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Tetrakis(μ-benzoato)bis[(*N*,*N*-diethylnicotinamide)-zinc(II)]

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.072
Data-to-parameter ratio = 13.5

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

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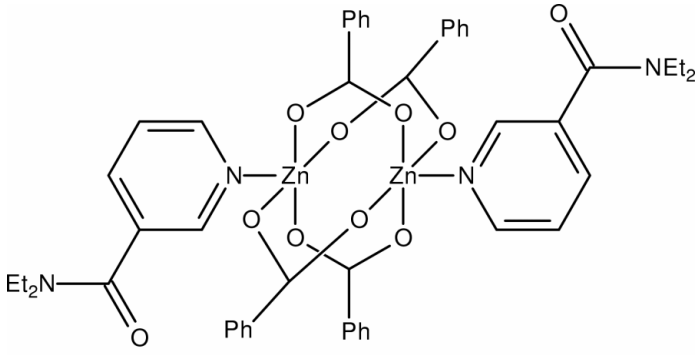
The title compound, $[Zn_2(C_7H_5O_2)_4(C_{10}H_{14}N_2O)_2]$, has a centrosymmetric dimeric molecule with four symmetrical benzoate bridges and two axial nitrogen-base ligands. The Zn···Zn separation of 2.8860 (4) Å does not represent a formal direct metal–metal bond. Coordination of zinc is square pyramidal.

Comment

The title compound, (I), was prepared as part of a series of studies of the coordination chemistry of the ligand *N*,*N*-diethylnicotinamide (DENA), particularly in conjunction with carboxylate ligands.



(I)

Crystallographic investigation shows that the molecule is a centrosymmetric dimer (Fig. 1), in which the two zinc ions are bridged by four benzoate ligands. DENA acts as a terminal monodentate ligand through its pyridine N atom, with one DENA ligand attached to each end of the Zn···Zn axis. Such a dimeric arrangement with two metal ions bridged by four carboxylates is common for transition metals, whether the metals have a direct formal metal–metal bond or not, but it has been observed for only four previous zinc complex structures, two of them being polymorphs of the same complex (Clegg *et al.*, 1986, 1995; Singh *et al.*, 1997).

The two zinc ions are not considered to be directly bonded to each other in this structure. The Zn···Zn separation of 2.8660 (4) Å is the shortest observed for zinc complexes of this kind, previous values ranging from 2.893 to 2.976 Å. The benzoate bridges are essentially symmetrical, with no significant variation in the C—O bond lengths. The central $Zn_2(O_2C)_4N_2$ core of the molecule has approximate 4/*mmm* (D_{4h}) symmetry. Each Zn atom has square-pyramidal coordination, with DENA in the apical site.

There are no significant intermolecular interactions.

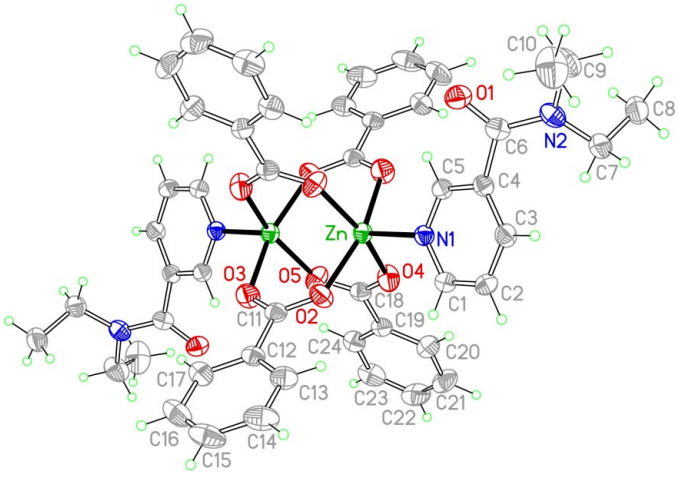


Figure 1
The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms.

Experimental

The title complex was prepared by the reaction of zinc benzoate (10 mmol) and diethylnicotinamide (20 mmol) in 100 ml of water. The reaction mixture was filtered and set aside for crystallization at room temperature for several days, during which time colourless single crystals were deposited.

Crystal data

$[Zn_2(C_7H_5O_2)_4(C_{10}H_{14}N_2O)_2]$
M_r = 971.64
Orthorhombic, *Pbca*
a = 19.5680 (19) Å
b = 10.6989 (10) Å
c = 21.617 (2) Å
V = 4525.6 (8) Å^3
Z = 4
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 194 reflections
θ = 2.1–25.6°
μ = 1.12 mm^{-1}
T = 160 (2) K
Block, colourless
0.32 × 0.26 × 0.26 mm

Data collection

Siemens SMART 1K CCD diffractometer
ω scans with narrow frames
Absorption correction: multi-scan (*SHELXTL*; Sheldrick, 1997)
T_{min} = 0.683, T_{max} = 0.782
18 548 measured reflections
3940 independent reflections
3659 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.7°
$h = -23 \rightarrow 20$
$k = -12 \rightarrow 12$
$l = -22 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.07
3940 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 2.6031P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn—N1	2.0254 (14)	O2—C11	1.257 (2)
Zn—O2	2.0530 (13)	O3—C11	1.256 (2)
Zn—O3^i	2.0417 (14)	O4—C18	1.259 (2)
Zn—O4	2.0130 (14)	O5—C18	1.255 (2)
Zn—O5^i	2.0471 (13)		
N1—Zn—O2	96.93 (6)	O2—Zn—O4	88.87 (6)
N1—Zn—O3^i	100.94 (6)	O2—Zn—O5^i	87.82 (6)
N1—Zn—O4	101.71 (6)	O3^i—Zn—O4	89.35 (6)
N1—Zn—O5^i	96.31 (6)	O3^i—Zn—O5^i	88.34 (6)
O2—Zn—O3^i	162.03 (6)	O4—Zn—O5^i	161.95 (6)

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

The data at the highest angles are incomplete because of a relatively large crystal-to-detector distance in an early experiment with one of the first commercial CCD diffractometers before operating parameters were optimized; nevertheless, data are essentially complete to θ = 24°. H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

Data collection: *SMART* (Siemens, 1995); cell refinement: local programs; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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