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Structure of 1-Deoxycastanospermine

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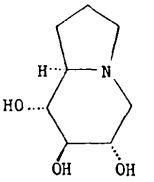
Abstract

The crystal structure of (6*S*,7*R*,8*R*,8a*R*)-6,7,8-trihydroxyoctahydroindolizine, $C_8H_{15}NO_3$, has been determined by X-ray diffraction. The six-membered ring is in the chair form and the puckering parameters [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1358] are $Q = 0.574$ Å, $\theta = 8.3°$, $\varphi_2 = 13.9°$. The five-membered ring is in the envelope form with $Q = 0.428$ Å and $\varphi_2 = 9.2°$.

Comment

The naturally occurring polyhydroxylated indolizidine alkaloid castanospermine is a potent inhibitor of glycosidase enzymes, particularly those involved in the Golgi processing of the N-linked oligosaccharide moiety of glycoproteins in mammals. (Pan *et al.*, 1983; Szumilo, Kaushal & Elbein, 1986; Campbell, Molyneux & Jones, 1987). It has since attracted considerable interest because of its antiviral activity against HIV and related viruses (Tyms *et al.*, 1987; Sunkara, Bowlin, Liu & Sjoerdsma, 1987; Gunters *et al.*, 1987; Walker *et al.*, 1987). The enantiospecific synthesis of polyhydroxylated indolizidines related to castanospermine has also received much attention (Reymond, Pinkerton & Vogel, 1991). The synthesis of the title compound (1) has been reported by Hendry, Hough & Richardson (1988). We now



(1)

report the X-ray crystal structure of the compound. The crystal is orthorhombic, space group $P2_12_12_1$. The structure was solved by direct methods using *SHELXTL-Plus* (Siemens Analytical X-ray Instruments, Inc., 1990). Coordinates of non-H atoms are given in Table 1. Selected bond lengths and angles are given in Table 2. The molecule and its atomic numbering is shown in Fig. 1. Crystal packing and hydrogen bonding are shown in Fig. 2. These figures also depict the correct absolute configuration of the molecule as established by the synthesis of the compound. The two rings are joined with the torsion angles C(3)—N(1)—C(8a)—C(8) and C(5)—N(1)—C(8a)—C(1) being 172.2 (3) and 169.1 (3)°, respectively, so that all the non-H atoms lie close to a plane

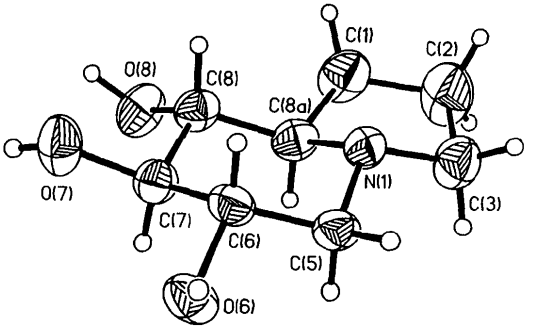


Fig. 1. Perspective view of the 1-deoxycastanospermine molecule with atomic numbering.

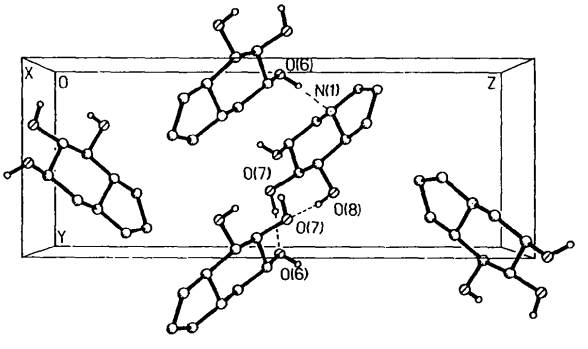


Fig. 2. Packing of the crystal viewed along the *a* axis showing the hydrogen bonds.

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with maximum deviations of 0.5 Å from the plane. Bond lengths are within the expected ranges. The average C—C bond length is 1.524 (4) Å, with N—C 1.469 (6) and C—O 1.430 (5) Å. The average bond angle in the six-membered ring is 111 (1)° and in the five-membered ring 104 (1)°. Neighbouring molecules are held together by three different intermolecular hydrogen bonds: O(7)···O(6) 2.743, O(6)···N(1) 2.770, O(8)···O(7) 2.821 Å.

Experimental

Crystal data

$C_8H_{15}NO_3$
$M_r = 173.2$
Orthorhombic
$P2_12_12_1$
$a = 6.841$ (8) Å
$b = 7.219$ (6) Å
$c = 18.51$ (2) Å
$V = 914.3$ (17) Å^3
$Z = 4$
$D_x = 1.258$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 12 reflections
$\theta = 3.5$–$10.5°$
$\mu = 0.096$ mm^{-1}
$T = 298$ K
Prism
0.45 × 0.45 × 0.25 mm
Colourless
Crystal source: recrystallized from mixture of methanol, ether and ethyl acetate

Data collection

Siemens *R3m/V* diffractometer
ω scans
Absorption correction: none
875 measured reflections
857 independent reflections
731 observed reflections [$I>2.5\sigma(I)$]
$\theta_{max} = 24°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 21$
2 standard reflections monitored every 98 reflections
intensity variation: 2%

Refinement

Refinement on F
Final $R = 0.040$
$wR = 0.050$
$S = 1.74$
731 reflections
110 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F)+0.0004F^2]$
$(\Delta/\sigma)_{max} = 0.009$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: $F^* = F[1+0.002\chi F^2/\sin(2\theta)]^{-0.25}$
Extinction coefficient: $\chi = 0.0042$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

U_{eq} = 1/3(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
C(1)	0.8350 (6)	0.1943 (6)	0.3107 (2)	0.060 (1)
C(2)	0.7567 (6)	0.3837 (6)	0.2859 (3)	0.072 (2)
C(3)	0.5552 (5)	0.4018 (5)	0.3214 (2)	0.053 (1)
C(5)	0.3778 (5)	0.2205 (5)	0.4135 (2)	0.038 (1)
C(6)	0.4096 (4)	0.0911 (4)	0.4766 (2)	0.036 (1)
C(7)	0.5292 (5)	−0.0777 (4)	0.4543 (2)	0.038 (1)
C(8)	0.7134 (5)	−0.0262 (4)	0.4128 (2)	0.038 (1)
C(8a)	0.6664 (5)	0.1087 (5)	0.3526 (2)	0.041 (1)
N(1)	0.5664 (3)	0.2731 (3)	0.3827 (2)	0.037 (1)
O(6)	0.2242 (3)	0.0274 (3)	0.5033 (1)	0.044 (1)
O(7)	0.5742 (3)	−0.1803 (3)	0.5181 (1)	0.050 (1)
O(8)	0.8005 (4)	−0.1854 (3)	0.3806 (1)	0.053 (1)

Table 2. *Geometric parameters* (Å, °)

C(1)—C(2)	1.539 (6)	C(1)—C(8a)	1.521 (5)
C(2)—C(3)	1.533 (6)	C(3)—N(1)	1.468 (5)
C(5)—C(6)	1.512 (5)	C(5)—N(1)	1.461 (4)
C(6)—C(7)	1.524 (5)	C(6)—O(6)	1.437 (4)
C(7)—C(8)	1.522 (5)	C(7)—O(7)	1.428 (4)
C(8)—C(8a)	1.514 (5)	C(8)—O(8)	1.425 (4)
C(8a)—N(1)	1.479 (5)		
C(2)—C(1)—C(8a)	104.4 (3)	C(1)—C(2)—C(3)	105.1 (3)
C(2)—C(3)—N(1)	103.3 (3)	C(6)—C(5)—N(1)	109.6 (3)
C(5)—C(6)—C(7)	111.2 (3)	C(5)—C(6)—O(6)	109.6 (3)
C(7)—C(6)—O(6)	108.1 (2)	C(6)—C(7)—C(8)	112.7 (3)
C(6)—C(7)—O(7)	107.8 (3)	C(8)—C(7)—O(7)	111.4 (3)
C(7)—C(8)—C(8a)	110.6 (3)	C(7)—C(8)—O(8)	111.1 (3)
C(8a)—C(8)—O(8)	107.4 (3)	C(1)—C(8a)—C(8)	118.4 (3)
C(1)—C(8a)—N(1)	102.5 (3)	C(8)—C(8a)—N(1)	109.7 (3)
C(3)—N(1)—C(5)	114.8 (3)	C(3)—N(1)—C(8a)	103.9 (3)
C(5)—N(1)—C(8a)	110.3 (3)		

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55479 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1013]

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