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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.028
 wR factor = 0.089
Data-to-parameter ratio = 15.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[1,3-dihydroxy-2-hydroxymethyl-2-(5-nitro-
2-oxidobenzylideneamino)propane- $\kappa^3\text{N},\text{O},\text{O}'$]-
zinc(II) pyridine solvate

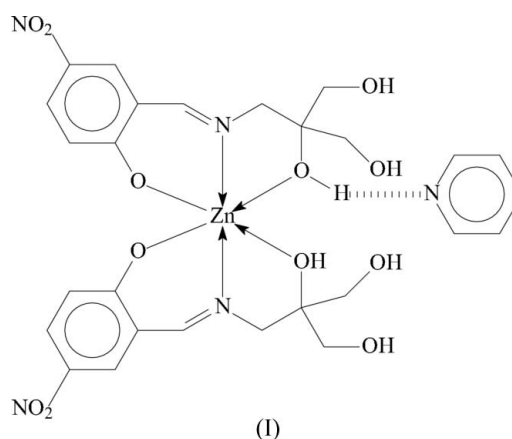
The Zn^{II} atom in the title complex, $[\text{Zn}(\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_6)_2] \cdot \text{C}_5\text{H}_5\text{N}$, is chelated by a terdentate Schiff base ligand in a slightly distorted octahedral geometry. One of the coordinated hydroxyl groups forms a hydrogen bond with the pyridine solvent molecule. In the crystal structure, other hydroxyl groups are engaged in intermolecular hydrogen bonding, forming a two-dimensional layer.

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Comment

The crystal structures of several metal complexes of the Schiff base synthesized by condensing salicylaldehyde (and its substituted derivatives) with tris(hydroxymethyl)amino-methane have been reported; among these are two zinc compounds, the unsubstituted (Dey *et al.*, 2002) and 5-formyl-substituted (Sah *et al.*, 2003) complexes, both of which crystallize as methanol solvates. The latter complex is reported to be water-soluble. The title 5-nitro substituted complex exists as a pyridine solvate, (I) (Fig. 1). Two deprotonated Schiff base ligands chelate to the metal through phenoxy O, imino N and hydroxyl O atoms in a slightly distorted octahedral environment. One of the coordinated hydroxyl groups forms a hydrogen bond with the pyridine solvent molecule. In the crystal structure, other hydroxyl groups are engaged in extensive hydrogen bonding with the hydroxyl groups of neighboring molecules, leading to a tightly held two-dimensional layer (Table 2).



Experimental

1,3-Dihydroxy-2-hydroxymethyl-2-(2-hydroxy-5-nitrobenzylideneamino)propane was synthesized from tris(hydroxymethyl)amino-methane and 5-nitrosalicylaldehyde according a literature procedure (Chumakov *et al.*, 2003, 2005). This compound (0.23 g, 0.85 mmol) was dissolved in ethanol (25 ml) and several drops of

aqueous sodium hydroxide were added to raise the pH of the solution to about 8.5. Zinc acetate (0.09 g, 0.43 mmol) was then added and the mixture heated for 5 h. The solvent was removed and the product recrystallized from pyridine.

Crystal data

[Zn(C₁₁H₁₃N₂O₆)₂].C₅H₅N
M_r = 682.94
 Monoclinic, *P*2₁/*c*
a = 11.3298 (2) Å
b = 11.4928 (2) Å
c = 22.0450 (3) Å
 β = 101.348 (1)°
V = 2814.39 (8) Å³

Z = 4
D_x = 1.612 Mg m⁻³
 Mo *K*α radiation
 μ = 0.95 mm⁻¹
T = 173 (2) K
 Prism, pale yellow
 0.23 × 0.15 × 0.12 mm

Data collection

Bruker APEXII area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.716, *T_{max}* = 0.895

41884 measured reflections
 6431 independent reflections
 5457 reflections with *I* > 2σ(*I*)
R_{int} = 0.038
 θ_{max} = 27.5°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.028
wR (*F*²) = 0.089
S = 1.10
 6431 reflections
 430 parameters
 H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(*F_o*²) + (0.051*P*)² + 1.1321*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.61 e Å⁻³
 Δρ_{min} = -0.51 e Å⁻³

Table 1 Selected geometric parameters (Å, °).

Zn1—O1	2.037 (1)	Zn1—O10	2.214 (1)
Zn1—O4	2.168 (1)	Zn1—N2	2.072 (2)
Zn1—O7	2.065 (1)	Zn1—N4	2.068 (2)
O1—Zn1—O7	93.06 (5)	O4—Zn1—N4	91.77 (5)
O1—Zn1—O4	167.42 (5)	O7—Zn1—O10	164.10 (5)
O1—Zn1—O10	87.41 (5)	O7—Zn1—N2	99.39 (5)
O1—Zn1—N2	91.01 (5)	O7—Zn1—N4	87.30 (5)
O1—Zn1—N4	99.68 (5)	O10—Zn1—N2	96.50 (5)
O4—Zn1—O7	92.66 (5)	O10—Zn1—N4	76.97 (5)
O4—Zn1—O10	90.14 (5)	N2—Zn1—N4	167.11 (6)
O4—Zn1—N2	77.00 (5)		

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H40...N5	0.85 (1)	1.84 (1)	2.681 (2)	171 (3)
O5—H50...O6 ⁱ	0.85 (1)	1.78 (1)	2.604 (2)	164 (2)
O6—H60...O7 ⁱⁱ	0.85 (1)	1.77 (1)	2.618 (2)	175 (3)
O10—H100...O12 ⁱⁱⁱ	0.84 (1)	1.97 (1)	2.766 (2)	158 (3)
O11—H110...O1 ⁱⁱⁱ	0.84 (1)	1.98 (1)	2.803 (2)	169 (2)
O12—H120...O5 ^{iv}	0.85 (1)	1.92 (1)	2.745 (2)	163 (3)

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x - 1, y, z$.

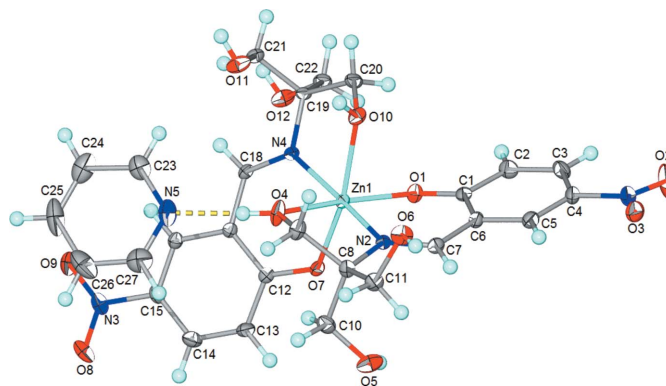


Figure 1 The molecular structure of (I), with displacement ellipsoids drawn at the 70% probability level and H atoms shown as spheres of arbitrary radii. The dashed line denotes a hydrogen bond.

Carbon-bound H atoms were placed in calculated positions (*C*—*H* = 0.95–0.99 Å) and were included in the refinement in the riding-model approximation, with *U_{iso}*(*H*) = 1.2*U_{eq}*(*C*). The hydroxyl H atoms were located in a difference Fourier map, and were refined with a distance restraint [*O*—*H* = 0.85 (1) Å] and isotropic displacement parameters.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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