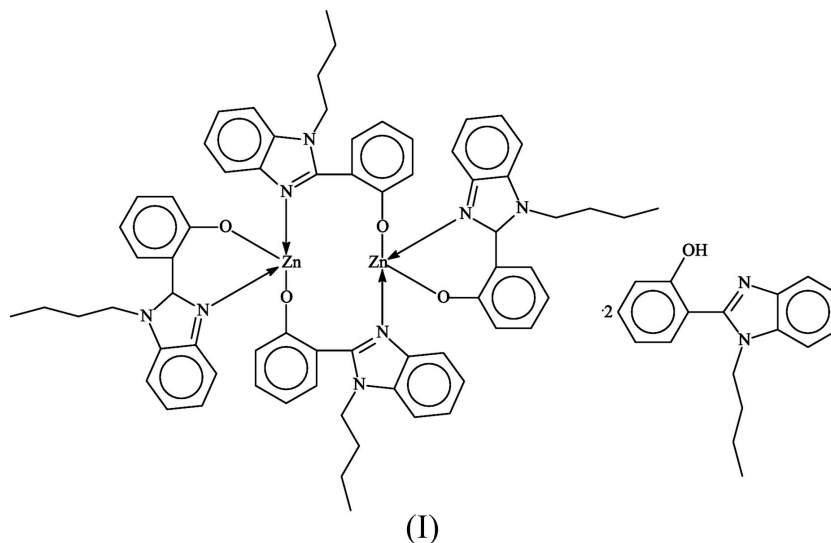
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Comment

Following a report on the exceptional electroluminescence of the dimeric zinc derivative of benzthiazol-2-ylphen-2-ol (Yu *et al.*, 2003), we have been able to synthesize the benzthiazol-2-ylphenol-2-ate/benzoxazol-2-ylphenol-2-ate, which is a mixed dinuclear compound (Tong *et al.*, 2004), the 5-amino-benzimidazol-2-ylphen-2-olate, which exists as a co-crystal with zinc bis(benzimidazol-2-yl-phen-2-olate) (Tong, 2005*a*), and the benzimidazol-2-yl-(4-bromophen-4-olate) (Tong, 2005*b*). The second and third are monomeric compounds that also exhibit luminescence. The title zinc derivative, (I), of 1-butylbenzimidazol-2-ylphen-2-ol exists as a centrosymmetric dimer, the dimer crystallizing with two molecules of the ligand (Fig. 1). One 1-butylbenzimidazol-2-ylphen-2-olate functions in an *O,N*-chelating mode [$Zn-O = 1.963(3)$ Å] and the other in an *O,N*-bridging mode [$Zn-O = 1.904(3)$ Å]; the longer zinc–oxygen bond length for the chelating anion suggests that chelation is probably sterically unfavorable. The Zn atom is four-coordinate in a tetrahedral geometry. The dinuclear molecule interacts with the uncoordinated heterocycle through a hydrogen bond [$O \cdots O = 2.660(4)$ Å].



Experimental

1-Butyl-1*H*-benzimidazol-2-yl-phen-2-ol was synthesized using the procedure for synthesizing 1-ethyl-1*H*-benzimidazol-2-ylphen-2-ol (Addison & Burke, 1981). Salicylic acid and *o*-phenylenediamine in syrupy phosphoric acid were heated at 520 K for 5 h; the product (in 10% yield) was then reacted with 1-bromobutane to form the desired heterocyclic compound in 70% yield. Zinc acetate dihydrate (0.022 g, 0.1 mmol), the heterocycle (0.064 g, 0.2 mmol), potassium hydroxide (0.020 g, 0.4 mmol) and water (10 ml) were heated in a 23 ml Teflon-lined Parr bomb at 368 K for 4 d. The bomb was cooled to room temperature at 5 K h⁻¹. Analysis found: C 71.14, H 6.21, N 9.62%; calculated for C₁₀₂H₁₀₄N₁₂O₆Zn₂: C 71.03, H 6.08, N 9.75%.

Crystal data

[Zn₂(C₁₇H₁₇N₂O)₄]₂·2C₁₇H₁₈N₂O
M_r = 1724.71
 Triclinic, *P* $\bar{1}$
a = 10.750 (1) Å
b = 11.839 (1) Å
c = 18.709 (2) Å
 α = 93.070 (2)°
 β = 105.927 (1)°
 γ = 103.382 (2)°
V = 2210.0 (3) Å³
Z = 1
D_x = 1.296 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 1973 reflections
 θ = 2.2–19.6°
 μ = 0.61 mm⁻¹
T = 295 (2) K
 Plate, colorless
 0.21 × 0.15 × 0.04 mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scan
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.448, *T_{max}* = 0.976
 11754 measured reflections
 7679 independent reflections
 5409 reflections with *I* > 2σ(*I*)
R_{int} = 0.028
 θ_{max} = 25.0°
h = -12 → 12
k = -14 → 13
l = -22 → 20

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.068
wR (*F*²) = 0.196
S = 1.02
 7679 reflections
 556 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1132P)^2 + 0.1952P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.52 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{min} = -0.36 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Zn1—O1	1.963 (3)	Zn1—N1	1.987 (3)
Zn1—O2	1.904 (3)	Zn1—N3 ⁱ	1.981 (3)
O1—Zn1—O2	111.3 (1)	O2—Zn1—N1	106.4 (1)
O1—Zn1—N1	92.5 (1)	O2—Zn1—N3 ⁱ	110.4 (1)
O1—Zn1—N3 ⁱ	111.9 (1)	N1—Zn1—N3 ⁱ	123.1 (2)

Symmetry code: (i) -x + 1, -y + 1, -z + 1.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H3O...O1	0.82	1.84	2.660 (4)	172

The 2-, 3- and 4-C atoms of the three butyl groups are disordered over two positions; the occupancies refined to 0.53 (1):0.47 (1),

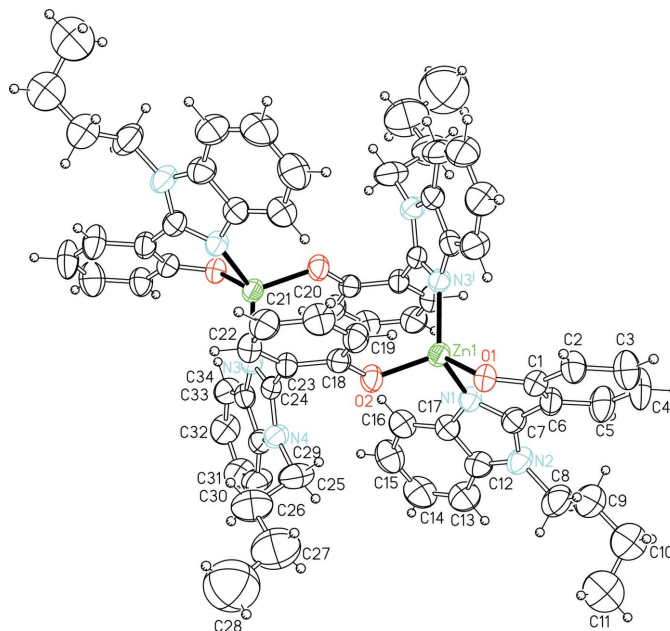


Figure 1

ORTEP (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 30% probability level. Only the major disorder component is shown; the uncoordinated heterocycle is not shown. H atoms are drawn as spheres of arbitrary radii. [Symmetry code (i): 1 - x, 1 - y, 1 - z.]

0.68 (1):0.32 (1) and 0.53 (1):0.47 (1). The occupancies for two of the three were set to equal each other to avoid any short interaction between the unprimed butyl group of the uncoordinated ligand and the primed butyl group of the coordinated ligand. The C—C distances were restrained to 1.540 (5) and the 1,3-related distances to 2.51 (1) Å. The vibrations of the disordered C atoms were restrained to be nearly isotropic; additionally, the displacement parameters of the disordered atoms along the butyl chains were restrained to increase only gradually. The benzene and *o*-phenylene rings were refined as rigid hexagons of 1.39 Å sides so as to increase the reflections-to-variables ratio; refining their atoms freely did not significantly lower the *R* index. H atoms were placed at calculated positions in the riding-model approximation (C—H_{aromatic} = 0.93 Å, C—H_{methylene} = 0.97 Å and C—H_{methyl} = 0.96 Å), with their displacement parameters tied to those of the parent atoms by *U*_{iso}(H) = 1.2–1.5*U*_{eq}(C). The hydroxy group was rotated to fit the electron density [O—H = 0.82 Å, *U*_{iso}(H) = 1.5*U*_{eq}(O)]. The unprimed butyl component of the chelating anion is 2.67 Å from the primed butyl component of the free ligand.

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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