metal-organic papers

Zhenming Zhang,[a] Jian Gao,[b]* Daqi Wang[c] and Tongtao Xu[b]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [b]Department of Chemical Engineering, Lianyungang Technical College, Lianyungang 222006, People's Republic of China, and [c]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: gaojian553@163.com

Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.011 Å
Disorder in solvent or counterion
R factor = 0.054
wR factor = 0.098
Data-to-parameter ratio = 12.7

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

Bis{μ-1-[bis(3-aminopropyl)amino]propan-2-olato}-zinc(II) bis(perchlorate)

The title centrosymmetric dinuclear Zn^{II} complex, $[Zn_2(C_9H_{22}N_3O)_2](ClO_4)_2$, is centrosymmetric and comprises two Zn atoms bridged by O atoms from two 1-[bis(3-aminopropyl)amino]propan-2-olate ligands. Each Zn atom is five-coordinate with a distorted trigonal–bipyramidal coordination geometry; the Zn···Zn distance is 3.102 (2) Å.

Comment

Organic polyamines are of interest for their ability to coordinate to transition metal ions. The resulting polyamine complexes can exhibit catalytic, redox and antimicrobial activity, for example mimicking superoxide dismutases (Gao *et al.*, 2005). Complexes of tris(3-aminopropyl)amine have been studied extensively. The title compound, (I), is the first complex of 1-[bis(3-aminopropyl)amino]propan-2-ol to be reported.

(I)
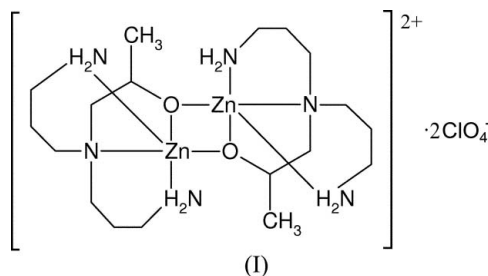
The dinuclear cation is centrosymmetric. Atom Zn1 is located in a five-coordinate environment with a distorted trigonal bipyramidal geometry (Fig. 1 and Table 1). The equatorial plane comprises O1, N2 and N3 from the same polyamine ligand, and the axial sites comprise the tertiary nitrogen atom N1 and O1^i from the other polyamine ligand [symmetry code: (i) $2 - x$, $1 - y$, $-z$]. The value of the τ parameter (0.66) indicates that the coordination polyhedron lies 66% along the pathway of distortion from square-pyramidal to trigonal–bipyramidal geometry (Addison *et al.* 1984). Atoms N2 and N3 form intermolecular hydrogen bonds with O atoms of the perchlorate anions (Table 2).

Experimental

A solution of 1-[bis(3-aminopropyl)amino]propan-2-ol (0.5 mmol) in 10 ml absolute methanol was added dropwise to a stirred solution of $Zn(ClO_4)_2 \cdot 6H_2O$ (0.5 mmol) in 15 ml absolute methanol at room temperature. After stirring for 1 h at 320 K, the precipitate was filtered off, washed with methanol and dried *in vacuo*. Colourless single crystals suitable for X-ray analysis were obtained after 10 d by slow evaporation of the filtrate at ambient temperature.

Crystal data

[Zn$_2$(C$_9$H$_{22}$N$_3$O)$_2$](ClO$_4$)$_2$
$M_r = 706.23$
Monoclinic, $P2_1/n$
$a = 7.961$ (5) Å
$b = 15.255$ (10) Å
$c = 12.128$ (8) Å
$\beta = 98.228$ (9)°
$V = 1457.7$ (16) Å^3

$Z = 2$
$D_x = 1.609$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.89$ mm^{-1}
$T = 298$ (2) K
Prism, colourless
$0.24 \times 0.11 \times 0.09$ mm

Data collection

Bruker SMART CCD
  diffractometer
φ and ω scans
Absorption correction: multi-scan
  (*SADABS*; Sheldrick, 2002)
  $T_{min} = 0.660$, $T_{max} = 0.849$

7190 measured reflections
2543 independent reflections
1294 reflections with $I > 2\sigma(I)$
$R_{int} = 0.156$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.098$
$S = 0.95$
2543 reflections
200 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	1.963 (4)	Zn1—N3	2.050 (5)
Zn1—N1	2.231 (5)	Zn1—O1^i	2.087 (4)
Zn1—N2	2.043 (5)	Zn1—Zn1^i	3.102 (2)
O1—Zn1—N1	82.70 (18)	N2—Zn1—N3	117.2 (3)
O1—Zn1—N2	119.7 (2)	O1—Zn1—O1^i	80.07 (17)
O1—Zn1—N3	122.9 (2)	N2—Zn1—O1^i	91.7 (2)
N1—Zn1—N2	94.5 (2)	N3—Zn1—O1^i	95.20 (18)
N1—Zn1—N3	96.4 (2)	O1^i—Zn1—N1	162.57 (17)

Symmetry code: (i) $-x + 2, -y + 1, -z$.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$O3$'^{ii}$	0.90	2.22	3.087 (13)	160
N3—H3$A\cdots$O2^{iii}	0.90	2.21	3.078 (8)	163
N3—H3$A\cdots$O3^{iii}	0.90	2.59	3.30 (3)	137
N3—H3$B\cdots$O4^{iv}	0.90	2.21	3.083 (19)	164
N3—H3$B\cdots$O4$'^{iv}$	0.90	2.27	3.142 (11)	164

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

Methylene H atoms and amino H atoms were positioned geometrically, with C—H = 0.97 or N—H = 0.90 Å, and allowed to ride
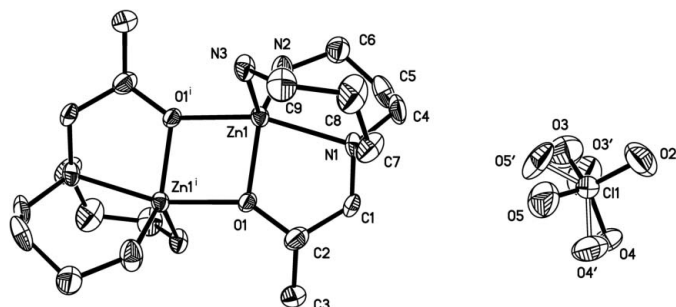

Figure 1
Molecular structure of (I) showing displacement ellipsoids at the 30% probability level. H atoms have been omitted. The two components of the disordered perchlorate anion are shown with solid bonds and open bonds, respectively. [Symmetry code: (i) $2 - x, 1 - y, -z$.]

during subsequent refinement with U_{iso}(H) = 1.2U_{eq}(C,N). Methyl H atoms were placed in calculated positions (C—H = 0.96 Å) and were allowed to ride with U_{iso}(H) = 1.5U_{eq}(C). The methyl group was also allowed to rotate about its local threefold axis. The O atoms of the perchlorate anion were found to be disordered. Atom O2 is common to both anion components, while the site occupancy factors of atoms O3/O4/O5 and O3′/O4′/O5′ were refined to 0.292 (18) and 0.708 (18), respectively. All Cl—O and O$\cdots$O distances were restrained to 1.40 (2) and 2.32 (2) Å, respectively, and the displacement parameters of the O atoms were restrained to approximate isotropic behaviour.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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