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N,N,N′,N′-Tetramethylstreptamine 2,4,6-orthoformate hydrochloride

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In the title compound, *rac*-(1*s*,3*s*,5*R*,6*r*,7*S*,8*R*,9*S*)-8,9-bis(dimethylamino)-2,4,10-trioxatricyclo[3.3.1.1^{3,7}]decan-6-ol hydrochloride, $C_{11}H_{21}N_2O_4^+\cdot Cl^-$, there is an intramolecular asymmetric hydrogen-bond system N—H···N and a further classical hydrogen bond O—H···Cl$^-$. The structure is pseudo-*A*-centred.

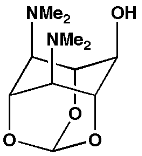
Key indicators

Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.135
Data-to-parameter ratio = 17.0

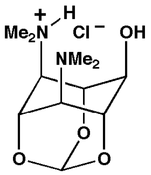
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We have studied the reactions of a number of derivatives of the synaxial diaminoalcohol (1) (Beckmann, 1998; Dean, 2000). The system is of interest in the context of enzyme mechanism and efficiency because of the close and fixed geometrical relationship between the two amino groups and the axial oxygen centre, and we have reported the crystal and molecular structure of (1) in the previous paper (Beckmann *et al.*, 2003). We report here the structure of the chloride of the conjugate acid, ($1H^+\cdot Cl^-$), and the different hydrogen-bonding arrangement in the ammonium cation.



(1)



($1H^+\cdot Cl^-$)

In contrast to the diaminoalcohol (1), which has an internal hydrogen bond between the alcohol OH group and one of the dimethylamino groups, the intramolecular hydrogen bond in the conjugate acid ($1H^+$) is unsymmetrically shared (Table 2 and Fig. 1) between the two amino groups. The angle N—H···N is 146° and the H atom is also relatively close to the neighbouring axial hydroxy-group O atom [H···O = 2.44 (3) Å]. Despite the narrow N—H···O angle, this could be regarded as the minor component of a highly asymmetric three-centre hydrogen bond. The hydroxy group is itself involved in a hydrogen bond to the chloride anion. An extensive system of non-classical hydrogen bonds (Table 2) links the residues to form a three-dimensional network (Fig. 2).

The bond lengths at the protonated nitrogen N1 are, as expected, longer than those at N2 (Table 1).

Experimental

Crystals of compound ($1H^+\cdot Cl^-$) (Brown & Kirby, 1997) were grown from a saturated solution in ethanol.

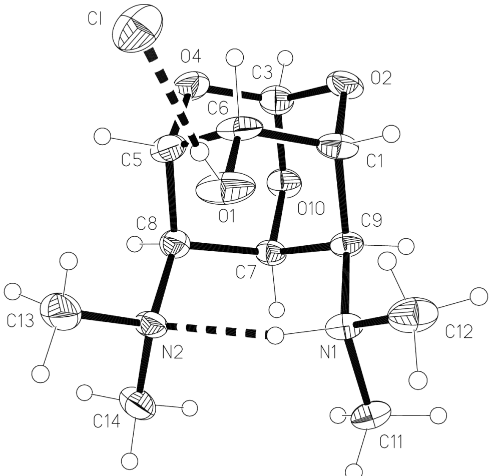


Figure 1
The cation of the title compound in the crystal. Displacement ellipsoids are drawn at the 50% probability level. H-atom radii are arbitrary. The dashed lines indicate hydrogen bonds.

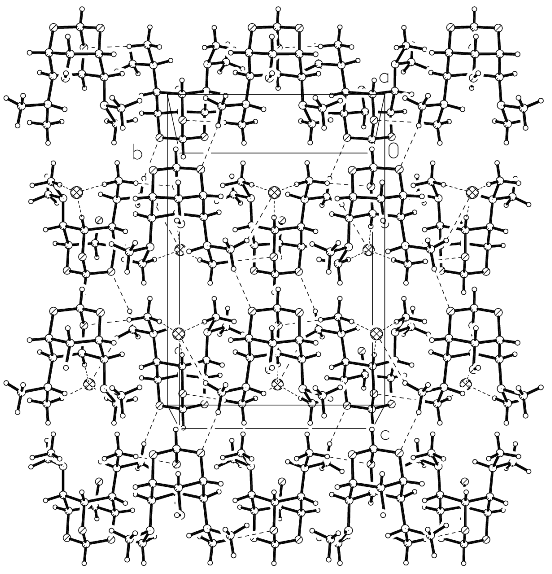


Figure 2
Packing diagram of the title compound, showing hydrogen bonds as thin dashed lines. The view direction is perpendicular to the *bc* plane.

Crystal data

$C_{11}H_{21}N_2O_4^+\cdot Cl^-$
$M_r = 280.75$
Monoclinic, $P2_1/c$
$a = 9.804$ (2) Å
$b = 9.730$ (2) Å
$c = 13.896$ (3) Å
$\beta = 100.82$ (2)°
$V = 1302.1$ (5) Å^3
$Z = 4$

$D_x = 1.432$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 52 reflections
$\theta = 10$–11.5°
$\mu = 0.30$ mm^{-1}
$T = 143$ (2) K
Tablet, colourless
0.50 × 0.40 × 0.15 mm

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
3416 measured reflections
2977 independent reflections
2090 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$

$\theta_{\rm max} = 27.5°$
$h = -12 \rightarrow 1$
$k = 0 \rightarrow 12$
$l = -17 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.135$
$S = 1.05$
2977 reflections
175 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.641P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.33$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—N2	1.481 (3)	C12—N1	1.495 (3)
C9—N1	1.507 (2)	C13—N2	1.470 (3)
C11—N1	1.499 (3)	C14—N2	1.477 (3)
C12—N1—C11	107.97 (17)	C13—N2—C14	107.11 (18)
C12—N1—C9	112.99 (17)	C13—N2—C8	112.64 (17)
C11—N1—C9	111.58 (16)	C14—N2—C8	109.56 (17)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H01⋯Cl	0.85 (4)	2.14 (5)	2.9821 (18)	170 (4)
N1—H02⋯N2	0.99 (3)	1.94 (3)	2.801 (3)	144 (3)
N1—H02⋯O1	0.99 (3)	2.44 (3)	2.863 (2)	105 (2)
C12—H12B⋯O2$^{\rm i}$	0.98	2.40	3.283 (3)	150
C13—H13B⋯O4$^{\rm ii}$	0.98	2.62	3.414 (3)	138
C3—H3⋯O10$^{\rm iii}$	1.00	2.67	3.555 (3)	148
C11—H11C⋯O10$^{\rm iv}$	0.98	2.55	3.516 (3)	169
C7—H7⋯Cl$^{\rm v}$	1.00	2.67	3.654 (2)	167
C11—H11A⋯Cl$^{\rm v}$	0.98	2.77	3.708 (2)	161
C14—H14C⋯Cl$^{\rm v}$	0.98	2.87	3.839 (2)	170
C12—H12A⋯Cl$^{\rm vi}$	0.98	2.82	3.685 (3)	148

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

H atoms bonded to N and O atoms were identified in a difference synthesis and refined freely. Methyl H atoms were similarly identified, the methyl groups then idealized (C—H = 0.98 Å, all angles 109.5°) and refined as rigid groups allowed to rotate but not tip. Other H atoms were included using a riding model with fixed C—H bond lengths of 1.00 Å; $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$ of the parent atom. The structure is pseudo-*A*-centred; the higher symmetry cell, corresponding after reorientation to space group $C2/m$, would, however, require the cation to display mirror symmetry, which is only approximate in the true structure (*cf.* Fig. 2). Furthermore, a large number of highly significant reflections are observed that do not correspond to a centred lattice.

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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