[N,N,N′,N′-Tetrakis(2-benzimidazolyl-methyl)-1,2-ethanediamine]zinc(II) dinitrate ethanol solvate

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In the title compound, [Zn(EDTB)](NO₃)₂·C₂H₅OH, where EDTB = N,N,N′,N′-tetrakis(2-benzimidazolylmethyl)-1,2-ethanediamine (C₃₄H₃₂N₁₀), the coordination geometry around each zinc ion can be described as distorted octahedral, with two equatorial benzimidazole N atoms, two equatorial amine atoms, and two axial benzimidazole N atoms. The nitrate anions do not coordinate to the zinc(II) cations, but act as counter-anions, and are hydrogen bonded to EDTB ligands.

Comment

N,N,N′,N′-Tetrakis(2-benzimidazolylmethyl)-1,2-ethanediamine (EDTB) was first prepared by Hendriks et al. (1979) in order to investigate the relationships between the activity and structures of metalloproteins, such as hemocyanin, laccase, tyrosinase and superoxide dismutase. Nowadays, many complexes with EDTB and its derivatives have been synthesized and characterized, but only a few crystal structures of these complexes have been reported. [Cu(EDTB)(BF₄)(B-F₃OC₂H₅)]·H₂O exhibits a mononuclear structure, in which the Cu^{II} ion is coordinated by the six N atoms from one molecule of EDTB (Birker et al., 1981). The Cu^{I} complex, Cu₂(EDTB)(ClO₄)₂, however, has a dinuclear structure, in which each Cu^{I} ion is coordinated by two benzimidazole N atoms from one molecule of EDTB, and each EDTB is coordinated to two Cu^{I} ions (Hendriks et al., 1982). In an Fe^{III} complex, EDTB shows an interesting coordination mode, with two amine N atoms and three benzimidazole N atoms coordinated to the same Fe^{III} ion, while the remaining benzimidazole group is non-coordinating (Gomez-Romero et al., 1990). The structure of a nickel complex with EDTB consists of the cationic species [Ni(EDTB)]$^{2+}$, in which the Ni^{II} ion is six-coordinated by four benzimidazole N atoms and two amine N atoms (Holt et al., 1987). In this paper, the crystal structure of a zinc complex, (I), with EDTB is presented.

(I)
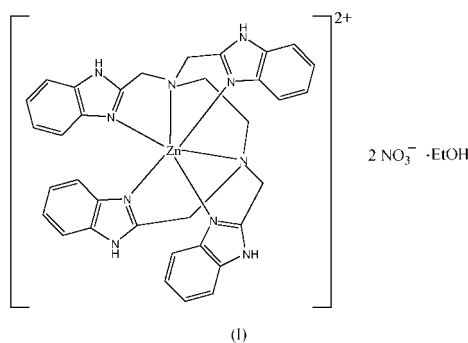
The structure of (I) is shown in Fig. 1 and contains the monomeric cationic species [Zn(EDTB)]$^{2+}$ in which the zinc ion is coordinated by four benzimidazole N atoms and two
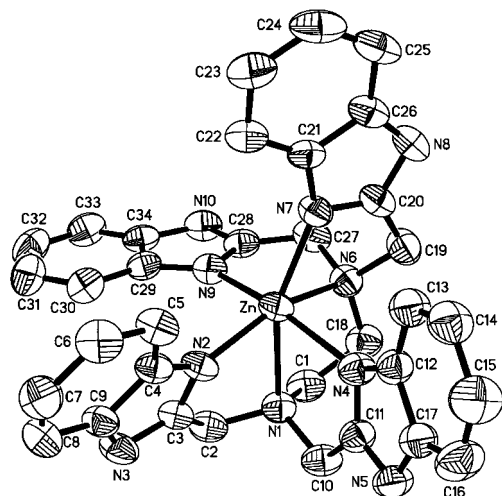
Figure 1
View of the local coordination of Zn(II), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
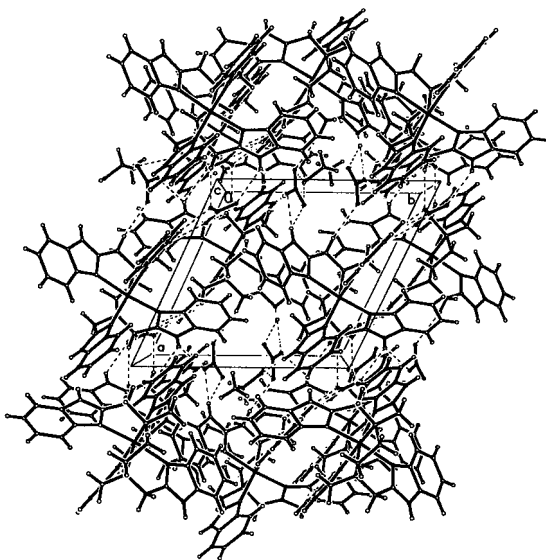

Figure 2
Packing diagram of (I).

amine N atoms, forming five five-membered chelate rings. The coordination geometry around the zinc ion can be described as distorted octahedral. The two zinc amine nitrogen distances, Zn—N1 [2.344 (3) Å] and Zn—N6 [2.240 (3) Å], are very similar to the values found for other Zn^{II} complexes with amines (Bu *et al.*, 1996). The two distances Zn—N2 [2.033 (3) Å] and Zn—N7 [2.064 (3) Å] are very similar to the values found for other related Zn^{II} complexes (Szlyk *et al.*, 1997), while the other two Zn benzimidazole nitrogen distances, Zn—N4 [2.178 (3) Å] and Zn—N9 [2.163 (3) Å], are somewhat longer and presumably a consequence of the geometrical requirements of the ligand. The bond angles related to the coordination sphere of the Zn^{II} ion range from 76.88 (10) to 126.89 (11)° for *cis* atoms and from 154.65 (10) to 163.56 (9)° for *trans* atoms. The largest deviation from ideal geometry is the *cis* N2—Zn—N7 angle of 126.89 (11)°. The benzene and imidazole rings are planar, the maximum

deviation from the least-squares planes being less than 0.02 Å. Two non-coordinated nitrate ions are hydrogen bonded to benzimidazole N atoms and there is also a hydrogen bond between the ethanol molecule and the benzimidazole N atom. The hydrogen-bonding geometry details are listed in Table 1.

Experimental

Crystal data

[Zn(C34H32N10)](NO3)2·C2H6O	$Z = 2$
$M_r = 816.15$	$D_x = 1.474$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 11.783$ (3) Å	Cell parameters from 33
$b = 12.219$ (4) Å	reflections
$c = 14.468$ (6) Å	$\theta = 5.3$–$10.4°$
$\alpha = 91.42$ (3)°	$\mu = 0.74$ mm^{-1}
$\beta = 103.51$ (3)°	$T = 293$ (2) K
$\gamma = 113.653$ (18)°	Block, colorless
$V = 1838.9$ (11) Å^3	$0.50 \times 0.36 \times 0.36$ mm

Data collection

Siemens $P4$ diffractometer	$R_{int} = 0.022$
ω scans	$\theta_{max} = 25.0°$
Absorption correction: ψ scan	$h = -1 \rightarrow 13$
(North *et al.*, 1968)	$k = -14 \rightarrow 13$
$T_{min} = 0.735$, $T_{max} = 0.768$	$l = -17 \rightarrow 16$
7751 measured reflections	3 standard reflections
6479 independent reflections	every 97 reflections
3961 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	H atoms treated by a mixture of
$R[F^2 > 2\sigma(F^2)] = 0.042$	independent and constrained
$wR(F^2) = 0.089$	refinement
$S = 0.85$	$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2]$
6479 reflections	where $P = (F_o^2 + 2F_c^2)/3$
509 parameters	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.26$ e Å^{-3}
	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$\cdots$O1^i	0.86	2.00	2.787 (4)	152
N3—H3$\cdots$O2^i	0.86	2.35	3.037 (4)	137
N5—H5$\cdots$O5	0.86	2.05	2.875 (4)	160
N8—H8$\cdots$O7^{ii}	0.86	1.91	2.762 (4)	169
N10—H10$\cdots$O5^{iii}	0.86	2.28	2.988 (4)	140
O7—H7A$\cdots$O4^{iv}	0.849 (19)	2.26 (3)	3.039 (5)	152 (5)
O7—H7A$\cdots$O6^{iv}	0.849 (19)	2.28 (3)	3.049 (5)	151 (5)

Symmetry codes: (i) $x, 1+y, z$; (ii) $1+x, y, z$; (iii) $1+x, 1+y, z$; (iv) $-x, 1-y, 1-z$.

All H atoms on C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The H atom of the hydroxyl group was located from a difference Fourier map and refined with $U_{iso}(H) = 1.5U_{eq}(O)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL*97.

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