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Key indicators

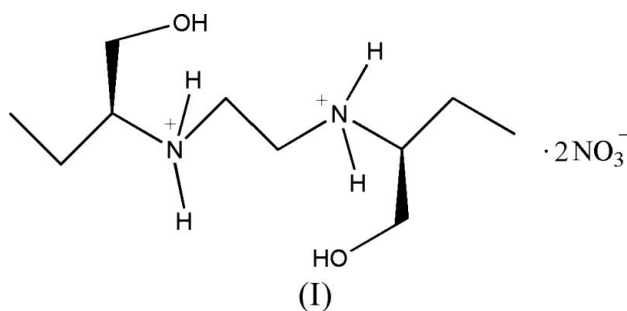
Single-crystal X-ray study
 $T = 113$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.037
 wR factor = 0.082
Data-to-parameter ratio = 9.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(*S,S*)-2,2'-(Ethane-1,2-diyl-diiminio)dibutan-1-ol
dinitrate**

In the title structure, $\text{C}_{10}\text{H}_{26}\text{N}_2\text{O}_2^{2+} \cdot 2\text{NO}_3^-$, the ethambutol molecule exists in the cationic form, having two protonated amine groups, and is charge-balanced by two nitrate anions. In the crystal structure, a two-dimensional network is formed *via* intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.

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Comment

The title compound, (I), is the dinitrate salt of ethambutol (Bai *et al.*, 2006), which is a key intermediate in the synthesis of ethambutol hydrochloride, a widely used chiral anti-tuberculosis agent (Fadnavis *et al.*, 1999). A view of the asymmetric unit of (I) is shown in Fig. 1. The ethambutol molecule in (I) exists in the cationic form, having two protonated amino groups. The dication is charged balanced by two nitrate anions. The bond distances and angles in the cation and the anion are normal within experiment error (Allen *et al.*, 1987). Selected torsion angles are given in Table 1.



In the crystal structure, anions and cations are connected *via* intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds, forming a two-dimensional network perpendicular to the a axis (Fig. 2 and Table 2).

Experimental

The title compound was prepared by the reaction of nitric acid (0.065 g, 65%) with ethambutol (0.1 g, 0.0003 mol). Colourless single crystals were grown by slow evaporation of a solution of (I) in methanol–water (6:1).

Crystal data

 $\text{C}_{10}\text{H}_{26}\text{N}_2\text{O}_2^{2+} \cdot 2\text{NO}_3^-$
 $M_r = 330.35$
Monoclinic, $P2_1$
 $a = 8.769$ (3) Å
 $b = 9.598$ (4) Å
 $c = 9.583$ (4) Å
 $\beta = 97.829$ (6)°
 $V = 799.0$ (5) Å³ $Z = 2$
 $D_x = 1.373$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 113$ (2) K
Block, colorless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART-1000
diffractometer
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
 $T_{\min} = 0.964$, $T_{\max} = 0.977$

7494 measured reflections
2011 independent reflections
1830 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 27.9^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.082$
 $S = 1.07$
2011 reflections
218 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.28 \text{ e } \text{\AA}^{-3}$

Table 1

Selected torsion angles ($^\circ$).

C5–N1–C1–C3	55.3 (2)	N1–C5–C6–N2	174.91 (17)
C5–N1–C1–C2	179.53 (17)	C6–N2–C7–C9	56.3 (2)
N1–C1–C3–C4	–173.70 (16)	C6–N2–C7–C8	–176.23 (18)
C1–N1–C5–C6	169.86 (16)		

Table 2

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2–H2D \cdots O8 ⁱ	0.85 (3)	2.13 (3)	2.939 (3)	158 (2)
N2–H2D \cdots O6 ⁱ	0.85 (3)	2.54 (3)	3.041 (2)	119.0 (19)
N1–H1C \cdots O3 ⁱⁱ	0.95 (3)	1.87 (3)	2.804 (2)	168 (2)
N1–H1C \cdots O4 ⁱⁱ	0.95 (3)	2.53 (2)	3.073 (2)	116.8 (17)
N2–H2C \cdots O7	1.05 (2)	1.83 (2)	2.880 (3)	176 (2)
N2–H2C \cdots O6	1.05 (2)	2.44 (2)	3.068 (2)	117.2 (16)
N1–H1B \cdots O3 ⁱⁱⁱ	0.90 (2)	2.06 (3)	2.877 (2)	149 (2)
N1–H1B \cdots O5 ⁱⁱⁱ	0.90 (2)	2.39 (2)	3.180 (3)	146 (2)
O2–H2 \cdots O4	0.76 (3)	2.07 (3)	2.818 (2)	166 (3)
O2–H2 \cdots O5	0.76 (3)	2.63 (3)	3.262 (2)	141 (3)
O1–H1A \cdots O7 ⁱⁱⁱ	0.83 (3)	1.99 (3)	2.824 (2)	173 (3)

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

In the absence of significant anomalous dispersion effects, Friedel pairs were merged; the absolute configuration is known from that of the starting material. H atoms bonded to N and O atoms were refined, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ or $1.5U_{\text{eq}}(\text{O})$. H atoms bonded to C atoms were included in calculated positions, with $C-H = 0.98-1.00 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

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

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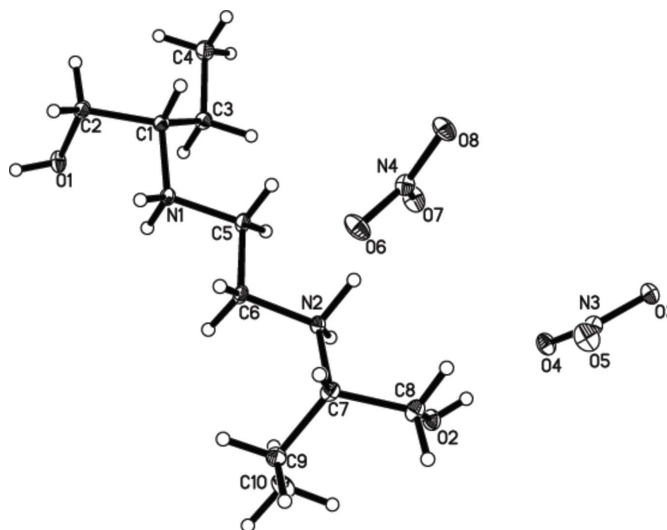


Figure 1

The asymmetric unit of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

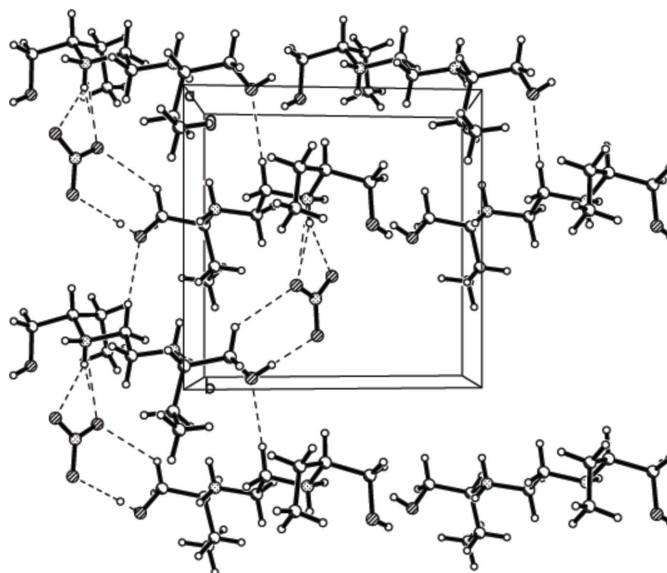


Figure 2

Part of the crystal structure of (I), with hydrogen bonds shown as dashed lines.

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