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Bis[*N*,*N*′-bis(3,4-methylenedioxybenzyl)-propane-1,3-diamine]dichlorozinc(II)

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The title complex, $[ZnCl_2(C_{19}H_{22}N_2O_4)]$, is a mononuclear compound; the central zinc ion is coordinated by two Cl^- ligands and two N atoms of the bis(3,4-methylenedioxybenzyl)propane-1,3-diamine in a distorted tetrahedral geometry. The complex adopts a 'hawk' conformation. The molecules are linked by pairs of N—H···Cl hydrogen bonds into a $C_2^2(8)C_2^2(8)[R_2^2(8)R_2^2(8)]$ chain of rings along the [001] direction and the molecules are linked by a pair of C—H···O and also by a pair of C—H···Cl hydrogen bonds into a chain of alternating $R_2^2(6)$ rings and $R_2^2(20)$ rings along the [111] direction. The combination of the [111] chain and the [001] chain generates $[1\bar{1}0]$ stacks.

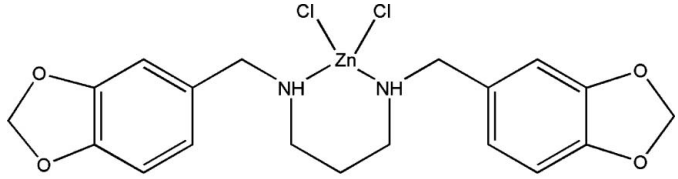
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.030
wR factor = 0.080
Data-to-parameter ratio = 14.4

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

Comment

Zn^{II} complexes have a broad range of biological activities, including inhibition of carbonic anhydrase (CA) (Scozzafava *et al.*, 2001; Puccetti *et al.*, 2005), antibacterial (Zhang *et al.*, 2003) and anti-HIV-1 (Masami Otsuka *et al.*,1997; Wang *et al.*, 2004); the crystal structures of various Zn^{II} complexes have been described (Feinberg *et al.*, 1995; You, 2005; Johansson & Håkansson, 2004). We report here the crystal structure of the title Zn^{II} complex, (I).



(I)

Complex (I) is a mononuclear compound (Fig. 1). The central zinc ion is coordinated by two Cl^- and two N atoms of *N*,*N*′-(3,4-methylenedioxybenzyl)propane-1,3-diamine in a distorted tetrahedral geometry (Table 1). The dihedral angle

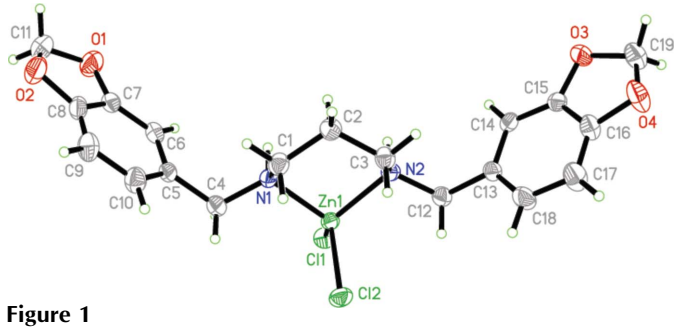


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

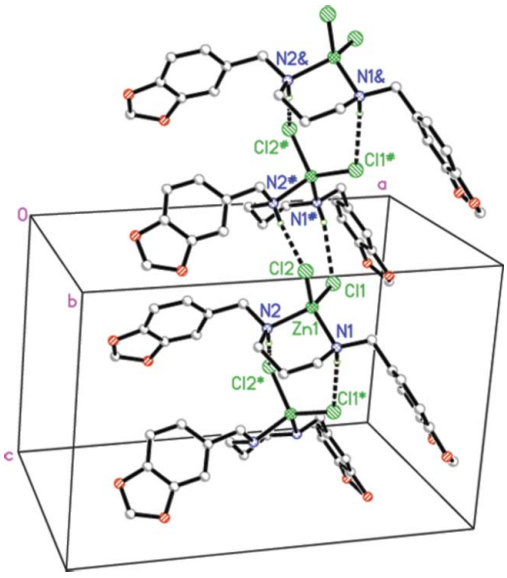


Figure 2
The molecular structure of (I), showing the formation of a [001] chain of rings. For clarity, H atoms not involved in the motif shown have been omitted [symmetry codes: (*) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (#) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (&) $x, y, -1+z$]. Dashed lines indicate hydrogen bonds.

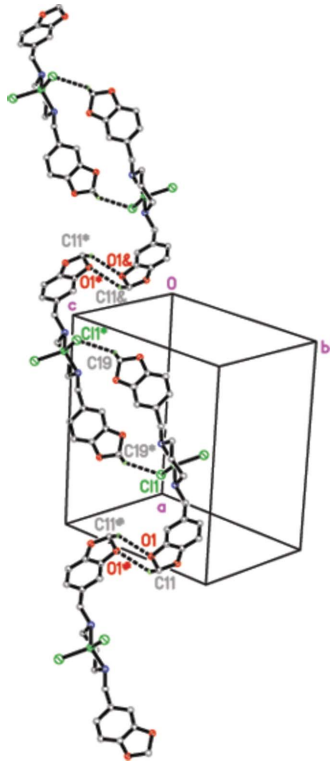


Figure 3
Part of the crystal structure of (I), showing the formation of the the [111] chain of rings. For clarity, H atoms not involved in the motif shown have been omitted [symmetry codes: (*) $1-x, -y, 1-z$; (#) $2-x, 1-y, 2-z$; (&) $-1+x, -1+y, -1+z$]. Dashed lines indicate hydrogen bonds.

between the N1/Zn1/N2 and Cl1/Zn1/Cl2 planes is 87.40 (6)°. The six-membered ring Zn/N1/N2/C1–C3 has a chair conformation, in which the sum of the internal angles is 656 (1)° and the dihedral angles between the N1/Zn1/N2 and N1/N2/C3/C1 planes, and the C1/C2/C3 and N1/N2/C3/C1 planes, are,

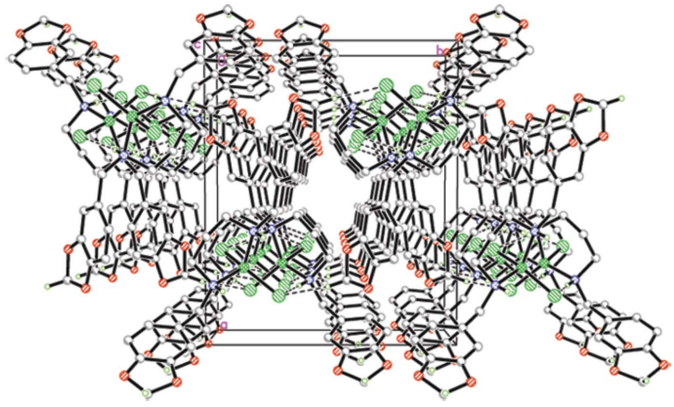


Figure 4
The packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds. For clarity, H atoms not involved in the motif shown have been omitted.

respectively, 46.1 (1) and 63.1 (3)°. Complex (I) adopts a 'hawk' conformation, and the two 3,4-methylenedioxybenzyl rings are located on opposite sides of the six-membered ring; the two benzene rings enclose a dihedral angle of 76.2 (1)°.

In the crystal structure of (I), the molecules are linked bya pairs of N—H···Cl hydrogen bonds into a $C_2^2(8)C_2^2(8)[R_2^2(8)R_2^2(8)]$ chain of rings (Bernstein *et al.*, 1995) along the [001] direction. Atoms N1 and N2 in the molecule at (x, y, z) both act as hydrogen-bond donors to atoms Cl1 and Cl2 in the molecule at $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ (Fig. 2). The molecules are linked by a pair of C—H···O and a pair of C—H···Cl hydrogen bonds into a chain of alternating $R_2^2(6)$ and $R_2^2(20)$ rings (García-Báez *et al.*, 2002) along the [111] direction. Atoms C11 and C19 in the molecule at (x, y, z) both act as hydrogen-bond donors to atom O1 in the molecule at $(2-x, 1-y, 2-z)$ and atom Cl1 in the molecule at $(1-x, -y, 1-z)$ (Fig. 3). The combination of the [111] chain and the [001] chain generates a $[1\bar{1}0]$ stack. Neighbouring stacks are connected by van der Waals forces, resulting in a three-dimensional network structure (Fig. 4).

Experimental

To a solution containing *N*,*N*′-di(3,4-methylenedioxybenzyl)propane-1,3-diamine (3.42 g, 10 mmol) and ethanol–chloroform (1:1, 30 ml), a solution of zinc chloride (1.36 g, 10 mmol) and ethanol (10 ml) was added with stirring for 2 h at room temperature (298–300 K); the solid obtained was filtered off, washed successively with chloroform and ethanol, and dried at room temperature. Colourless crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a DMF solution over a period of one week (m.p. 493–495 K).

Crystal data

$[ZnCl_2(C_{19}H_{22}N_2O_4)]$
M_r = 478.66
Monoclinic, $P2_1/c$
a = 15.612 (4) Å
b = 12.934 (3) Å
c = 10.318 (3) Å
β = 94.557 (3)°
V = 2076.9 (9) Å^3
Z = 4
D_x = 1.531 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.47 mm^{-1}
T = 298 (2) K
Block, colourless
0.42 × 0.22 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	10572 measured reflections
φ and ω scans	3650 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2705 reflections with $I > 2\sigma(I)$
$T_{min} = 0.578$, $T_{max} = 0.810$	$R_{int} = 0.029$
	$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 0.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.080$	$\Delta\rho_{max} = 0.35$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
3650 reflections	
253 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	2.045 (2)	Zn1—Cl1	2.2007 (9)
Zn1—N2	2.065 (2)	Zn1—Cl2	2.2416 (9)
N1—Zn1—N2	97.17 (9)	C1—N1—Zn1	108.61 (17)
N1—Zn1—Cl1	115.73 (7)	C3—N2—Zn1	108.44 (17)
N2—Zn1—Cl1	118.10 (7)	N1—C1—C2	111.9 (2)
N1—Zn1—Cl2	108.80 (7)	C3—C2—C1	117.0 (3)
N2—Zn1—Cl2	104.66 (7)	N2—C3—C2	112.4 (2)
Cl1—Zn1—Cl2	111.05 (4)		
Cl2—Zn1—N1—C1	58.28 (19)	Cl1—Zn1—N2—C12	−61.55 (19)
Cl1—Zn1—N1—C4	58.7 (2)	Cl2—Zn1—N2—C12	62.55 (19)
Cl2—Zn1—N1—C4	−67.1 (2)	C4—N1—C1—C2	−173.4 (2)
Cl2—Zn1—N2—C3	−62.45 (18)	C12—N2—C3—C2	177.3 (2)
N1—Zn1—N2—C12	174.18 (19)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{i}	0.91	2.63	3.526 (3)	167
N2—H2···Cl2^{i}	0.91	2.51	3.386 (3)	163
C11—H11A···O1^{ii}	0.97	2.72	3.404 (5)	128
C19—H19A···Cl1^{iii}	0.97	2.76	3.461 (4)	130

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with N—H = 0.91 Å, C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}$ (C,N).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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