Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(+)-(1*S*,2*S*,3*S*,4*R*,5*R*)-*N*-(1,3-Dihydroxyprop-2-yl)-2,3,4-trihydroxy-5-(hydroxymethyl)-1-cyclohexanaminium picrate

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Received 22 March 2005
Accepted 20 April 2005
Online 27 April 2005

In the title compound, $C_{10}H_{22}NO_6^+\cdot C_6H_2N_3O_7^-$, the cyclohexane ring has a slightly distorted chair conformation. Molecules are linked into chains by hydrogen bonds.

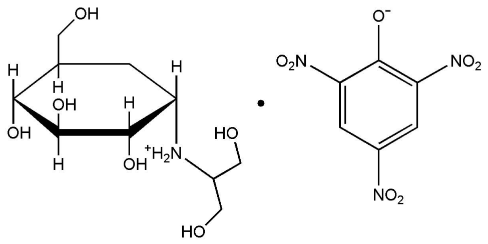
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.103
Data-to-parameter ratio = 7.8

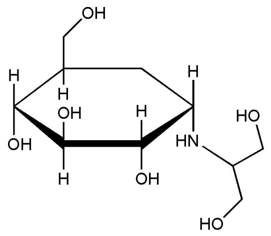
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

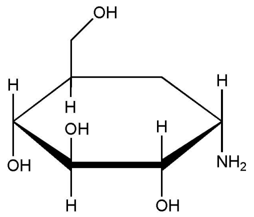
(+)-(1*S*,2*S*,3*S*,4*R*,5*R*)-1-Amino-5-hydroxymethyl-*N*-(1,3-dihydroxyprop-2-yl)cyclohexane-2,3,4-triol, (II), one of the derivatives of validamine, (III), shows strong activity as an inhibitor of α-glucosidase (Horii *et al.*, 1987). As a result of its high hygroscopicity, it is difficult to obtain crystals of (II) suitable for X-ray analysis. We have, therefore, prepared the picrate of (II), which did provide suitable single crystals.



(I)



(II)



(III)

The cyclohexane ring of the title picrate salt, (I), has a slightly distorted chair conformation (Table 1), which is similar to the crystal structure of validamine chloride (Chang *et al.*, 2004). In (I), there are three intramolecular hydrogen bonds (Fig. 1). Molecules of (I) are linked into infinite one-dimensional chains in the [010] direction by intermolecular hydrogen bonds (Fig. 2 and Table 2). The packing mode of (I) is shown in Fig. 3.

Experimental

Compound (I) was prepared by picration of (II), which was obtained according to the procedure of Horii *et al.* (1987). Compound (II) (1.55 g, 6.17 mmol) was dissolved in MeOH (20 ml) and 2,4,6-trinitrophenol (2.82 g, 12.31 mmol) was added. The mixture was heated under reflux for 4 h and then stirred at ambient temperature (298 K)

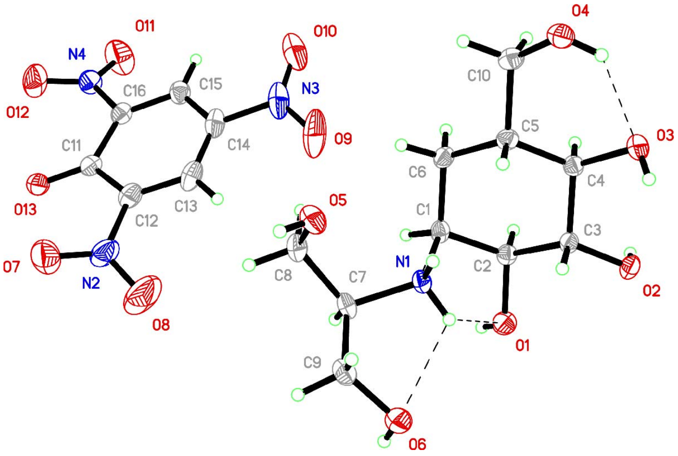


Figure 1
View of (I), shown with 50% probability displacement ellipsoids. H atoms have been omitted. Dashed lines indicate hydrogen bonds.

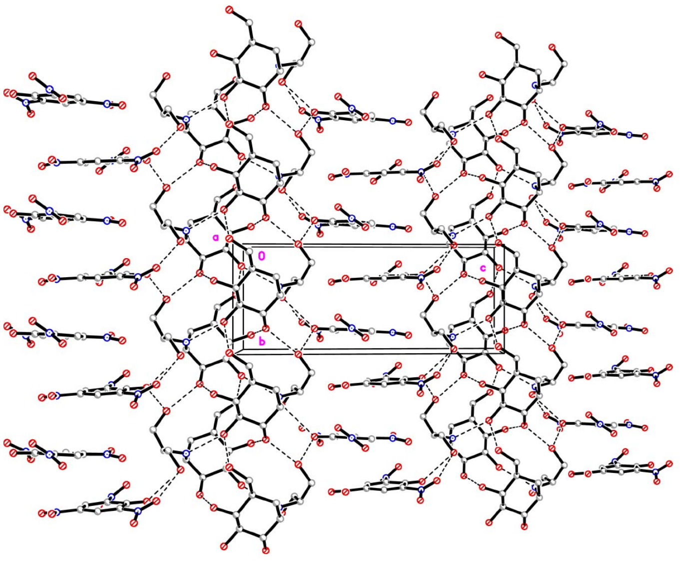


Figure 2
A view of the infinite one-dimensional hydrogen-bonded (dashed lines) chains extending in the [010] direction. H atoms have been omitted.

until it crystallized. The crystals were recrystallized from 80% MeOH (m.p. 436.5–438.5 K). ^{1}H NMR (D_2O): δ 1.62–1.73 (*m*, 2H), 2.10 (*m*, 1H), 3.20–3.30 (*m*, 1H), 3.38–3.48 (*m*, 2H), 3.54–3.62 (*m*, 2H), 3.62–3.84 (*m*, 6H), 8.60 (*s*, 2H); ^{13}C NMR (D_2O): δ 25.18, 39.10, 58.03, 58.63, 58.75, 62.32, 62.38, 70.99, 72.60, 74.80, 127.86, 128.54, 141.85, 163.21; ESI–MS *m*/*z*: 503 ($M^+ + 1 +$ Na).

Crystal data

$C_{10}H_{22}NO_6^+ \cdot C_6H_2N_3O_7^-$
$M_r = 480.39$
Monoclinic, $P2_1$
$a = 8.7362\ (11)$ Å
$b = 6.8235\ (8)$ Å
$c = 16.957\ (2)$ Å
$\beta = 95.769\ (2)°$
$V = 1005.7\ (2)$ Å^3
$Z = 2$

$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 966 reflections
$\theta = 3.2$–$26.8°$
$\mu = 0.14$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
0.33 × 0.25 × 0.19 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.884$, $T_{max} = 0.974$
6583 measured reflections

2383 independent reflections
2138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.0°$
$h = -10 \rightarrow 11$
$k = -8 \rightarrow 7$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.05$
2383 reflections
304 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.2641P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.520 (3)	C5—C10	1.528 (4)
C1—C6	1.523 (4)	C5—C6	1.528 (4)
C1—C2	1.534 (4)	C7—N1	1.509 (3)
C2—O1	1.421 (3)	C7—C8	1.515 (4)
C2—C3	1.537 (3)	C7—C9	1.523 (4)
C3—O2	1.423 (3)	C8—O5	1.413 (4)
C3—C4	1.522 (4)	C9—O6	1.420 (4)
C4—O3	1.423 (3)	C10—O4	1.427 (4)
C4—C5	1.527 (4)		
N1—C1—C6	112.7 (2)	C4—C5—C10	111.1 (2)
N1—C1—C2	107.34 (19)	C4—C5—C6	110.5 (2)
C6—C1—C2	111.5 (2)	C10—C5—C6	109.9 (2)
O1—C2—C1	110.6 (2)	C1—C6—C5	113.4 (2)
O1—C2—C3	107.94 (19)	N1—C7—C8	111.5 (2)
C1—C2—C3	111.4 (2)	N1—C7—C9	105.7 (2)
O2—C3—C4	112.08 (19)	C8—C7—C9	114.0 (2)
O2—C3—C2	111.5 (2)	O5—C8—C7	110.8 (2)
C4—C3—C2	108.94 (19)	O6—C9—C7	110.5 (3)
O3—C4—C3	111.6 (2)	O4—C10—C5	112.8 (2)
O3—C4—C5	111.5 (2)	C7—N1—C1	117.03 (19)
C3—C4—C5	111.22 (19)		
N1—C1—C2—O1	49.8 (3)	N1—C1—C6—C5	70.2 (3)
N1—C1—C2—C3	−70.3 (2)	C2—C1—C6—C5	−50.6 (3)
C6—C1—C2—C3	53.6 (3)	C4—C5—C6—C1	52.1 (3)
O1—C2—C3—O2	55.8 (3)	N1—C7—C8—O5	−58.1 (3)
C1—C2—C3—C4	−58.3 (3)	C9—C7—C8—O5	61.5 (3)
O2—C3—C4—O3	−50.5 (3)	N1—C7—C9—O6	−50.6 (3)
C2—C3—C4—C5	60.4 (3)	C4—C5—C10—O4	−73.6 (3)
O3—C4—C5—C10	55.4 (3)	C8—C7—N1—C1	−70.2 (3)
C3—C4—C5—C6	−57.2 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O6	0.90	2.22	2.707 (3)	114
N1—H1A···O1	0.90	2.24	2.751 (3)	115
N1—H1B···O2^{i}	0.90	2.17	2.839 (3)	131
O1—H1C···O5^{ii}	0.82	1.96	2.671 (3)	144
O2—H2A···O4^{ii}	0.82	1.86	2.676 (3)	171
O3—H3A···O1^{i}	0.82	2.03	2.844 (3)	171
O4—H4A···O3	0.82	2.14	2.790 (3)	136
O4—H4A···O6^{i}	0.82	2.30	2.932 (3)	134
O5—H5···O13^{iii}	0.82	1.84	2.662 (3)	175
O6—H6C···O13^{iv}	0.82	2.15	2.885 (3)	149
O6—H6C···O12^{iv}	0.82	2.26	2.898 (4)	135

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

H atoms on C and N atoms were positioned geometrically and refined with a riding model, with distances C—H = 0.97 (CH_2), 0.98 (CH) or 0.93 Å (aromatic) and N—H = 0.90 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). H atoms of OH groups were constrained to an ideal geometry, with O—H distances of 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O), but each group was allowed to rotate. In the absence of any significant anomalous scattering, Friedel equivalents were merged prior to the final refinement and the absolute configuration was assigned by reference to the known chirality of (III) established in our previous paper (Chang *et al.*, 2004).

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*.

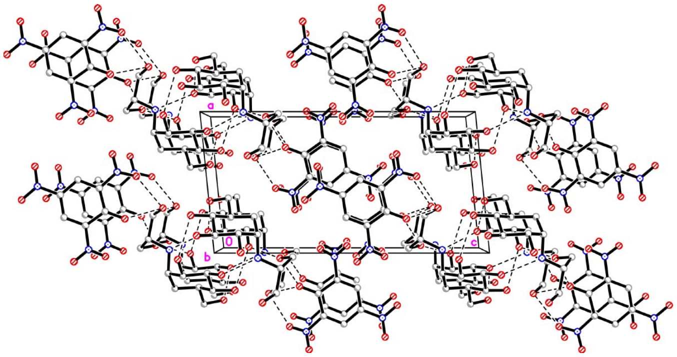


Figure 3

Packing diagram of (I), viewed along the *b* axis. Hydrogen-bonded (dashed lines) chains are perpendicular to the plane of the paper. H atoms have been omitted.

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