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mer-Bis(diethylenetriamine)zinc(II) dichloride monohydrate

Bing Liu, Fang-Jun Huo, Xiao-Ming Jiang and Bing-Sheng Yang*

Institute of Molecular Science, Chemical Biology and Molecular Engineering Laboratory of the Education Ministry, Shanxi University, Taiyuan, Shanxi 030006, People's Republic of China

Correspondence e-mail: yangbs@sxu.edu.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.041
wR factor = 0.081
Data-to-parameter ratio = 17.6

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

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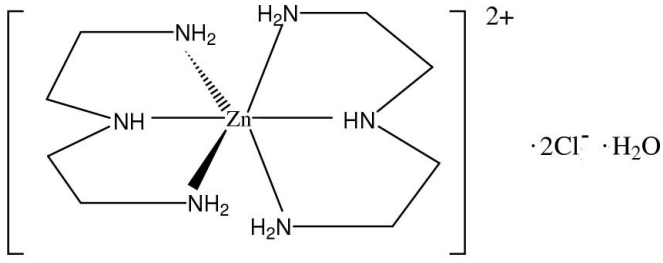
In the title compound, $[Zn(C_4H_{13}N_3)_2]Cl_2\cdot H_2O$, the Zn^{II} atom is coordinated by six N atoms from two diethylenetriamine ligands in a distorted octahedral geometry. The complex cation is the *mer* isomer.

Comment

Zinc is a trace element that is essential to life. It functions as a catalyst in over 300 enzymes, including those responsible for DNA replication, RNA and protein synthesis, and gene transcription (Vallee *et al.*, 1993). Metal ions may bind to tissue and interstitial fluid proteins as soon as they are released from their complexes. One of the major reasons for the biotoxicity of certain metal ions is their ability to bring about protein damage (Michalak *et al.*, 1992). We designed and synthesized the title complex, (I), as a new zinc complex.



(I)

The molecular structure of (I) is illustrated in Fig. 1. There are three possible geometrical isomers for an octahedral $[M(\text{dien})_2]^{n+}$ complex, *viz.* sym-*fac*, unsym-*fac* and *mer* isomers (dien is diethylenetriamine). The complex cation in (I) is the *mer*-isomer. Some features of the molecular geometry are listed in Table 1.

The hydrogen-bonding geometry is listed in Table 2 and illustrated in Fig. 2. A number of intermolecular hydrogen bonds stabilize the crystal structure.

Experimental

Compound (I) was prepared by the following method, although the authors' intention was to synthesize a Cr^{II} complex and zinc was added as a reducing reagent. Diethylenetriamine (6 ml, 0.055 mol) was slowly added to a mixture of $CrCl_3\cdot 6H_2O$ (0.01 mol, 2.665 g) and zinc (0.65 g, 0.01 mol) in methanol (20 ml) with refluxing for 2 h. During this time, the solution turned from green to purple–red. The resulting solution was put aside for 2 d, after which colourless block crystals of (I) appeared (yield 48%).

Crystal data

$[Zn(C_4H_{13}N_3)_2]Cl_2·H_2O$
M_r = 360.63
Monoclinic, $P2_1/c$
a = 13.436 (4) Å
b = 8.811 (2) Å
c = 13.924 (4) Å
β = 102.030 (4)°
V = 1612.2 (8) Å^3
Z = 4
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1897 reflections
θ = 2.8–22.9°
μ = 1.86 mm^{-1}
T = 298 (2) K
Block, colourless
0.20 × 0.20 × 0.10 mm

Data collection

SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.708, T_{max} = 0.836
7671 measured reflections
2862 independent reflections
2166 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.1°
$h = -15 \rightarrow 16$
$k = -9 \rightarrow 10$
$l = -8 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.081
S = 0.95
2862 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.162 (3)	Zn1—N4	2.212 (3)
Zn1—N2	2.144 (3)	Zn1—N5	2.137 (3)
Zn1—N3	2.230 (3)	Zn1—N6	2.233 (3)
N1—Zn1—N2	80.36 (10)	N2—Zn1—N6	102.12 (10)
N1—Zn1—N3	158.71 (11)	N3—Zn1—N4	90.22 (11)
N1—Zn1—N4	98.55 (12)	N3—Zn1—N5	101.44 (10)
N1—Zn1—N5	99.21 (10)	N3—Zn1—N6	87.22 (10)
N1—Zn1—N6	91.43 (11)	N4—Zn1—N5	79.55 (11)
N2—Zn1—N3	79.17 (10)	N4—Zn1—N6	158.25 (11)
N2—Zn1—N4	98.59 (11)	N5—Zn1—N6	79.80 (10)
N2—Zn1—N5	178.02 (11)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.90	2.34	3.166 (4)	152
N1—H1B···Cl2^{ii}	0.90	2.55	3.388 (3)	155
N2—H2···Cl1	0.91	2.40	3.311 (3)	174
N3—H3A···Cl1^{iii}	0.90	2.45	3.339 (3)	171
N3—H3B···Cl1^{iv}	0.90	2.56	3.462 (3)	178
N5—H5···Cl2^{v}	0.91	2.45	3.348 (3)	169
N6—H6A···Cl1^{iii}	0.90	2.55	3.391 (3)	155
N6—H6B···Cl2^{ii}	0.90	2.57	3.404 (3)	154
O1—H30···Cl2^{ii}	0.84	2.35	3.184 (3)	172
O1—H31···Cl2^{vi}	0.84	2.39	3.196 (3)	161

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

H atoms attached to O atoms (water) were located in a difference Fourier map and their geometry idealized, with O—H = 0.84 Å and U_{iso}(H) = 1.2U_{eq}(O); they were then fixed in position. The other H atoms were treated as riding atoms, with C—H (CH_2) = 0.97 Å, N—H (NH) = 0.91 Å and N—H (NH_2) = 0.90 Å, and with U_{iso}(H) = 1.5U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve

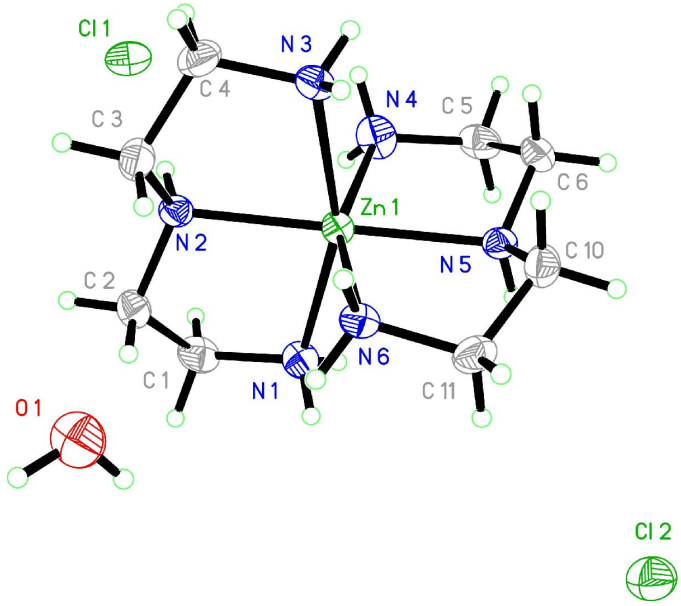


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

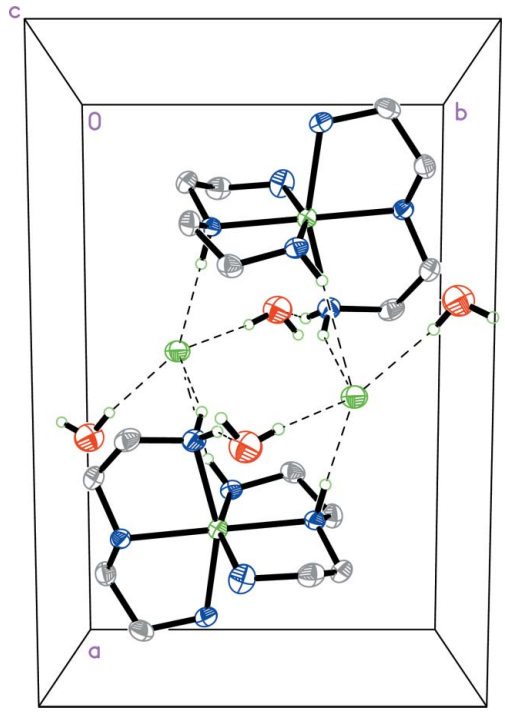


Figure 2
The crystal structure of (I), projected along the c axis. H atoms have been omitted except for those involved in hydrogen bonding. Dashed lines show hydrogen bonds.

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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