

Trichloro[μ -2,2,*N*-tris(2-pyridylmethyl-amino)acetamido]dizinc(II)Dong-Zhao Gao,* Hua-Kuan Lin
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.032
 wR factor = 0.076
Data-to-parameter ratio = 16.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $[\text{Zn}_2(\text{C}_{20}\text{H}_{20}\text{N}_5\text{O})\text{Cl}_3]$, was prepared from the multidentate ligand 2-[bis(pyridin-2-ylmethyl)amino]-*N*-(pyridin-2-ylmethyl)acetamide and zinc(II) chloride. The deprotonated ligand bridges the two Zn^{II} ions, coordinating to one in a tripodal fashion through the amide O and three N atoms, and to the second as a bidentate chelate through the amide and one pyridyl N atoms. Cl^- anions complete the distorted trigonal-bipyramidal coordination for one Zn^{II} ion and a distorted tetrahedral arrangement around the other.

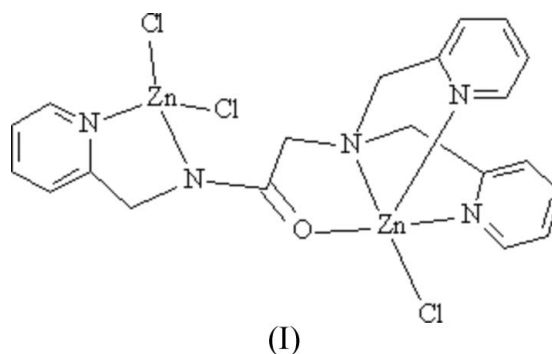
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Comment

Literature results show that a deprotonated amide N atom is a good donor for Fe and Cu ions and that amidate coordination can effectively stabilize metal ions in high oxidation states (Kostka *et al.*, 1993; Hamburg & Margerum, 1983). The strong donor ability of a deprotonated amide is also expected to imbue the metal centre to which it is coordinated with novel electronic properties. Recently, several designer ligands with pyridine-2-carboxamide units have been synthesized by different groups and their coordination properties with many transition metal ions have been explored (Rowland *et al.*, 2000; Zhu *et al.*, 2002).



In the title complex (I), the deprotonated 2-[bis(pyridin-2-ylmethyl)amino]-*N*-(pyridin-2-ylmethyl)acetamide ligand bridges two Zn^{II} ions, coordinating to one in a tripodal fashion through the amide O and three N atoms, and to the second as a bidentate chelate through the amidate and one pyridyl N atoms. Cl^- anions make up the distorted trigonal-bipyramidal coordination for one Zn^{II} ion and a distorted tetrahedral arrangement around the other (Fig. 1).

The trigonal-bipyramidal coordination of Zn1 comprises the tertiary atom N3 and a Cl^- anion in axial positions, and two pyridyl N atoms and the amide O atom in the equatorial

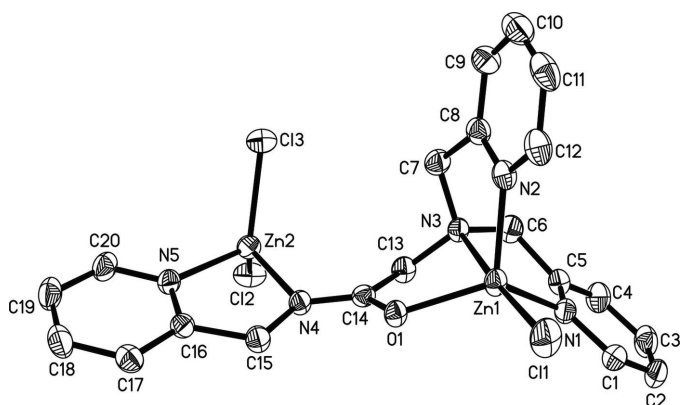


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

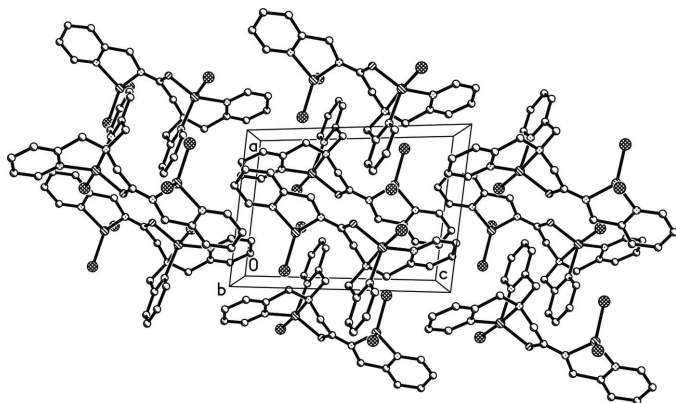


Figure 2
The molecular packing of compound (I), viewed along the *b* axis. H atoms have been omitted.

trigonal plane. The axial Zn1–N3 bond *trans* to the Cl1 ligand is significantly longer than the Zn1–N bonds in the equatorial plane (Table 1).

Atom Zn2 adopts a distorted tetrahedral geometry, chelated by the amidate atom N4 and the third pyridinyl N atom (N5) and binding two Cl[−] anions. At 2.051 (3) Å, the Zn2–N5 bond is significantly shorter than the other Zn–N bonds in the structure, reflecting the donor ability of the deprotonated N atom. The distance between the two Zn centres in the complex is 5.673 (7) Å. The crystal packing of the complex is shown in Fig. 2.

Experimental

The title compound was prepared by adding an aqueous solution (5 ml) of ZnCl₂ (0.2 mmol) to a methanol solution (10 ml) of 2-[bis(pyridin-2-ylmethyl)amino]-*N*-(pyridin-2-ylmethyl)acetamide (0.1 mmol), followed by several drops of triethylamine. The mixture was stirred for 1 h and then filtered. The filtrate was evaporated slowly at room temperature and colourless block-shaped crystals of (I) suitable for X-ray analysis were obtained. Analysis, calculated for C₂₀H₂₀Cl₃N₅OZn₂: C 41.16, H 3.45, N 12.00%; found: C 40.81, H 3.26, N 12.17%.

Crystal data

[Zn₂(C₂₀H₂₀N₅O)Cl₃]
M_r = 583.50
 Triclinic, *P* $\bar{1}$
a = 8.371 (3) Å
b = 12.462 (4) Å
c = 12.719 (4) Å
 α = 63.600 (4)°
 β = 81.880 (5)°
 γ = 85.524 (5)°
V = 1176.4 (6) Å³

Z = 2
D_x = 1.647 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 814 reflections
 θ = 2.9–26.2°
 μ = 2.40 mm^{−1}
T = 293 (2) K
 Block, colourless
 0.36 × 0.32 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.593, *T_{max}* = 0.786
 6780 measured reflections

4738 independent reflections
 3645 reflections with *I* > 2σ(*I*)
R_{int} = 0.021
 θ_{max} = 26.4°
h = −7 → 10
k = −15 → 13
l = −15 → 15

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.033
wR(*F*²) = 0.076
S = 1.00
 4738 reflections
 280 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 0.6148P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.44 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{min} = -0.39 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Zn1–O1	2.0256 (19)	Zn2–N5	2.051 (3)
Zn1–N1	2.100 (2)	Zn2–Cl2	2.2196 (11)
Zn1–N2	2.114 (3)	Zn2–Cl3	2.2575 (11)
Zn1–N3	2.273 (2)	N4–C14	1.307 (3)
Zn1–Cl1	2.2783 (9)	O1–C14	1.275 (3)
Zn2–N4	2.016 (2)		
O1–Zn1–N1	122.96 (9)	N3–Zn1–Cl1	176.01 (6)
O1–Zn1–N2	111.63 (9)	N4–Zn2–N5	82.86 (10)
N1–Zn1–N2	113.04 (9)	N4–Zn2–Cl2	117.91 (8)
O1–Zn1–N3	79.42 (8)	N5–Zn2–Cl2	113.84 (7)
N1–Zn1–N3	77.58 (9)	N4–Zn2–Cl3	111.70 (8)
N2–Zn1–N3	77.45 (9)	N5–Zn2–Cl3	109.61 (7)
O1–Zn1–Cl1	104.55 (6)	Cl2–Zn2–Cl3	116.19 (4)
N1–Zn1–Cl1	100.28 (7)	O1–C14–N4	125.7 (3)
N2–Zn1–Cl1	100.51 (8)	N4–C14–Cl3	116.7 (2)

H atoms were placed in calculated positions, with C–H = 0.93–0.96 Å, and were allowed to ride on their parent atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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