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Poly[[diaquadinitratozinc(II)]bis(μ-1,4-di-3-pyridyl-2,3-diaza-1,3-butadiene)]

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catena-Poly[[diaquadinitratozinc(II)]bis(μ-1,4-di-3-pyridyl-2,3-diaza-1,3-butadiene)]

Shakoya Paulin, Pierre Kelly, Kenneth B. Williams, Andrea M. Goforth, Mark D. Smith, LeRoy Peterson Jr and Hans-Conrad zur Loye
The crystal structure of (I) is built upon neutral Zn(L2)2(OH)2(NO3)2 units (Fig. 1). The ZnII ion is located on an inversion center and is situated in a distorted N2O4 octahedral coordination environment. The axial positions are occupied by two N atoms from pairs of equivalent L2 ligands. The equatorial positions are occupied by four O atoms, from equivalent pairs of water molecules and two O atoms from equivalent pairs of monodentate nitrates (Table 1). For L2, the two pyridyl rings N1/C1–C5 and N4/C8–C12 are twisted at a dihedral angle of 34.6 (1)°. As expected for the nitrate, the N—O bond corresponding to the coordinated O atom is slightly longer than the other two N—O bonds (Table 1). One of the uncoordinated nitrate O atoms is...
involved in an intramolecular O4—H4B···O2 hydrogen bond (Table 2) to a coordinated water molecule located on the same ZnII center.

It is noteworthy that only one of the two pyridyl N atoms of L2 directly coordinates a ZnII ion. The other pyridyl N atom interacts indirectly with an adjacent ZnII ion by forming an outer-sphere O4—H4A···N4 hydrogen bond (Table 2) with a coordinated water molecule located on the adjacent ZnII center. This interaction, along with the inner-sphere ZnII···N2 coordination bond noted previously, generates a double chain structure (Fig. 2) involving two equivalent L2 ligands. The chains run along the [111] direction with a non-bonded ZnII···ZnII distance of 15.578 (1) Å. To our knowledge, the generation of such a double chain structure involving both inner- and outer-sphere coordination by L2 is the first of its kind for this ligand.

**Experimental**

All chemicals and solvents were purchased from commercial sources and used without further purification. The L2 ligand (Dong et al., 2000) was prepared as previously described. Complex (I) was obtained by slow diffusion of an ethanol solution containing zinc nitrate hexahydrate (0.50 mmol) into a dichloromethane solution (8 ml) containing a mixture of L2 (1.0 mmol) and of 4,40'-bipyridine (1.0 mmol). A mixture of yellow, irregularly shaped crystals of (I) and colorless bar-shaped crystals of formula [ZnII(4,4'-bipyridine)2(ONO2)2CH2Cl2]n were obtained at the interface of the two solutions after several weeks.

**Crystal data**

[Zn(NO3)2(C24H20N8)(H2O)2] V = 691.12 (16) Å³

Z = 1

Dc = 1.556 Mg m⁻³

Mo Kα radiation

μ = 0.96 mm⁻¹

T = 150 (1) K

Irregular fragment, yellow

0.40 × 0.26 × 0.14 mm

**Data collection**

Bruker SMART APEX CCD
diffractometer

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2001)

Tmin = 0.670, Tmax = 0.870

6421 measured reflections

2831 independent reflections

2720 reflections with I > 2σ(I)

Rint = 0.027

θmax = 26.4°

**Refinement**

Refinement on F²

wR(F²) = 0.029

S = 1.07

2831 reflections

205 parameters

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(Fo²) + (0.0472P)² + 0.1395P]

where P = (Fo² + 2Fc²)/3

(Δ/σ)max < 0.001

Δρmax = 0.28 e Å⁻³

Δρmin = −0.27 e Å⁻³

Extinction correction: SHELXL97

Extinction coefficient: 0.026 (4)

**Table 1**

Selected geometric parameters (Å, °).

| ZnI—O1 | 2.1839 (12) | N5—O3 | 1.236 (2) |
| ZnI—O4 | 2.0795 (12) | N5—O2 | 1.239 (2) |
| ZnI—N1 | 2.1487 (13) | N5—O1 | 1.2653 (18) |
| O1—ZnI—O1i | 180 | O4—ZnI—N1 | 89.21 (5) |
| O4—ZnI—O1 | 95.05 (5) | N1—ZnI—O1i | 87.98 (5) |
| O4'—ZnI—O1 | 84.95 (5) | N1'—ZnI—N1 | 180 |
| O4—ZnI—O4' | 180 |

Symmetry code: (i) −x + 1, −y + 1, −z.
Table 2  
Hydrogen-bond geometry (Å, º).

<table>
<thead>
<tr>
<th>D—H</th>
<th>D···A</th>
<th>H···A</th>
<th>D···A</th>
<th>D—H···A</th>
</tr>
</thead>
<tbody>
<tr>
<td>O4—H4A···N4ii</td>
<td>0.79 (3)</td>
<td>1.97 (3)</td>
<td>2.750 (2)</td>
<td>170 (2)</td>
</tr>
<tr>
<td>O4—H4B···O2</td>
<td>0.78 (3)</td>
<td>2.29 (3)</td>
<td>2.856 (2)</td>
<td>130 (2)</td>
</tr>
</tbody>
</table>

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

The water-bound H atoms were refined without constraint; see Table 2. The remaining H atoms were included in the riding-model approximation, with C—H = 0.95 Å and Uiso(H) = 1.2Ueq(C).

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2000) and DIAMOND (Brandenburg, 2005); software used to prepare material for publication: SHELXTL.

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References


