Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

(2*R*,3*R*,4*R*,5*S*)-3,4,5-Trihydroxy-2-(2-hydroxyethyl)piperidinium chloride

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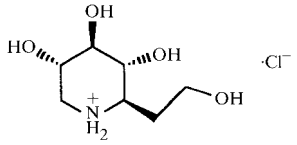
Received 20 December 1999
Accepted 1 March 2000

Data validation number: IUC0000050

The absolute configuration at the new stereogenic centre during the key step of the total synthesis was established by X-ray analysis of the title compound, $C_7H_{15}NO_4^+\cdot Cl^-$.

Comment

The title compound [(I); alternative name: 1,5,6-trideoxy-1,5-imino-D-*gluco*-heptitol hydrochloride] was prepared by palladium(II)-catalyzed aminocarbolation of (2*S*,3*S*,4*R*)-1-benzylamino-2,3-di-*O*-benzylhex-5-ene-2,3,4-triol with subsequent lactone-ring opening and deprotection (Szolcsányi *et al.*, 2000).



(I)

The bond lengths are consistent with average values in the usual sources [*International Tables for Crystallography* (1992, Vol. C, Table 9.5.1.1)]. The Cl^- anion is bonded in the structure by hydrogen bonds [$Cl\cdots$H15 2.225 Å, $Cl^{i}\cdots$H16 2.725 Å, $Cl^{ii}\cdots$H12 2.420 Å and $Cl^{iii}\cdots$H1 2.300 Å; symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z$; (ii) $-x+1, y-\frac{1}{2}, -z$; (iii) $x+1, y, z$].

Experimental

The title compound was prepared by hydrogenolysis of the benzylated diol with H_2 on Pd/C at room temperature in EtOH containing a few drops of aqueous HCl. The crude product was purified by ion-exchange chromatography (H^+ form, DOWEX 50WX8–400) and recrystallized from methanol/*tert*-butyl methyl ether (m.p. 450–452 K, $[\alpha]_D^{20} = +30$, $c = 0.55$, MeOH).

Crystal data

$C_7H_{16}NO_4^+\cdot Cl^-$	$D_x = 1.478$ Mg m^{-3}
$M_r = 213.66$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 20 reflections
$a = 6.8910$ (10) Å	$\theta = 4.53$–$10.24°$
$b = 7.3450$ (10) Å	$\mu = 0.382$ mm^{-1}
$c = 9.497$ (2) Å	$T = 293$ (2) K
$\beta = 92.46$ (3)°	Rectanglukar, colourless
$V = 480.24$ (14) Å^3	0.6 × 0.5 × 0.2 mm
$Z = 2$	

Data collection

Syntex $P2_1$ diffractometer	$R_{int} = 0.048$
θ–2θ scans	$\theta_{max} = 30.08°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 9$
$T_{min} = 0.791$, $T_{max} = 0.926$	$k = -10 \rightarrow 10$
1663 measured reflections	$l = -13 \rightarrow 13$
1566 independent reflections	2 standard reflections every 100 reflections
1222 reflections with $I > 2\sigma(I)$	intensity decay: 15%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1308P)^2 + 0.3128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.157$	$\Delta\rho_{max} = 0.84$ e Å^{-3}
$S = 0.933$	$\Delta\rho_{min} = -0.54$ e Å^{-3}
1461 reflections	
118 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.414 (6)	O4—C6	1.419 (5)
O2—C4	1.412 (4)	N1—C7	1.479 (5)
O3—C5	1.425 (4)	N1—C3	1.500 (5)
C7—N1—C3	114.2 (3)	C5—C4—C3	113.1 (3)
O1—C1—C2	107.6 (3)	O3—C5—C6	108.2 (3)
C1—C2—C3	113.8 (3)	O3—C5—C4	110.8 (3)
N1—C3—C2	109.8 (3)	C6—C5—C4	110.4 (3)
N1—C3—C4	109.6 (3)	O4—C6—C5	110.9 (3)
C2—C3—C4	110.9 (3)	O4—C6—C7	108.6 (3)
O2—C4—C5	110.1 (3)	C5—C6—C7	108.8 (3)
O2—C4—C3	106.0 (3)	N1—C7—C6	109.3 (3)

The number of Friedel pairs in the data set is 60.

Data collection: *Syntex $P2_1$ Software* (Syntex, 1973); cell refinement: *Syntex $P2_1$ Software*; data reduction: *XP*21 (Pavelčík, 1993); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); software used to prepare material for publication: *SHELXL*93.

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