

Qiong Shen,^a Xing-Can Shen,^b
Ming-Hua Zeng^b and
Seik Weng Ng^{c*}^aDepartment of Chemistry, School of
Pharmaceutical Sciences, GuangDong
Pharmaceutical University, Guangzhou 510006,
People's Republic of China, ^bDepartment of
Chemistry, Guangxi Normal University, Guilin
541000, Guangxi, People's Republic of China,
and ^cDepartment of Chemistry, University of
Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study

T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.010 \text{ \AA}$

R factor = 0.077

wR factor = 0.234

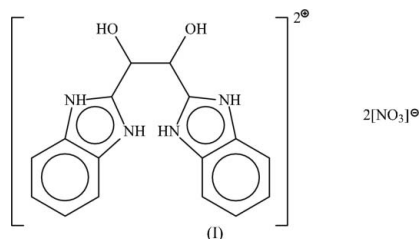
Data-to-parameter ratio = 6.4

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Racemic 2,2'-(1,2-dihydroxyethane-1,2-diyl)-
bis(benzimidazolium) dinitrateIn the crystal structure of the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_4\text{O}_2^{2+} \cdot 2\text{NO}_3^-$, the cation, which lies on a twofold rotation axis, interacts with the nitrate anions, giving rise to a hydrogen-bonded layer structure.

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Comment

Bis(2-benzimidazolyl)ethane-1,2-diol is a ligand that can bind to a metal atom in a terdentate manner; the structures of several metal complexes have been reported (Isele *et al.*, 2002, 2005; Shi *et al.*, 2001). There are three forms of the compound, two being optically active as chirality arises from either the D- or the L-tartaric acid reactant. The compound exhibits biological activity; its efficacy against the polio (Akihama *et al.*, 1968; O'Sullivan & Wallis, 1963) and rhino viruses (Roderick *et al.*, 1972) was discovered a long time ago.

The crystal structure of the neutral ligand has not been reported; the attempt to crystallize it from aqueous ethanol to which some nitric acid was added led to the isolation of the nitrate salt, (I) (Fig. 1). The dication lies on a special position of site symmetry 2; the dication interacts with the anions through hydrogen bonds (Table 1), giving rise to a layer structure.

As the compound is racemic, its crystallization in a space group that lacks inversion and mirror symmetry elements is merely coincidental.

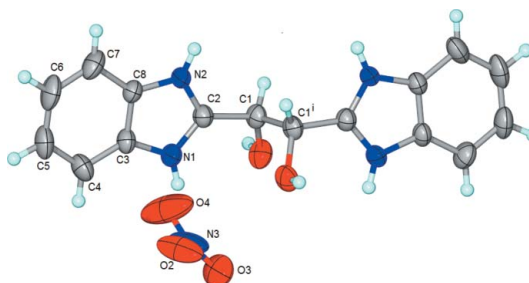


Figure 1

The cation and anion of the title compound, with displacement ellipsoids drawn at the 50% probability level and H atoms shown as spheres of arbitrary radii. [Symmetry code: (i) $1 - x, y, 1 - z$.]

Experimental

o-Phenylenediamine (10.8 g, 100 mmol) was dissolved in 5.5 M hydrochloric acid (100 ml). To this solution, racemic tartaric acid (7.5 g, 50 mmol) was added. The solution was refluxed overnight. The 1,2-bis(benzimidazolyl)-1,2-ethanediol hydrochloride that separated was collected and then neutralized with 10% aqueous ammonia. The yellow base was recrystallized twice from a 2:1 ethanol–water mixture to which several drops of nitric acid had been added.

Crystal data

$C_{16}H_{16}N_4O_2^{2+} \cdot 2NO_3^-$ $Z = 2$
 $M_r = 420.35$ $D_x = 1.504 \text{ Mg m}^{-3}$
 Monoclinic, $C2$ $Mo K\alpha$ radiation
 $a = 13.120 (2) \text{ \AA}$ $\mu = 0.12 \text{ mm}^{-1}$
 $b = 7.596 (1) \text{ \AA}$ $T = 295 (2) \text{ K}$
 $c = 10.391 (2) \text{ \AA}$ Block, yellow
 $\beta = 116.357 (3)^\circ$ $0.35 \times 0.18 \times 0.12 \text{ mm}$
 $V = 927.9 (2) \text{ \AA}^3$

Data collection

Bruker SMART area-detector 876 independent reflections
 diffractometer 839 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{int} = 0.032$
 Absorption correction: none $\theta_{max} = 25.0^\circ$
 2266 measured reflections

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.1739P)^2 + 1.1826P]$
 $R[F^2 > 2\sigma(F^2)] = 0.077$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.234$ $(\Delta/\sigma)_{max} = 0.001$
 $S = 1.06$ $\Delta\rho_{max} = 1.15 \text{ e \AA}^{-3}$
 876 reflections $\Delta\rho_{min} = -0.36 \text{ e \AA}^{-3}$
 137 parameters
 H-atom parameters constrained

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1o \cdots O3^i$	0.85	1.98	2.790 (7)	159
$N1-H1n \cdots O2$	0.85	2.01	2.845 (9)	169
$N2-H2n \cdots O2^{ii}$	0.85	2.11	2.839 (9)	144

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

In the absence of anomalous scattering atoms, Friedel pairs were merged. The three N–O distances were restrained to be within 0.01 Å of each other, as were the O···O distances in the nitrate anion. The anion was restrained to be nearly planar. H atoms were placed at calculated positions (C–H = 0.93–0.98, N–H = 0.85 and O–H = 0.85 Å) and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C,N,O)$. The final difference Fourier map had a peak of 1.15 e \AA^{-3} at 2.59 Å from O3, 2.64 Å from O2 and 2.55 Å from H1; attempts to refine this peak as either an O or an N atom led to a large displacement parameter. The peak is probably an artefact as PLATON (Spek, 2003) did not find any solvent-accessible voids.

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XSEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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