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Crystallographic report

Bis(2-diphenylglycolato-*O*)bis(1,10-phenanthroline-*N*,*N*′)zinc(II), $[Zn(C_{14}H_{11}O_3)_2(C_{12}H_8N_2)_2]$

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The structure of $[Zn(HB)_2(1,10\text{-phen})_2]$ (HB = diphenylglycolato) comprises mononuclear molecules with the zinc(II) cation situated on a two fold axis and octahedrally coordinated by an N_4O_2 donor set. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; carboxylato complexes; zinc; $\pi-\pi$ interactions; C–H $\cdots$ π interactions

COMMENT

The investigation of the title compound $[Zn(HB)_2(1,10\text{-phen})_2]$ (**I**; HB = diphenylglycolato) is a contribution to the study of the solid-state coordination chemistry of mixed ligand complexes of divalent metals with α-hydroxycarboxylic acids.[1–4] In **I**, the zinc(II) atom lies on a two fold axis and the diphenylglycolato ligands are monodentate. The greatest deviation from the ideal octahedral geometry is found in the chelating angle N1–Zn–N2 of 74.08(6)°. No hydrogen bonds were found. In the lattice, molecules associate via $\pi-\pi$ and C–H $\cdots$ π interactions. In this way, an inter-ring distance of 3.6728(16) Å between two rings of neighbouring phenanthroline molecules (symmetry operation: $-x, 1-y, 1-z$) and a C109–H109 $\cdots$ centroid (Cg) of (C13–C18) interaction (d(H $\cdots$ Cg) = 2.69 Å; d(C $\cdots$ Cg) = 3.612(3)Å; $\langle$(CH $\cdots$ Cg) = 172°; symmetry operation: $\frac{1}{2}-x$, $\frac{3}{2}-y, 1-z$) are found. These interactions are responsible of the three-dimensional arrangement of the molecules in the crystal packing (Fig. 1b).

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EXPERIMENTAL

A solution of 2-diphenylglycolic acid (benzylic acid; 2.00 mmol) in ethanol (10 ml) was slowly added to a suspension of $ZnCO_3$ (1 mmol) in ethanol (10 ml). To the resulting white suspension was added a solution of 1,10-phenanthroline (2 mmol) in ethanol (10 ml). The colourless solution obtained was refluxed for 4 h and magnetic stirring was maintained at room temperature for 3 days. The white solid that formed was filtered off, washed with ethanol and dried *in vacuo*. Colourless single crystals of **I** suitable for X-ray diffraction studies were obtained by slow concentration of the filtrate. Yield: 73%; m.p. 248 °C; IR (KBr, cm^{-1}): ν(OH), 3425m,b, 3275m; ν_{asym}(OCO), 1623m; ν_{sym}(OCO), 1361s; ν(CO), 1171m; phenanthroline bands: 1644vs, 1515m, 1424m, 1050m; ν(ZnO), 412w; ν(ZnN), 240w. Anal. Found: C, 70.3; H, 4.5; N, 6.3. Calc. for $C_{52}H_{38}N_4O_6Zn$: C, 71.0; H, 4.4; N, 6.4%. Thermogravimetric analysis: two steps; T = 250–500 °C; gases evolved: CO_2, H_2O, CO, N_2O, NO, NO_2; final residue: $Zn(OH)_2$. Intensity data for **I** were collected at 293 K on a Bruker SMART CCD diffractometer for a colourless crystal 0.16 × 0.27 × 0.37 mm^3; $C_{52}H_{38}N_4O_6Zn$, M = 880.23, monoclinic, $C2/c$, a = 25.9169(19), b = 11.3327(9), c = 17.4743(13) Å, β = 123.701(1)°, V = 4269.8(6) $Å^3$, Z = 4; 4881 unique data (θ_{max} = 28.0°), R = 0.039 (2921 data with $I > 2\sigma(I)$), wR = 0.065 (all data), ρ_{max} = 0.41 e^- $Å^{-3}$. Programs used: SAINT, SHELXS97, SHELXL97 and PLATON. CCDC deposition number: 225407.

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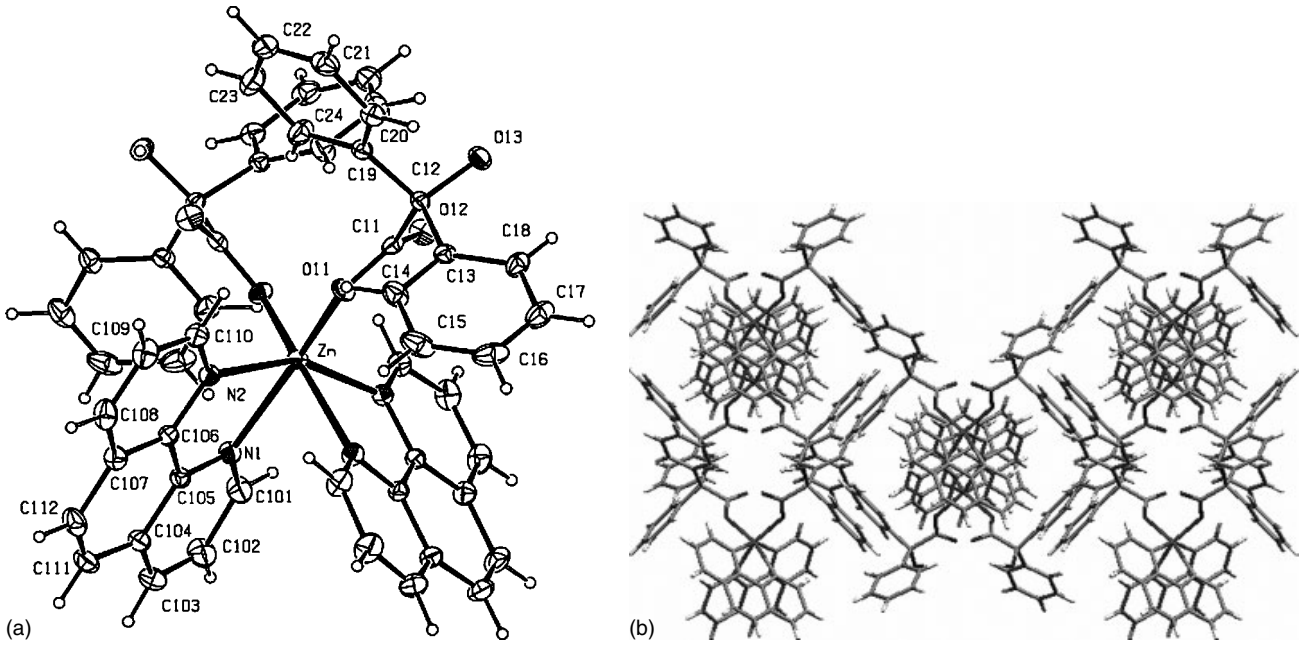

Figure 1. (a) Molecular structure of **I**. Key geometric parameters: Zn–O11 2.0094(12), Zn–N1 2.2996(16), Zn–N2 2.1586(15) Å; O11–Zn–N1 161.93(6), O11–Zn–N2 89.73(6), O11–Zn–O11^{i} 102.21(8), O11–Zn–N1^{i} 90.27(6), O11–Zn–N2^{i} 106.38(5), N1–Zn–N2 74.08(6), N1–Zn–N1^{i} 80.94(8), N1–Zn–N2^{i} 86.46(6), N2–Zn–N2^{i} 154.48(9)°. Symmetry operation: $i = -x, y, \frac{1}{2} - z$. (b) View of the three-dimensional network in **I**.

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