

*N,N,N',N'*-Tetramethylstreptamine  
2,4,6-orthoformateClaus Beckmann,<sup>a</sup> Peter G.  
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## Key indicators

Single-crystal X-ray study

T = 173 K

Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$ 

R factor = 0.036

wR factor = 0.101

Data-to-parameter ratio = 16.0

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

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<sup>3,7</sup>]decan-6-ol, C<sub>11</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub>, an intramolecular hydrogen bond with H···N = 2.09 (2) Å is observed between the hydroxy group and one of the dimethylamino groups; this hydrogen bond is appreciably non-linear (146° at H). There is a short H···N contact of 2.61 (2) Å from the same hydroxy H atom to the other N atom.

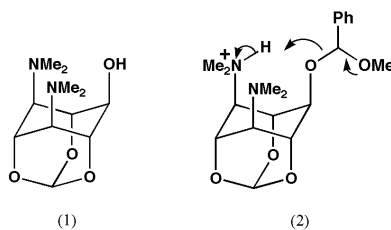
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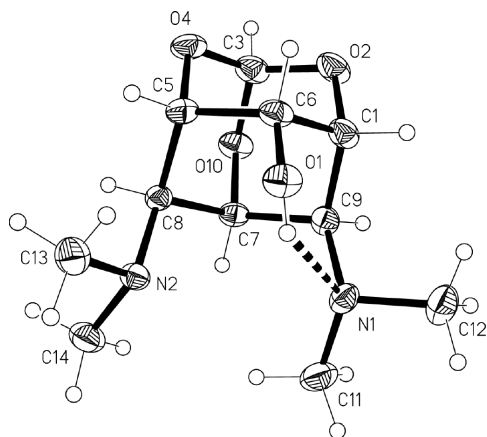
Online 31 March 2003

## Comment

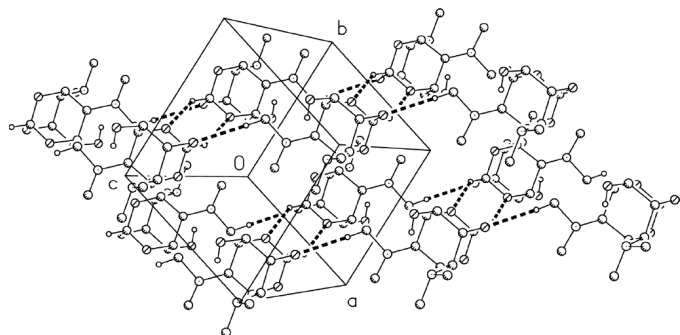
We have studied the reactions of a number of derivatives of the synaxial diaminoalcohol (1) (Beckmann, 1998; Dean, 2000). The system is of interest in the context of enzyme mechanism and efficiency because of the close and fixed geometrical relationship between the two amino groups and the axial oxygen centre. The interatomic distances between these centres are of the order of 2.8 Å, typical of the distances observed between centres involved in intramolecular hydrogen bonds, and this might be expected to favour proton transfer catalysis when an in-flight proton can be stabilized by interactions with two (perhaps even all three) centres. The acetal (2) (Brown & Kirby, 1997) is indeed one of three dialkyl acetals of benzaldehyde shown to be hydrolysed with intramolecular general acid catalysis. The detailed geometry of the system, and in particular the properties of any hydrogen bonds present, is thus of particular interest. We report here the crystal and molecular structure of (1), and in the following paper (Beckmann *et al.*, 2003) the structure of its hydrochloride, which shows a different pattern of hydrogen bonding.



The diaminoalcohol (1) has an intramolecular O—H···N bond hydrogen bond between the alcohol OH group and one of the dimethylamino groups, which prevents the molecule from displaying more than approximate mirror symmetry. The hydrogen bond is not linear (the angle O—H···N is 146°) and the proton is displaced slightly in the direction of the second nitrogen centre [N2···H = 2.61 (2) Å and N2···O = 3.018 (1) Å]. But, to all intents and purposes, this is a normal three-centre hydrogen bond. The dimensions involving the N



**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 line indicates the intramolecular hydrogen bond.



**Figure 2**  
Two columns of molecules associated by C—H...O interactions (thick dashed bonds). The view direction is perpendicular to the *ab* plane.

atoms (Table 1) are not significantly affected by the hydrogen bond.

Two weak C—H...O contacts (Table 2) link the molecules to form columns parallel to [110] (Fig. 2).

## Experimental

Crystals of compound (1) (Brown & Kirby, 1997) were grown from a saturated solution in toluene.

### Crystal data

$C_{11}H_{20}N_2O_4$	$Z = 2$
$M_r = 244.29$	$D_x = 1.416 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.9344 (8) \text{ \AA}$	Cell parameters from 62 reflections
$b = 7.5439 (10) \text{ \AA}$	$\theta = 3.5\text{--}12.5^\circ$
$c = 12.1650 (16) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$\alpha = 100.386 (10)^\circ$	$T = 173 (2) \text{ K}$
$\beta = 104.367 (8)^\circ$	Lath, colourless
$\gamma = 105.528 (6)^\circ$	$0.60 \times 0.40 \times 0.20 \text{ mm}$
$V = 572.85 (13) \text{ \AA}^3$	

### Data collection

Siemens P4 diffractometer	$\theta_{\text{max}} = 27.5^\circ$
$\omega$ scans	$h = -8 \rightarrow 8$
Absorption correction: none	$k = -9 \rightarrow 9$
2719 measured reflections	$l = 0 \rightarrow 15$
2598 independent reflections	3 standard reflections
1931 reflections with $I > 2\sigma(I)$	every 297 reflections
$R_{\text{int}} = 0.016$	intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.101$   
 $S = 1.04$   
 2598 reflections  
 162 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.0004P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C8—N2	1.4729 (14)	C12—N1	1.4740 (17)
C9—N1	1.4772 (15)	C13—N2	1.4613 (16)
C11—N1	1.4639 (17)	C14—N2	1.4680 (16)
C11—N1—C12	107.41 (10)	C13—N2—C14	106.91 (10)
C11—N1—C9	111.14 (10)	C13—N2—C8	112.89 (9)
C12—N1—C9	110.08 (10)	C14—N2—C8	109.32 (10)

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H01...N1	0.83 (2)	2.09 (2)	2.8109 (15)	145.8 (19)
C13—H13B...O2 <sup>i</sup>	0.98	2.55	3.5198 (16)	170
C3—H3...O4 <sup>ii</sup>	1.00	2.66	3.5442 (16)	148

Symmetry codes: (i)  $x - 1, y - 1, z$ ; (ii)  $1 - x, 2 - y, -z$ .

The H atom of the OH group was identified in a difference synthesis and refined freely. Methyl H atoms were similarly identified, the methyl groups then idealized (C—H = 0.98  $\text{\AA}$ , all angles  $109.5^\circ$ ) 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  $\text{\AA}$ ;  $U_{\text{iso}}(\text{H})$  values were fixed at  $1.2U_{\text{eq}}$  of the parent atom.

Data collection: XSCANS (Fait, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1994); software used to prepare material for publication: SHELXL97.

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