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(+)-(1*S*,2*S*,3*S*,4*R*,5*R*)-1-Ammonio-5-hydroxymethylcyclohexane-2,3,4-triol chloride

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.124
Data-to-parameter ratio = 15.9

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

The six-membered ring of the title compound, $C_7H_{16}NO_4{\cdot}Cl$ has a slightly distorted chair conformation. One of the hydroxyl groups is disordered. The molecules of (I) are linked into a three-dimensional network by hydrogen bonds.

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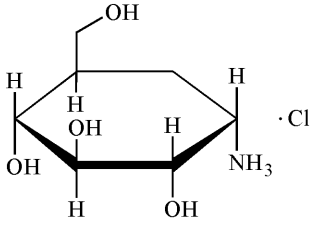
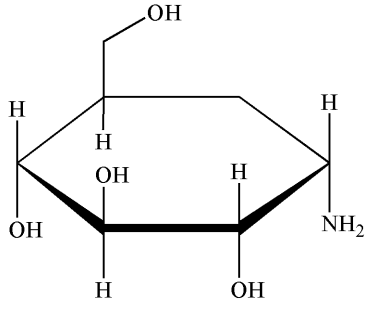
Comment

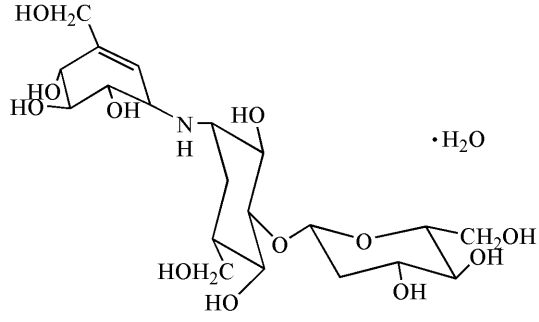
(+)-(1*S*,2*S*,3*S*,4*R*,5*R*)-1-Amino-5-hydroxymethylcyclohexane-2,3,4-triol (validamine), (II), is one of the degradation products from validamycins, (III), which are antibiotics used in controlling the sheath blight disease of rice plants (Kameda & Horii, 1972). Biological experiments show that it acts by inhibition of α-glucosidase (Kameda *et al.*, 1984).



(I)



(II)



(III)

In the course of synthesis of validamine derivatives, we have prepared the title compound, (I) (Fig. 1). The puckering parameters (Cremer & Pople, 1975) for the six-membered ring are: Q = 0.5469 Å, θ = 176.23° and φ = 300.14°. These indicate that the ring has a slightly distorted chair conformation. Such a chair-like conformation has been observed in the crystal structure of the hydrobromide of compound (II) (Kamiya *et al.*, 1971). The geometric parameters for (I) are given in Table 1.

The hydroxymethyl group is disordered and was modeled using two sets of atomic sites with refined occupancies of

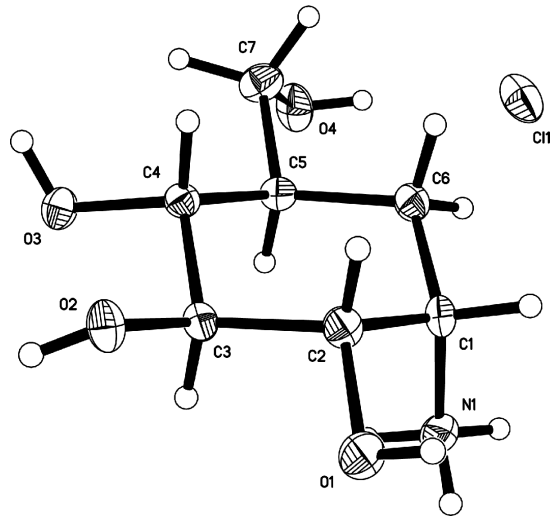


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids. Only one of the disordered components is shown.

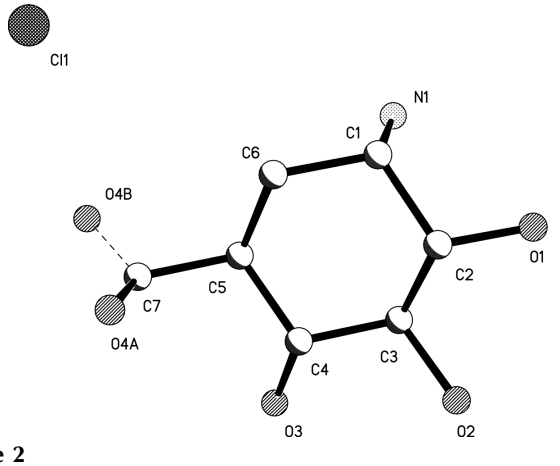


Figure 2
The disorder of the hydroxyl group in (I). H atoms have been omitted for clarity.

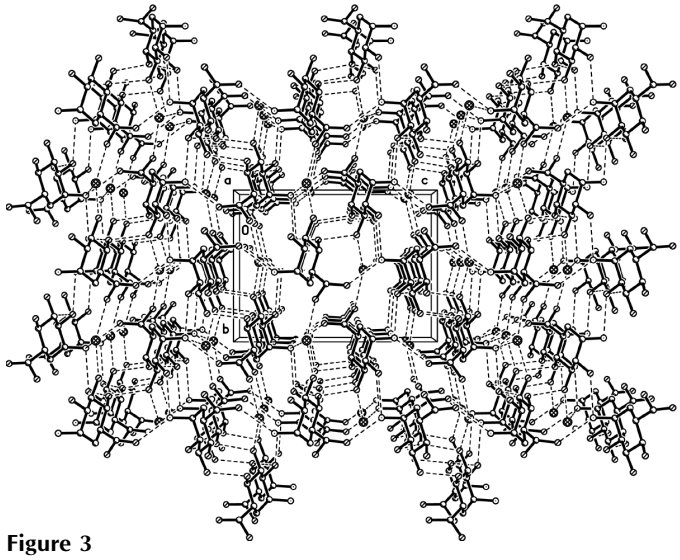


Figure 3
The packing of (I), viewed approximately down the *a* axis. Hydrogen bonds involving atoms O4*A* and O4*B* are shown as dashed lines.

0.513 (6) and 0.487 (6) (Fig. 2). The molecules are linked into a complex three-dimensional network by hydrogen bonds (Fig. 3 and Table 2).

Experimental

Compound (I) was prepared by hydrochlorination of compound (II), which was obtained according to the procedure of Ogawa *et al.* (1989). 2 *N* HCl (4 ml, 8 mmol) was added dropwise to a solution of compound (II) (1.3 g, 7.3 mmol) in water (5 ml) and the mixture was evaporated under vacuum to dryness. The resulting solid was dissolved in 80% MeOH to yield colorless crystals (m.p. 509–510 K). Spectroscopic analysis, ^{1}H NMR (D_2O, p.p.m.): 1.59–1.74 (*m*, 2H), 1.75–1.98 (*m*, 1H), 3.25 (*t*, 1H, J = 9.6 Hz), 3.42 (*t*, 1H, J = 9.3 Hz), 3.56–3.70 (*m*, 4H); ^{13}C NMR (D_2O, p.p.m.): 28.86, 40.80, 54.17, 64.37, 72.83, 74.89, 76.57; ESI–MS *m*/*z*: 178 ($M^+ + 1 - HCl$).

Crystal data

$C_7H_{16}NO_4^+\cdot Cl^-$
M_r = 213.66
Orthorhombic, $P2_12_12_1$
a = 6.6151 (8) Å
b = 10.3964 (12) Å
c = 13.9546 (17) Å
V = 959.7 (2) Å^3
Z = 4
D_x = 1.479 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 960 reflections
θ = 2.9–27.0°
μ = 0.38 mm^{-1}
T = 293 (2) K
Block, colorless
0.50 × 0.34 × 0.32 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.832, T_{max} = 0.887
6249 measured reflections
2096 independent reflections
2007 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.1°
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -17 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.31
2096 reflections
132 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0098P)^2 + 1.3067P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 857 Friedel pairs
Flack parameter = 0.21 (14)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.497 (4)	C3—C4	1.520 (4)
C1—C6	1.516 (5)	C4—C5	1.527 (4)
C1—C2	1.529 (5)	C5—C7*A*	1.526 (5)
C2—C3	1.520 (4)	C7A—O4*A*	1.475 (6)
N1—C1—C6	112.3 (3)	O3—C4—C5	110.7 (3)
C6—C1—C2	111.1 (3)	C7*A*—C5—C4	111.4 (3)
C3—C2—C1	111.9 (3)	C1—C6—C5	114.0 (3)
C2—C3—C4	110.9 (3)	O4*A*—C7*A*—C5	111.8 (4)
N1—C1—C2—O1	49.5 (4)	O3—C4—C5—C7*A*	63.4 (3)
N1—C1—C2—C3	−72.0 (4)	C3—C4—C5—C6	−53.2 (3)
C6—C1—C2—C3	53.5 (4)	N1—C1—C6—C5	73.1 (4)
C1—C2—C3—O2	−176.9 (3)	C2—C1—C6—C5	−51.5 (4)
C1—C2—C3—C4	−56.3 (4)	C4—C5—C6—C1	51.3 (4)
O2—C3—C4—C5	174.0 (2)	C6—C5—C7*A*—O4*A*	−60.7 (4)
C2—C3—C4—C5	56.4 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2^{i}	0.82	1.92	2.738 (4)	176
O1—H1D···O3^{ii}	0.82	2.04	2.745 (4)	143
N1—H1C···O2^{iii}	0.89	2.41	3.006 (4)	125
N1—H1C···O1^{iii}	0.89	2.11	2.954 (4)	157
N1—H1A···Cl1^{iv}	0.89	2.32	3.183 (3)	162
N1—H1B···O4A^{v}	0.89	2.20	2.862 (6)	131
O2—H2A···Cl1^{vi}	0.82	2.24	3.026 (3)	161
O4B—H4B···Cl1	0.82	2.21	2.999 (5)	160

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

All H atoms were positioned geometrically and refined with a riding model, with C—H distances of 0.97 (CH_2) or 0.98 Å (CH), N—H distances of 0.89 Å and O—H distances of 0.82 Å, and with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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