

**R. Seth Baker and
Richard E. Norman***Chemistry Department, CNSB-210, University of
Louisiana at Monroe, Monroe, LA 71209, USA

Correspondence e-mail: rnorman@ulm.edu

Key indicatorsSingle-crystal X-ray study
 $T = 102$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
Disorder in main residue
 R factor = 0.073
 wR factor = 0.150
Data-to-parameter ratio = 17.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**Bis(acetato- κO)[bis(2-pyridylmethyl)amine- $\kappa^3\text{N},\text{N}',\text{N}''$]zinc(II)–acetic acid (1/2)**

The title compound, $[\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{12}\text{H}_{13}\text{N}_3)] \cdot 2\text{C}_2\text{H}_4\text{O}_2$, consists of a five-coordinate trigonal-bipyramidal zinc complex (one $\text{N},\text{N}',\text{N}''$ -tridentate donor and two O-monodentate donors) and two acetic acid molecules. The acetic acid molecules hydrogen bond with the coordination complex. The coordination complex is located on a twofold axis that passes through the Zn atom and the secondary amine N atom is disordered across that axis.

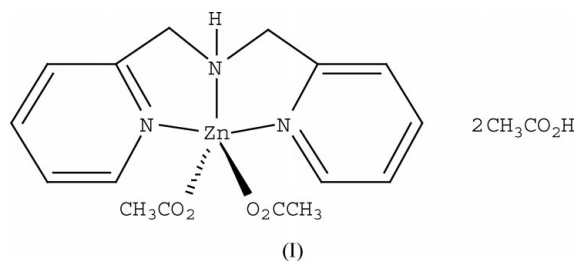
Received 19 October 2004

Accepted 28 October 2004

Online 6 November 2004

Comment

We obtained the title compound as an unexpected crystalline product during a literature preparation of bis(2-pyridylmethyl)amine (DPA) (Matouzenko *et al.*, 1997). The synthesis of the non-solvated form $[\text{Zn}(\text{DPA})(\text{OAc})_2]$ and its ^1H NMR spectrum were previously reported (Wirbser & Vahrenkamp, 1992).



The structure consists of a five-coordinate zinc(II) coordination complex of bis(2-pyridylmethyl)amine (DPA) (bonding to Zn through all three N atoms) with two acetate ligands, and two acetic acid molecules; half of the coordination complex and one solvent molecule comprise the asymmetric unit. The coordination complex is located on a twofold axis that passes through the Zn atom and the secondary amine N atom is disordered across that axis. This disorder renders discussion of distances and angles involving N2, other than the Zn–N distance, problematic. That being said, the other distances and angles of the Zn(DPA) moiety are within the ranges reported for similar complexes (Brand *et al.*, 1996; Glerup *et al.*, 1992; Gross *et al.*, 2000; Wirbser & Vahrenkamp, 1992). The coordination geometry has a degree of trigonality (τ) of 0.983 (Addison *et al.*, 1984); consequently, the geometry is described as trigonal bipyramidal. The Zn–O bond [2.019 (2) Å] is longer than is typical for Zn–acetate complexes; in di(acetato- O)[2,6-bis(aminomethyl)pyridine- $\text{N},\text{N}',\text{N}''$]zinc (Molenveld *et al.*, 1999), which is also a five-coordinate complex with an N_3O_2 donor set, the analogous distances are 1.9623 (14) and 1.9883 (18) Å. However, the Zn–O distance falls into the normal range reported for

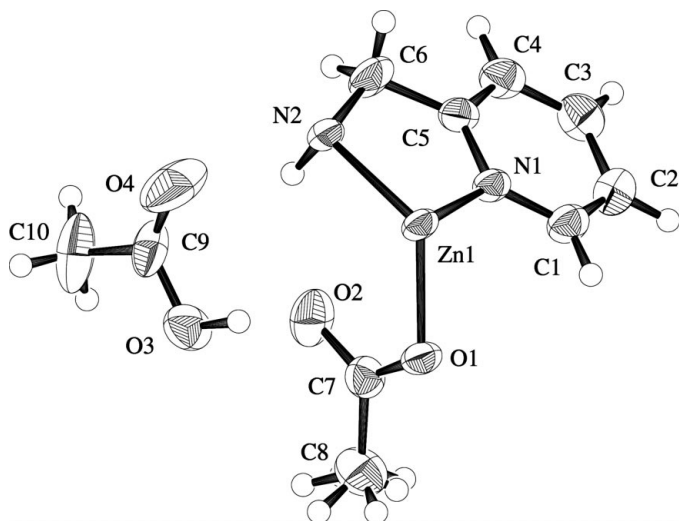


Figure 1
View of the asymmetric unit of (I), with displacement ellipsoids shown at the 50% probability level.

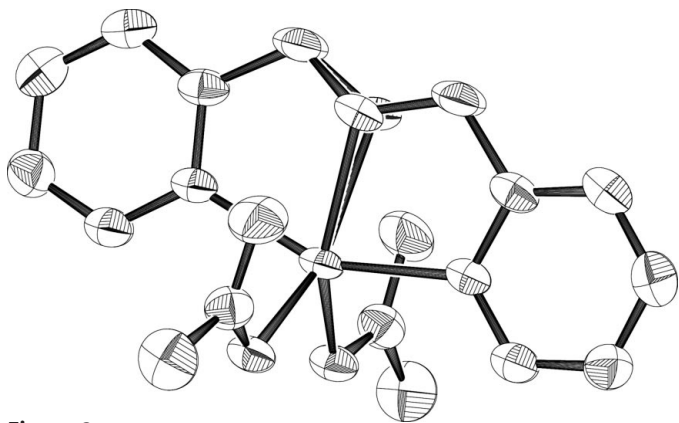


Figure 2
View of the symmetry-expanded structure of (I), with H atoms and acetic acid molecules omitted. Both disorder components of N2 are shown.

terminal zinc alkylcarboxylates (mean 2.038 Å; Orpen *et al.*, 1989).

The acetic acid solvent molecule donates a hydrogen bond to O2 [the non-coordinated O atom of the acetate ligand; the O3...O2 separation is 2.563 (3) Å]. The disordered secondary amine N atom donates a bifurcated hydrogen bond to O2 and to the acetic acid solvent molecule [the N2...O2 separation is 2.831 (5) Å, while the N2...O4 separation is 3.146 (5) Å].

Experimental

2-(Aminomethyl)pyridine (10.3 ml, 0.10 mol), Zn powder (20.069 g), and glacial acetic acid (20 ml) were added to 100 ml of 95% ethanol and heated to *ca.* 338 K under nitrogen. Pyridine-2-carboxaldehyde (9.5 ml, 0.10 mol) was added over a period of 2 hr, as were additional Zn (40.1206 g) and acetic acid (40 ml). The mixture was stirred for 5 hr, and then allowed to stand overnight at room temperature. The mixture was filtered. The filtrate was roto-evaporated to a syrup which was made alkaline with excess 5 M NaOH. The resultant organic layer was extracted twice with diethyl ether (50 ml each). The other extracts were combined, dried over MgSO₄ and roto-evapor-

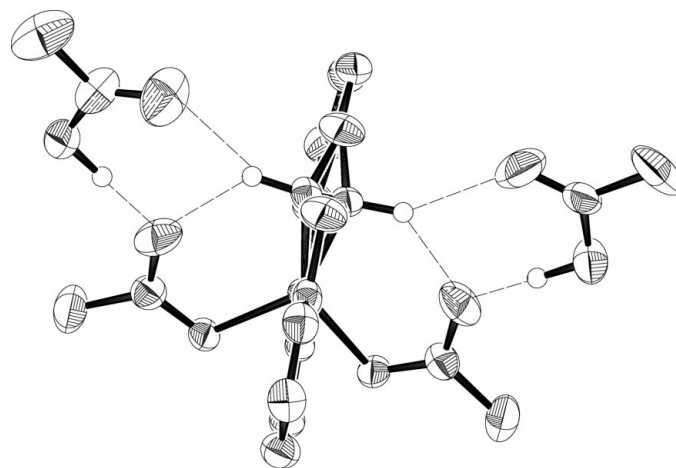


Figure 3
View of the symmetry-expanded structure of (I) emphasizing the hydrogen bonding (dashed lines). Only those H atoms participating in hydrogen bonding are shown.

ated to a syrup. This syrup produced large (several cm in length) crystals over *ca.* 15 days.

Crystal data

[Zn(C₂H₃O₂)₂(C₁₂H₁₃N₃)]·2C₂H₄O₂
M_r = 502.83
 Monoclinic, C2/c
a = 8.703 (2) Å
b = 13.791 (3) Å
c = 19.623 (4) Å
 β = 93.80 (2)°
V = 2350.0 (8) Å³
Z = 4

D_x = 1.421 Mg m⁻³
 Mo K α radiation
 Cell parameters from 3432 reflections
 θ = 2.5–30.5°
 μ = 1.09 mm⁻¹
T = 102 K
 Irregular fragment, colorless
 0.43 × 0.42 × 0.37 mm

Data collection

Nonius KappaCCD (with Oxford Cryosystems Cryostream cooler) diffractometer
 ω scans with κ offset
 Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.631, *T_{max}* = 0.667
 6634 measured reflections

3572 independent reflections
 2597 reflections with *I* > 3 σ (*I*)
R_{int} = 0.020
 θ_{max} = 30.5°
h = -12 → 12
k = -19 → 19
l = -27 → 27

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.073
wR(*F*²) = 0.150
S = 1.01
 2597 reflections
 150 parameters

H atoms: see below
 $w = 1/[\sigma^2(F_o) + 0.004225|F_o|^2]$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 1.36 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{min} = -0.44 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Zn1—O1	2.019 (2)	Zn1—N2	2.161 (4)
Zn1—N1	2.128 (2)		
O1—Zn1—O1 ⁱ	96.1 (1)	N1—Zn1—N2	77.9 (2)
O1—Zn1—N1	96.81 (8)	N1—Zn1—N2 ⁱ	77.5 (2)
O1—Zn1—N1 ⁱ	99.80 (8)	Zn1—O1—C7	110.1 (2)
O1—Zn1—N2	122.5 (1)	Zn1—N1—C1	125.0 (2)
O1—Zn1—N2 ⁱ	141.4 (1)	Zn1—N1—C5	116.0 (2)
N1—Zn1—N1 ⁱ	155.1 (1)	C1—N1—C5	118.9 (2)

Symmetry code: (i) $-x, y, \frac{3}{2} - z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2–H14 \cdots O2	0.99	2.16	2.831 (5)	123
N2–H14 \cdots O4	0.99	2.27	3.146 (5)	146
O3–H13 \cdots O2	1.03	1.54	2.563 (3)	176

Atom Zn1 is located on a twofold axis and N2 is disordered about, but not on, the twofold axis. Atoms H13 and H14, the H atoms associated with O3 and N2, respectively, were found in difference maps, but were not refined. All of the H atoms were assigned displacement parameters 1.2 times larger than the atoms to which they are bound. The methyl group of the acetate ligand is disordered. The first set of H atoms (H7, H8 and H9) were placed in calculated positions. The second set (H15, H16 and H17) were found in difference maps, but not refined. These six H atoms have half occupancies and were assigned displacement parameters 0.6 times $U_{eq}(C8)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Version 1.96.2; Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); molecular graphics: *TEXSAN for Windows*; software used to prepare material for publication: *TEXSAN for Windows*.

We thank Frank Fronczek for the data collection and the Louisiana Board of Regents Support Fund for financial support.

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