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Diaquabis(*N*,*N*-diethylnicotinamide)bis(2-hydroxybenzoato)zinc(II)

Hacali Necefoglu,[a] William Clegg[b]* and Andrew J. Scott[b]

[a]Department of Chemistry, Faculty of Science and Letters, Kafkas University, 36100 Kars, Turkey, and [b]Department of Chemistry, University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England

Correspondence e-mail: w.clegg@ncl.ac.uk

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The title compound, $[Zn(C_7H_5O_3)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$, crystallizes as centrosymmetric mononuclear molecules with octahedrally coordinated zinc, all pairs of equivalent ligands being mutually *trans*. Intramolecular hydrogen bonding links each aqua ligand and the hydroxy group of a 2-hydroxybenzoate ligand with the uncoordinated carboxylate O atom of the same carboxylate ligand, lengthening this C—O bond so that it has almost the same length as that for the coordinated O atom. Intermolecular hydrogen bonding between aqua ligands and the carbonyl O atoms of N,*N*-diethylnicotinamide ligands link the molecules together into chains.

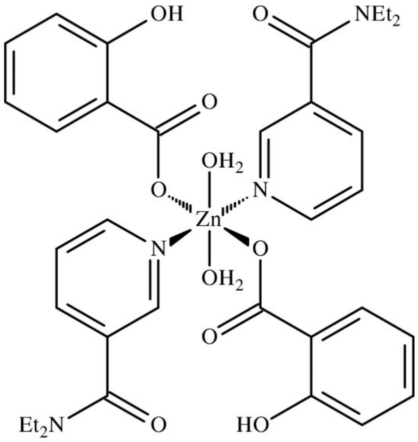
Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.007 Å
R factor = 0.050
wR factor = 0.128
Data-to-parameter ratio = 10.8

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

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). It has a relatively undistorted octahedral coordination geometry for the Zn atom, which lies on an inversion centre, so that all pairs of equivalent ligands lie mutually *trans* (Fig. 1). A rigorously centrosymmetric structure has been found for all previously reported metal complexes of DENA having a formula $[M(OH_2)_2(DENA)_2L_2]$, where L is any monodentate ligand (Bigoli *et al.*, 1973; Hökelek & Necefoglu, 1997; Hökelek *et al.*, 1997; Melnik *et al.*, 1996; Sergienko *et al.*, 1979, 1980).



(I)

The carboxylates act as monodentate ligands, but the near equality of the two C—O bond lengths in each carboxylate group (Table 1) indicate a delocalized bonding arrangement rather than localized single and double bonds. This may be due, in part, to the involvement of the uncoordinated O4 atom as acceptor of two hydrogen bonds. Both of these are intramolecular: one is within the ligand, with the *ortho*-hydroxy

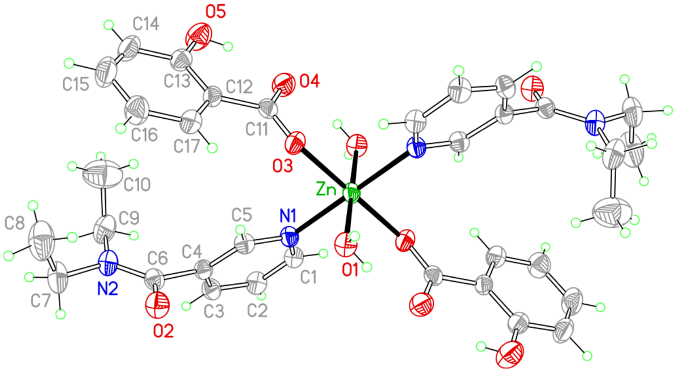


Figure 1
The molecular structure of (I) with atom labels and 30% probability ellipsoids for non-H atoms.

group O5 acting as donor, while the other is between ligands, the donor being an aqua ligand (Table 2). The aqua ligand also acts as donor in an intermolecular hydrogen bond, and pairs of these link molecules together into a chain along the *a* axis, as shown in Fig. 2.

We have found very similar features, but without the intraligand hydrogen bond, in the corresponding complex with 4-nitrobenzoate replacing 2-hydroxybenzoate (Necefoglu *et al.*, 2001).

Experimental

The title compound was prepared by the reaction of 0.01 mol of zinc salicylate and 0.02 mol of diethylnicotinamide in 80 ml water. The mixture was filtered and set aside for crystallization at ambient temperature for several days, giving suitable colourless single crystals.

Crystal data

$[Zn(C_7H_5O_3)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$
$M_r = 732.09$
Triclinic, $P\overline{1}$
$a = 7.652$ (7) Å
$b = 8.903$ (6) Å
$c = 14.664$ (16) Å
$\alpha = 97.38$ (8)°
$\beta = 95.06$ (7)°
$\gamma = 114.22$ (6)°
$V = 892.5$ (14) Å^3
$Z = 1$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 61 reflections
$\theta = 1.0$–23.5°
$\mu = 0.75$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.37 × 0.26 × 0.22 mm

Data collection

Bruker SMART 1K CCD diffractometer
ω rotation scans with narrow frame
Absorption correction: multi-scan (*XPREP*; Sheldrick, 1997)
$T_{min} = 0.75$, $T_{max} = 0.85$
3534 measured reflections
2523 independent reflections
2425 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 23.3°$
$h = -8 \rightarrow 4$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.128$
$S = 1.12$
2523 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 1.2437P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

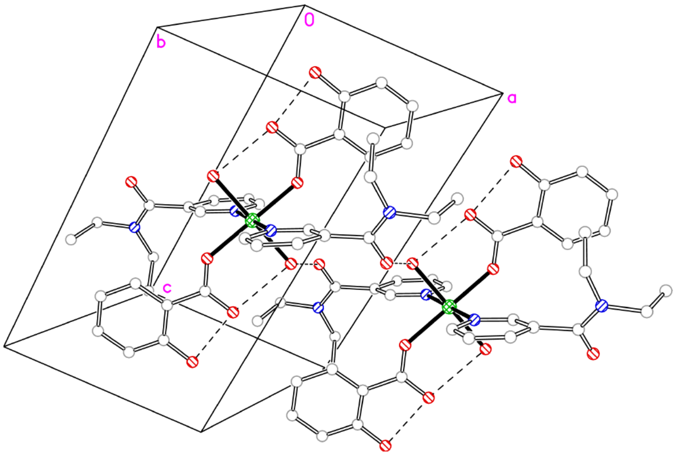


Figure 2
Two molecules showing intramolecular and intermolecular hydrogen bonds and the unit-cell outline.

Table 1
Selected geometric parameters (Å, °).

Zn—O1	2.153 (4)	C11—O3	1.259 (5)
Zn—O3	2.098 (3)	C11—O4	1.266 (5)
Zn—N1	2.154 (4)		
O3—Zn—O1	87.07 (14)	O1—Zn—N1	87.13 (14)
O3—Zn—N1	88.81 (14)	C11—O3—Zn	127.7 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$A\cdots$O2^{i}	0.81 (4)	2.01 (4)	2.817 (5)	175 (6)
O1—H1$B\cdots$O4^{ii}	0.81 (4)	1.90 (4)	2.698 (5)	166 (6)
O5—H5$A\cdots$O4	0.82	1.82	2.551 (5)	147

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$.

Data were collected at room temperature, because attempts to cool the crystals led to degradation of the crystal quality and the diffraction pattern, possibly as a result of a phase transition. Since this was an early experiment with one of the first commercial CCD diffractometers, operating parameters were not yet optimized; one consequence is the rather low maximum θ, as a result of a crystal-to-detector distance of approximately 6 cm. 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, except for the aqua ligands; for these, the H atoms were refined with the restraint of equal O—H bond lengths and with individual isotropic displacement parameters.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; 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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