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Key indicators

Single-crystal X-ray study
 $T = 113\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.029
 wR factor = 0.071
Data-to-parameter ratio = 9.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*(S,S)*-*N,N'*-Bis(2-hydroxy-2-butyl)ethylenediammonium oxalate pentahydrate

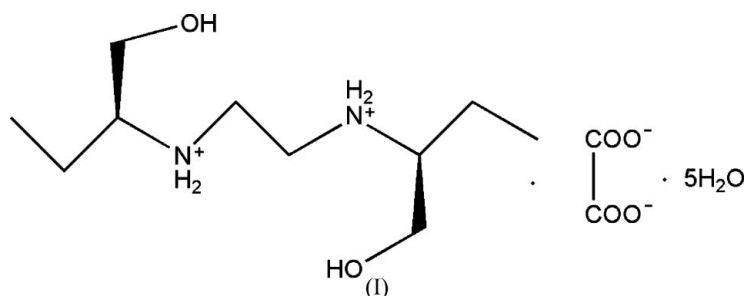
The title compound, $\text{C}_{10}\text{H}_{26}\text{N}_2\text{O}_2^{2+} \cdot \text{C}_2\text{O}_4^{2-} \cdot 5\text{H}_2\text{O}$, is the oxalate salt of the drug ethambutol. Both the ethambutol cation and the oxalate anion lie about a crystallographic twofold axis which bisects the central C—C bonds of both ions. The O atom of one solvent water molecule also lies on a twofold axis. The crystal structure is stabilized by intermolecular O—H...N and N—H...O hydrogen bonds.

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Comment

Ethambutol hydrochloride is a widely used chiral anti-tuberculosis agent (Fadnavis *et al.*, 1999). The title compound, (I), is the oxalate salt of (*S,S*)-ethambutol (Bai *et al.*, 2006), a precursor of ethambutol hydrochloride, and its structure is reported here (Fig. 1).



The asymmetric unit of (I) comprises one half of an *N*-protonated (*S,S*)-ethambutol cation, one half of an oxalate anion and 2.5 solvent water molecules. The anion, cation and one solvent water molecule all lie about crystallographic twofold axes. All bond lengths and angles in (I) are within

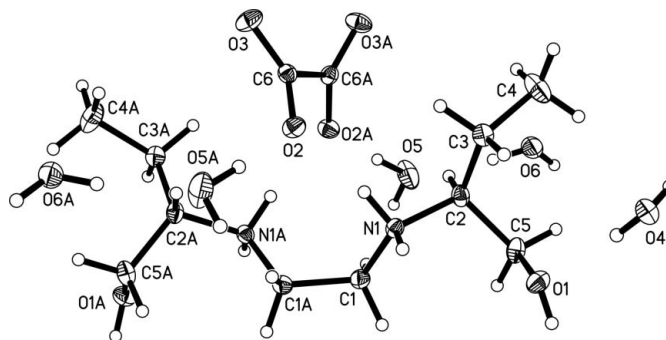


Figure 1

The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids. Atoms labeled with the suffix A are related by the symmetry code $(1 - x, y, \frac{3}{2} - z)$.

normal ranges (Allen *et al.*, 1987) and are similar to those observed in the corresponding chloride (Hamalainen *et al.*, 1985; Rubin-Preminger *et al.*, 2004) and bromide (Godfrey *et al.*, 1992) salts. The crystal structure is stabilized by extensive intermolecular O—H...N and N—H...O hydrogen bonding, linking cations, anions and water molecules into a three-dimensional network (Fig. 2 and Table 1).

Experimental

The title compound was prepared by the reaction of ethambutol (Bai *et al.*, 2006) with oxalic acid in water. Colourless single crystals of (I) were grown by slow evaporation of an aqueous solution.

Crystal data

$C_{10}H_{26}N_2O_2^{2+} \cdot C_2O_4^{2-} \cdot 5H_2O$	$Z = 4$
$M_r = 384.43$	$D_x = 1.269 \text{ Mg m}^{-3}$
Orthorhombic, $C222_1$	Mo $K\alpha$ radiation
$a = 8.2104 (15) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$b = 10.3092 (19) \text{ \AA}$	$T = 113 (2) \text{ K}$
$c = 23.771 (5) \text{ \AA}$	Block, colorless
$V = 2012.0 (7) \text{ \AA}^3$	$0.16 \times 0.14 \times 0.06 \text{ mm}$

Data collection

Rigaku Saturn diffractometer	12718 measured reflections
ω scans	1376 independent reflections
Absorption correction: multi-scan (Jacobson, 1998)	1314 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.976$, $T_{\max} = 0.993$	$R_{\text{int}} = 0.041$
	$\theta_{\text{max}} = 27.9^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.473P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
1376 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
141 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0137 (13)

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O4—H4...O1	0.81 (2)	2.02 (2)	2.8314 (16)	174 (2)
N1—H1C...O2	0.908 (19)	1.86 (2)	2.7599 (17)	168.3 (17)
N1—H1C...O2 ⁱ	0.908 (19)	2.622 (19)	3.1210 (17)	115.4 (15)
N1—H1D...O3 ⁱⁱ	0.89 (2)	1.90 (2)	2.7273 (17)	152.1 (17)
O5—H5C...O2 ⁱ	0.93 (3)	1.87 (3)	2.7789 (18)	166 (2)
O5—H5D...O3 ⁱⁱⁱ	0.84 (3)	1.91 (3)	2.7330 (18)	166 (3)
O6—H6A...O5	0.88 (3)	1.85 (3)	2.7132 (18)	169 (2)
O1—H1...O6 ^{iv}	0.82 (2)	1.85 (2)	2.6643 (17)	169 (2)
O6—H6B...O4 ^v	0.86 (3)	2.01 (3)	2.8630 (18)	173 (2)

Symmetry codes: (i) $-x + 1, y, -z + \frac{3}{2}$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (v) $x - 1, y, z$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. All H atoms bound to N or O atoms were located in a difference Fourier map and refined isotropically, with $U_{\text{iso}}(\text{H}) =$

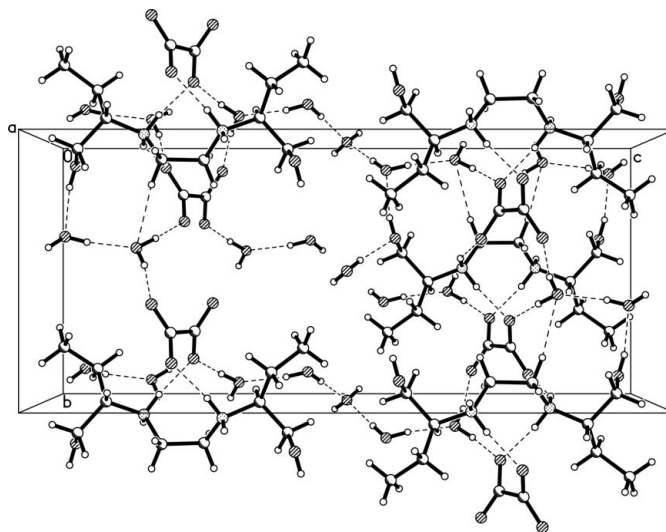


Figure 2
Part of the crystal structure of (I), with hydrogen bonds drawn as dashed lines.

$1.2U_{\text{eq}}(\text{N})$ and $1.5U_{\text{eq}}(\text{O})$. Other H atoms were positioned geometrically and refined using a riding model, with C—H = 1.00 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH groups, C—H = 0.99 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH₂ groups, C—H = 0.98 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for CH₃ groups.

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, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

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