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Tris(propane-1,2-diamine-κ^2N,N')zinc(II) dichloride dihydrate

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In the title compound, $[Zn(C_3H_{10}N_2)_3]Cl_2\cdot 2H_2O$, a complex three-dimensional network of N—H···O, N—H···Cl and O—H···Cl hydrogen bonds helps to establish the crystal packing.

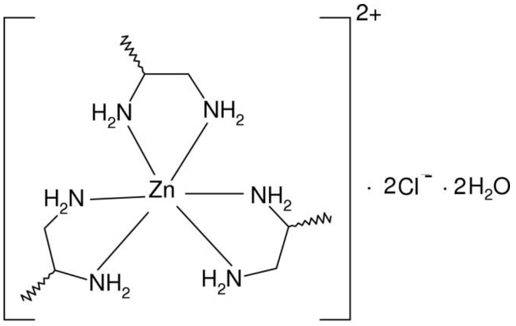
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.042
wR factor = 0.102
Data-to-parameter ratio = 21.5

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

Comment

The title compound, (I), containing $[Zn(C_3H_{10}N_2)_3]^{2+}$ dications accompanied by charge-balancing chloride ions and water molecules of crystallization (Fig. 1), complements many crystal structures containing tris(1,2-diaminoethane)zinc(II) [or $Zn(C_2H_8N_2)_3{}^{2+}$] dications (e.g. Muralikrishna *et al.*, 1983; Nesterova *et al.*, 2006). However, (I) represents the first crystal structure of a tris-chelate of zinc with 1,2-diaminopropane.



(I)

In (I), the Zn cation is bonded to three *N*,*N*-bidentate amine molecules, resulting in a somewhat distorted ZnN_6 octahedron (Table 1). The mean Zn—N distance of 2.200 (8) Å in (I) compares with a mean Zn—N of 2.215 Å in $[Zn(C_2H_8N_2)_3]Cl_2\cdot 2(H_2O)$ (Muralikrishna *et al.*, 1983). The

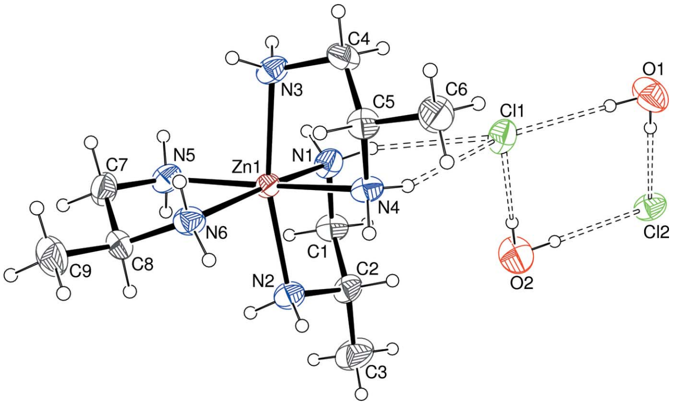


Figure 1
The molecular structure of (I) showing 30% displacement ellipsoids (H atoms are drawn as spheres of arbitrary radius). Hydrogen bonds are indicated by double dashed lines.

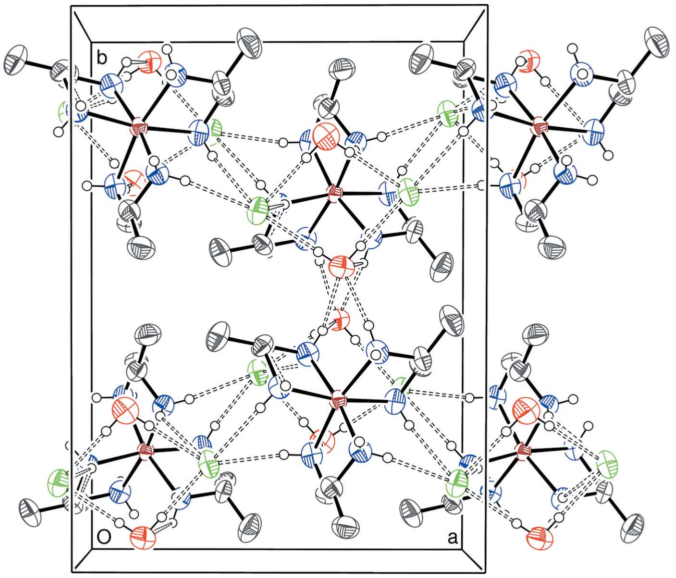


Figure 2
The packing of (I), with the C-bound H atoms omitted for clarity and hydrogen bonds indicated by double dashed lines.

three chelating N—Zn—N bite angles in (I) are almost identical (Table 1). Three five-membered chelate rings occur in the dication in (I), each of which approximates to an envelope: the r.m.s. deviation, π, for the mean plane defined by Zn1—N1—N2—C1 is 0.090 Å, and the deviation from the plane, δ, for C2 is 0.617 (7) Å. For the other two chelate rings, equivalent values are: π(Zn1—N3—N4—C4) = 0.048 Å and δ = 0.632 (7) Å for C5 and π(Zn1—N5—N6—C7) = 0.056 Å and δ = 0.596 (7) Å for C8.

The complex in (I) is chiral; in the arbitrarily chosen asymmetric unit, atoms C2, C5 and C8 all have an *S* configuration, but the *c*-glide generates a racemic mixture of enantiomers in the crystal structure, consistent with the racemic starting amine. Considering only the backbone of the amine (*i.e.* C3, C6, C9 and all H atoms are neglected), then the Λ form of the tris-chelate complex (Piper, 1961) is apparent in the asymmetric molecule shown in Fig. 1. Again, symmetry generates an equal number of Λ and Δ species in the crystal structure.

A large number of N—H···Cl and N—H···O hydrogen bonds (Table 2) help to establish the crystal packing in (I). The water molecules form O—H···Cl bonds, resulting in distinctive closed loops of two water molecules and two chloride ions, with an $R_4^2(8)$ graph-set motif (Bernstein *et al.*, 1995). Together, the hydrogen bonds form a three-dimensional network (Fig. 2,) with Cl1 and Cl2 accepting six hydrogen bonds each.

Experimental

10 ml of 0.1 *M* $ZnCl_2$, 10 ml of 0.1 *M* 1,2-diaminopropane and a few drops of dilute HCl solution were mixed at 293 K in a Petri dish, resulting in a colourless mixture. Colourless blocks of (I) grew over the course of a few days at 293 K as the water evaporated.

Crystal data

$[Zn(C_3H_{10}N_2)_3]Cl_2 \cdot 2H_2O$
M_r = 394.69
Monoclinic, *Cc*
a = 12.6384 (9) Å
b = 17.2427 (12) Å
c = 8.8721 (6) Å
β = 90.777 (1)°
V = 1933.2 (2) Å^3
Z = 4
Mo *K*α radiation
μ = 1.56 mm^{-1}
T = 293 (2) K
0.37 × 0.18 × 0.17 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.597, T_{max} = 0.778
8264 measured reflections
3984 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.102
S = 0.97
3984 reflections
185 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 1152 Friedel pairs
Flack parameter: 0.190 (17)

Table 1
Selected geometric parameters (Å, °).

Zn1—N5	2.177 (4)	Zn1—N2	2.207 (4)
Zn1—N3	2.193 (4)	Zn1—N1	2.207 (5)
Zn1—N4	2.206 (4)	Zn1—N6	2.211 (4)
N3—Zn1—N4	79.63 (16)	N5—Zn1—N6	79.36 (17)
N2—Zn1—N1	79.79 (17)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2^{i}	0.90	2.62	3.457 (5)	155
N1—H2···Cl1	0.90	2.54	3.419 (5)	166
N2—H3···Cl2^{ii}	0.90	2.83	3.601 (5)	144
N2—H4···O1^{iii}	0.90	2.30	3.185 (6)	166
N3—H11···O2^{i}	0.90	2.27	3.133 (6)	162
N3—H12···Cl2^{iv}	0.90	2.56	3.414 (5)	160
N4—H14···Cl1	0.90	2.73	3.599 (5)	162
N5—H21···Cl1^{i}	0.90	2.71	3.477 (5)	144
N5—H22···O1^{iii}	0.90	2.34	3.143 (6)	148
N6—H23···Cl1^{ii}	0.90	2.69	3.499 (5)	150
N6—H24···Cl2^{iv}	0.90	2.68	3.553 (5)	165
O1—H31···Cl1	0.90	2.32	3.214 (5)	179
O1—H32···Cl2	0.90	2.27	3.166 (5)	180
O2—H33···Cl1	0.90	2.26	3.164 (5)	180
O2—H34···Cl2	0.90	2.29	3.186 (5)	180

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

The crystal studied was a partial inversion twin with a 0.810 (17):0.190 (17) ratio of domains. The O-bound H atoms were positioned geometrically (O—H = 0.90 Å) to establish a reasonable hydrogen-bond network, and were refined as riding with U_{iso}(H) = 1.2U_{eq}(O). The C- and N-bound H atoms were placed in idealized locations (C—H = 0.96–0.98 Å and N—H = 0.90 Å) and were refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip, to best fit the electron density.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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