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Diaquabis(*N*,*N*-diethylnicotinamide)bis(4-nitrobenzoato)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 = 0.026
wR factor = 0.068
Data-to-parameter ratio = 12.8

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

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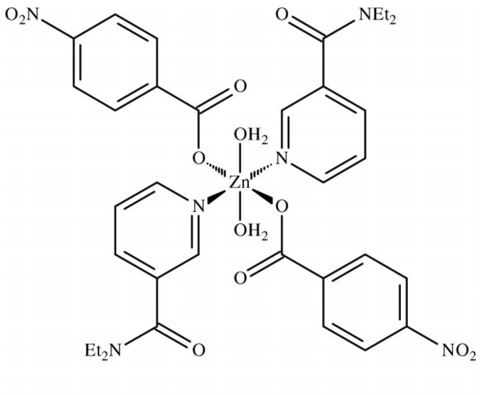
The title compound, $[Zn(C_7H_4NO_4)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$, crystallizes as centrosymmetric mononuclear molecules with octahedrally coordinated zinc, all pairs of equivalent ligands being mutually *trans*. Intramolecular hydrogen bonding links each aqua ligand with an uncoordinated carboxylate O atom, and intermolecular hydrogen bonding between aqua ligands and the carbonyl O atoms of *N*,*N*-diethylnicotinamide ligands link the molecules together into chains.

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 molecular structure (Fig. 1) very similar to that of the corresponding complex in which 2-hydroxybenzoate (salicylate) replaces 4-nitrobenzoate, reported in a parallel publication (Necefoglu *et al.*, 2001). Coordination of zinc is close to regular octahedral, with all pairs of equivalent ligands mutually *trans* across a crystallographic inversion centre.



(I)

Each aqua ligand is involved as donor in two hydrogen bonds. One of these is intramolecular, to an uncoordinated carboxylate O atom, lengthening the C═O nominal double bond to almost the same as C—O for the coordinated O atom. The other is intermolecular, linking the molecules together in chains along the *a* axis.

Experimental

The title compound was prepared by the reaction of 4.34 g (0.01 mol) of $[Zn(OH_2)_2(4\text{–}O_2NC_6H_4COO)_2]$ and 2.56 g (0.02 mol) of diethylnicotinamide in 100 ml water. The solution was filtered and set aside for crystallization at ambient temperature for a few days. Suitable colourless crystals were obtained.

Crystal data

$[Zn(C_7H_4NO_4)_2(C_{10}H_{14}N_2O)_2$-
$(H_2O)_2]$
$M_r = 790.09$
Triclinic, $P\overline{1}$
$a = 7.211$ (3) Å
$b = 8.588$ (4) Å
$c = 16.515$ (7) Å
$\alpha = 96.00$ (2)°
$\beta = 100.79$ (2)°
$\gamma = 112.09$ (3)°
$V = 913.6$ (7) Å^3

$Z = 1$
$D_x = 1.436$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 36
reflections
$\theta = 10.7$–12.5°
$\mu = 0.74$ mm^{-1}
$T = 160$ (2) K
Block, colourless
0.42 × 0.27 × 0.19 mm

Data collection

Stoe–Siemens four-circle
diffractometer
ω/θ scans with on-line profile fitting
(Clegg, 1981)
Absorption correction: ψ scan
(*XPREP*; Sheldrick, 1997)
$T_{min} = 0.821$, $T_{max} = 0.959$
6298 measured reflections
3214 independent reflections

2889 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0$°
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -19 \rightarrow 19$
5 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.068$
$S = 1.10$
3214 reflections
251 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2$
$+ 0.4095P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn—O1	2.1373 (15)	C11—O3	1.264 (2)
Zn—O3	2.1149 (14)	C11—O4	1.255 (2)
Zn—N1	2.1527 (16)		
O3—Zn—O1	87.54 (6)	O1—Zn—N1	86.71 (6)
O3—Zn—N1	88.54 (6)	C11—O3—Zn	125.41 (11)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O4^{i}	0.86 (3)	1.82 (3)	2.659 (2)	164 (3)
O1—H1*B*···O2^{ii}	0.81 (3)	1.95 (3)	2.765 (2)	173 (2)

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

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 aqua ligand H atoms, which were refined freely with individual U_{iso}.

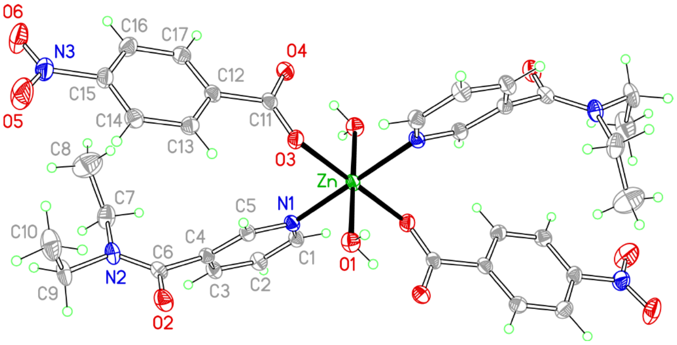


Figure 1
The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms.

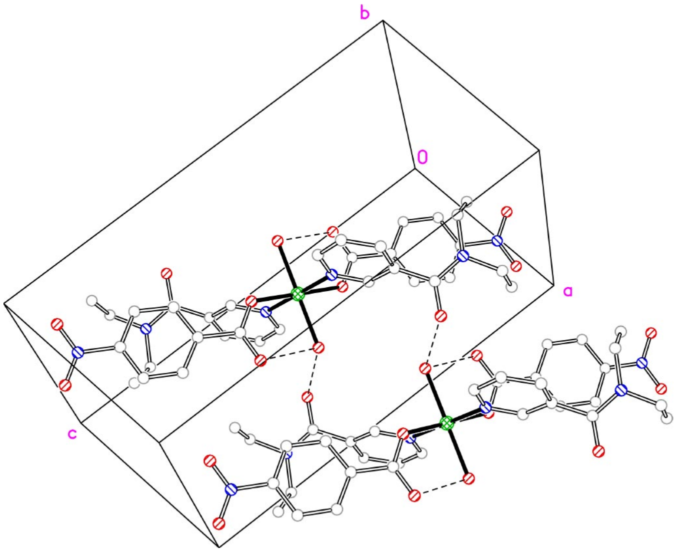


Figure 2
View of two molecules and the unit-cell outline, showing intramolecular and intermolecular hydrogen bonding as dashed lines.

Data collection: *DIF*4 (Stoe & Cie, 1988); cell refinement: *DIF*4; data reduction: local programs; 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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References

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.
Necefoglu, H., Clegg, W. & Scott, A. J. (2001). *Acta Cryst.* E**57**, m462–m464.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Stoe & Cie (1988). *DIF*4. Version 7.04. Stoe & Cie, Darmstadt, Germany.

m466 H. Necefoglu *et al.* • $[Zn(C_7H_4NO_4)_2(C_{10}H_{14}N_2O)_2(H_2O)_2]$ *Acta Cryst.* (2001). E**57**, m465–m466