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N,*N*,*N*′,*N*′-Tetrakis[(1*H*-benzimidazol-2-yl)methyl]-ethane-1,2-diamine glycol disolvate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.069
wR factor = 0.250
Data-to-parameter ratio = 12.6

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

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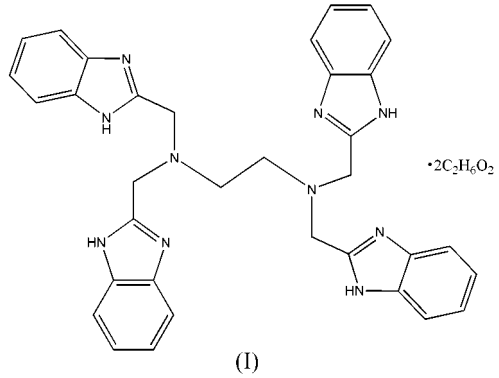
The title compound, $C_{34}H_{32}N_{10}\cdot 2C_2H_6O_2$, crystallizes in space group $P\overline{1}$, with the main molecule on an inversion centre. The crystal packing is stabilized by intermolecular N—H···O and O—H···N hydrogen bonds.

Comment

Multinuclear metal complexes containing several benzimidazole ligands have attracted much attention as superoxide dismutase (SOD) enzyme and nuclease mimics (Liao *et al.*, 2001; Liu *et al.*, 2004). Many metal complexes containing benzimidazole have been synthesized and characterized (Li *et al.*, 2003; Yang *et al.*, 2003; Yan *et al.*, 2004). The SOD-like activities of *N*,*N*,*N*′,*N*′-tetrakis-[(2-benzimidazolyl)methyl]-1,2-ethanediamine (EDTB) metal complexes (Liao *et al.*, 2001) attracted our interest. In the process of synthesizing EDTB–metal complexes, we obtained the title compound, (I), and present its crystal structure here.



(I)

The main geometric parameters of (I) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. The EDTB molecule has a crystallographically imposed centre of symmetry. The four benzimidazolyl groups in EDTB are individually planar. The dihedral angle between the two independent benzimidazolyl least-squares planes is 44.5 (1)°.

The crystal packing of (I) (Fig. 2) is stabilized by N—H···O and O—H···N hydrogen bonds (Table 2) between the EDTB and glycol solvent molecules.

Experimental

Chemicals of reagent grade were used without further purification. The title compound was synthesized by refluxing stoichiometric quantities (1:4 molar ratio) of EDTA and *o*-phenylenediamine in glycol at 463–468 K for 16 h. The solution was then cooled to room temperature. Crystals of (I) formed in the bottom of the flask after one month.

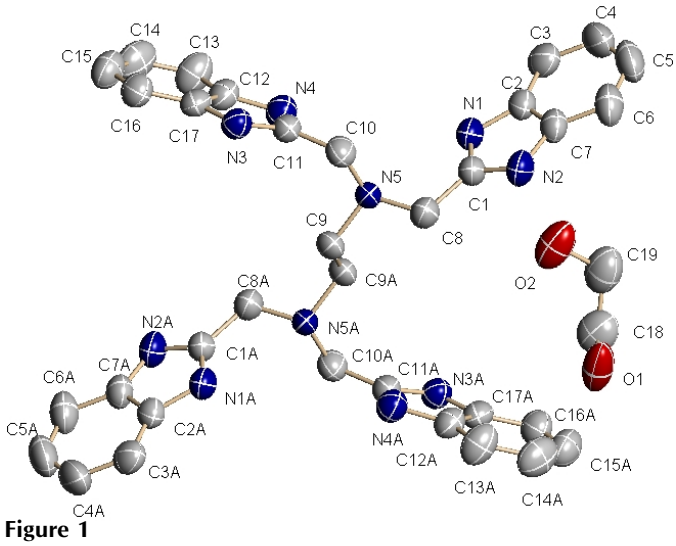


Figure 1
A view of (I), with 50% probability displacement ellipsoids [symmetry code: (A) $-x, 1-y, -z$]. H atoms have been omitted for clarity.

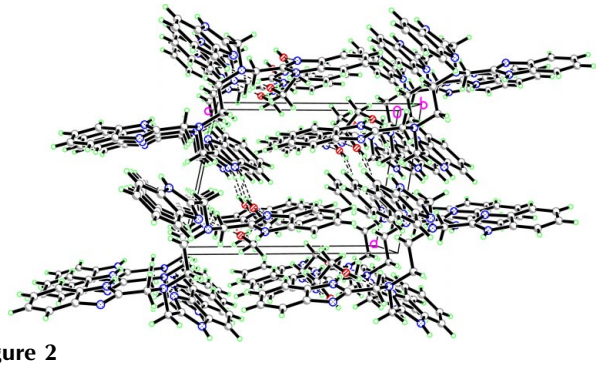


Figure 2
A packing diagram for (I), viewed along the *b* axis.

Crystal data

$C_{34}H_{32}N_{10}\cdot 2C_2H_6O_2$
$M_r = 704.83$
Triclinic, $P\overline{1}$
$a = 9.2367\ (15)$ Å
$b = 9.7383\ (16)$ Å
$c = 11.5791\ (19)$ Å
$\alpha = 85.764\ (3)^\circ$
$\beta = 76.748\ (3)^\circ$
$\gamma = 66.394\ (3)^\circ$
$V = 928.8\ (3)$ Å^3
$Z = 1$
$D_x = 1.260$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1058 reflections
$\theta = 2.3$–20.6°
$\mu = 0.09$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
0.33 × 0.33 × 0.12 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.973$, $T_{max} = 0.990$
4360 measured reflections
2992 independent reflections
2074 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.0^\circ$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.250$
$S = 1.08$
2992 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1469P)^2 + 0.1154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

C1—N2	1.315 (4)	C10—N5	1.470 (4)
C1—N1	1.339 (4)	C10—C11	1.489 (5)
C1—C8	1.488 (5)	C11—N4	1.316 (4)
C2—N1	1.377 (4)	C11—N3	1.347 (4)
C2—C3	1.384 (5)	C12—C13	1.380 (5)
C2—C7	1.385 (5)	C12—C17	1.385 (5)
C3—C4	1.374 (6)	C12—N4	1.390 (4)
C4—C5	1.361 (7)	C13—C14	1.379 (6)
C5—C6	1.369 (7)	C14—C15	1.392 (6)
C6—C7	1.392 (5)	C15—C16	1.359 (5)
C7—N2	1.406 (5)	C16—C17	1.389 (5)
C8—N5	1.463 (4)	C17—N3	1.372 (4)
C9—N5	1.466 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N4^{i}	0.82	1.94	2.734 (4)	164
O2—H2···N2	0.82	1.89	2.693 (4)	166
N3—H3*A*···O2^{ii}	0.86	1.93	2.767 (4)	164
N1—H1···O1^{iii}	0.86	1.95	2.774 (4)	160

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with O—H, N—H and C—H distances of 0.82, 0.86 and 0.93–0.97 Å, respectively, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(O)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*/*PC*.

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