Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*R*,*S*)-2,2′-(Ethane-1,2-diyldiiminio)dibutan-1-ol dinitrate

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Received 5 November 2006
Accepted 8 November 2006

Key indicators

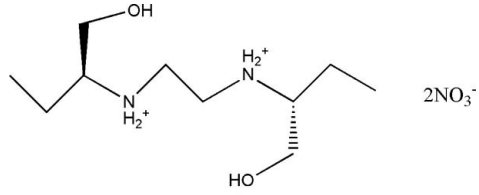
Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.003 Å
R factor = 0.060
wR factor = 0.158
Data-to-parameter ratio = 17.9

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

The asymmetric unit of the title compound, $C_{10}H_{26}N_2O_2^{2+}\cdot 2NO_3^-$, comprises one half of an N-diprotonated (*R*,*S*)-ethambutol cation, which lies about a centre of symmetry, and a nitrate anion. In the crystal structure, a two-dimensional network is formed *via* intermolecular O—H···O and N—H···O hydrogen bonds.

Comment

The title compound, (I), is a nitrate salt of (*R*,*S*)-ethambutol (Bai, Zhang, Zhang *et al.*, 2006). It is derived from the *meso* form of (*S*,*S*)-ethambutol (Bai, Zhang, Qin *et al.*, 2006), which is a key intermediate for the synthesis of ethambutol hydrochloride, a widely used chiral antitubercular agent (Fadnavis *et al.*, 1999). The cation, protonated on both N atoms, lies about a centre of inversion located at the mid-point of the central C—C bond (Fig. 1). Bond lengths and angles are normal (Allen *et al.*, 1987) and similar to those found in the neutral (*R*,*S*)-molecule (Bai, Zhang, Zhang *et al.*, 2006) and in the isomeric chiral (*S*,*S*)-cation (Bai, Ning *et al.*, 2006).



(I)

In the crystal structure, the two nitrate anions form N1—H···O2 hydrogen bonds to the protonated N atoms (Fig. 1). Further stabilization is provided by an extensive network of N—H···O and O—H···O hydrogen bonds (Fig. 2 and Table 1), forming layers parallel to the *ac* plane. The layers are further linked through hydrogen bonding to the nitrate anions.

Experimental

The title compound was prepared by the reaction of nitric acid with (*R*,*S*)-ethambutol. Colourless single crystals of (I) were grown by slow evaporation of a methanol solution.

Crystal data

$C_{10}H_{26}N_2O_2^{2+}\cdot 2NO_3^-$
M_r = 330.35
Monoclinic, *C*2/*c*
a = 23.350 (5) Å
b = 5.5367 (11) Å
c = 13.167 (3) Å
β = 99.03 (3)°
V = 1681.2 (6) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo *Kα* radiation
μ = 0.11 mm^{-1}
T = 113 (2) K
Block, colorless
0.20 × 0.12 × 0.06 mm

Data collection

Rigaku Saturn CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.978, T_{max} = 0.990
6205 measured reflections
1989 independent reflections
1321 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.158
S = 1.12
1989 reflections
111 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2 + 0.2435P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0105 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2	0.92 (2)	1.92 (2)	2.817 (2)	165.5 (18)
N1—H1A···O2^{i}	0.90 (2)	2.47 (2)	3.046 (2)	122.3 (16)
N1—H1A···O4^{i}	0.90 (2)	1.99 (2)	2.888 (2)	172.4 (19)
O1—H1···O4^{ii}	0.86 (3)	2.34 (3)	3.012 (3)	136 (3)
O1—H1···O3^{ii}	0.86 (3)	2.25 (3)	3.090 (3)	169 (3)

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

H atoms on N and O atoms were located in a difference Fourier map and then refined freely with their displacement parameterss tied to $1.2U_{eq}$(N) and $1.5U_{eq}$(O). Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.98 Å and U_{iso}(H) = $1.2U_{eq}$(C) for CH, C—H = 0.97 Å and U_{iso}(H) = $1.2U_{eq}$(C) for CH_2 and C—H = 0.96 Å and U_{iso}(H) = $1.5U_{eq}$(C) for CH_3 H atoms.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

Financial support by the Science Project of the Hebei Education Department (grant No. 2005350) and the Science Foundation of Hebei University (grant No. 2005046) is gratefully acknowledged.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bai, G.-Y., Ning, H.-S., Simpson, J., Qin, X.-Y. & Li, N. (2006). *Acta Cryst.* E**62**, o4567–o4568.
Bai, G.-Y., Zhang, C.-F., Qin, X.-Y., Zhang, Y.-C. & Zeng, T. (2006). *Acta Cryst.* E**62**, o4222–o4223.
Bai, G.-Y., Zhang, C.-F., Zhang, Y.-C., Zeng, T. & Li, J.-S. (2006). *Acta Cryst.* E**62**, o2173–o2174.
Bruker (2001). *SHELXTL*. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Fadnavis, N. W., Sharfuddin, M. & Vadivel, S. K. (1999). *Tetrahedron Asymmetry*, **10**, 4495–4500.

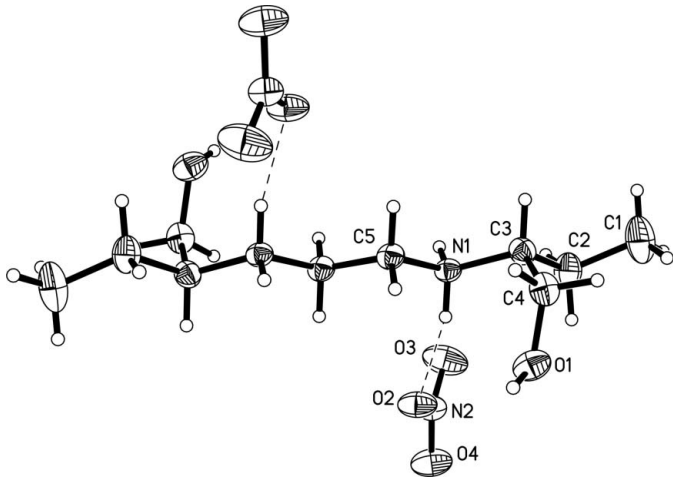


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids. Unlabeled atoms are related to the labeled atoms by the symmetry code $(1-x, -y, 1-z)$. Dashed lines indicate hydrogen bonds.

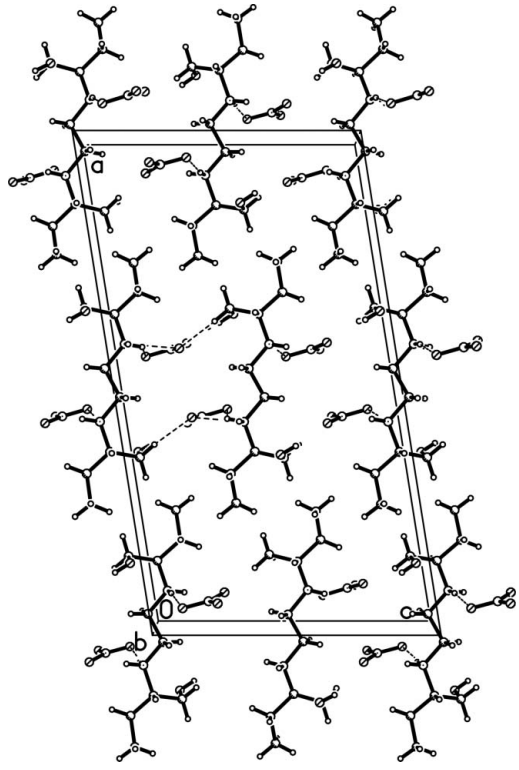


Figure 2
Packing diagram for (I), with hydrogen bonds shown as dashed lines.

Jacobson, R. (1998). Private communication to Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2005). *CrystalClear*. Version 1.36. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.