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Structure of Fortimicin B

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(*Received* 7 *February* 1978; *accepted* 18 *April* 1978)

Abstract. *O*-α-D-2,6-Diamino-6-methyl-2,3,4,6-tetradeoxyglucopyranosyl-(1 → 4)-L-3-amino-1-methoxy-6 methylamino-1,3,6-trideoxy-*chiro*-inositol, $C_{15}H_{31}N_4O_5.3{\cdot}5H_2O$, M_r = 347·438, orthorhombic, $P2_12_12_1$, a = 13·966 (2), b = 16·472 (2), c = 9·522 (1) Å, Z = 4, D_x = 1·245 g cm^{-3}. The structure was solved by direct methods. The final R value was 0·078 for 1713 reflexions. The two six-membered rings are in the chair conformation and are bound through the α-linkage. The solvent molecules are hydrogen bonded to each other and to the molecule.

Introduction. Fortimicin B is a member of the fortimicins, which are potent broad-spectrum aminocyclitol antibiotics produced by a strain of *Micromonospora olivoasterospona* (Nara *et al.*, 1977; Okachi *et al.*, 1977). X-ray structure determination of the title molecule was carried out in order to confirm the structure proposed from chemical and spectroscopic evidence (Egan *et al.*, 1977) and to determine the absolute configuration unequivocally.

Fortimicin B was recrystallized from a methanol solution containing a trace of water. A crystal with dimensions 0·5 × 0·4 × 0·3 mm was used, sealed in a glass capillary. The precise lattice constants and intensity data were obtained from measurements on a Rigaku computer-controlled four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. All reflexions within the range of $2\theta \leq 50°$ were collected by use of the θ–2θ scan mode with a scanning rate of 4° min^{-1}. Stationary background counts were accumulated for 10 s before and after each scan. A total of 2201 independent reflexions were obtained, of which 1713 were non-zero reflexions [$|F_o| \geq 3{\cdot}0\sigma(|F|)$]. No correction was made for absorption [μ(Mo $K\alpha$) = 1·082 cm^{-1}]. The phases for 436 reflexions with $|E| \geq$

Table 1. *Final fractional coordinates* ($\times 10^4$; *hydrogen atoms* $\times 10^3$), *with their standard deviations in parentheses*

The hydrogen atoms are numbered according to the atoms to which they are attached. O_w denotes the oxygen atom of water.

	x	*y*	*z*		*x*	*y*	*z*
C(1)	5080 (4)	1245 (3)	1425 (6)	H(C6)	463 (3)	73 (3)	−28 (5)
C(2)	4190 (4)	1716 (3)	1862 (6)	H*A*(C7)	418 (4)	100 (3)	590 (6)
C(3)	3499 (4)	1166 (3)	2652 (6)	H*B*(C7)	305 (4)	127 (3)	559 (6)
C(4)	3168 (4)	460 (3)	1736 (7)	H*C*(C7)	378 (4)	174 (3)	507 (5)
C(5)	4051 (4)	−7 (3)	1186 (6)	H*A*(C8)	133 (4)	53 (3)	151 (6)
C(6)	4792 (4)	531 (3)	524 (6)	H*B*(C8)	113 (4)	97 (3)	2 (6)
C(7)	3885 (6)	1251 (4)	5100 (7)	H*C*(C8)	153 (4)	159 (3)	160 (6)
C(8)	1623 (4)	1013 (5)	953 (8)	H(C1′)	540 (3)	3 (3)	−180 (5)
C(1′)	5891 (4)	−131 (3)	−1145 (5)	H(C2′)	620 (3)	−110 (3)	−227 (5)
C(2′)	6091 (4)	−1036 (3)	−1270 (6)	H*A*(C3′)	688 (3)	−120 (3)	58 (6)
C(3′)	6972 (4)	−1267 (3)	−451 (8)	H*B*(C3′)	716 (3)	−180 (3)	−55 (6)
C(4′)	7808 (4)	−736 (4)	−891 (7)	H*A*(C4′)	801 (3)	−81 (3)	−183 (5)
C(5′)	7565 (4)	165 (3)	−773 (6)	H*B*(C4′)	835 (3)	−91 (3)	−40 (5)
C(6′)	8333 (4)	714 (4)	−1375 (6)	H(C5′)	751 (3)	24 (3)	13 (5)
C(7′)	9253 (5)	655 (4)	−559 (7)	H(C6′)	847 (3)	61 (3)	−218 (5)
N(1)	5724 (4)	1767 (3)	594 (6)	H*A*(C7′)	937 (4)	10 (3)	−62 (6)
N(4)	2592 (3)	724 (3)	548 (6)	H*B*(C7′)	922 (4)	93 (3)	35 (6)
N(2′)	5238 (3)	−1496 (3)	−776 (6)	H*C*(C7′)	976 (3)	111 (3)	−79 (6)
N(6′)	8048 (4)	1565 (3)	−1486 (6)	H*A*(N1)	586 (3)	208 (3)	116 (5)
O(1)	5627 (2)	41 (2)	267 (4)	H*B*(N1)	615 (3)	151 (3)	29 (5)
O(2)	4436 (3)	2414 (2)	2639 (4)	H(N4)	243 (3)	86 (3)	8 (5)
O(3)	3909 (3)	799 (3)	3870 (4)	H*A*(N2′)	504 (4)	−188 (3)	−129 (6)
O(5)	3714 (3)	−623 (2)	257 (4)	H*B*(N2′)	602 (3)	−199 (3)	−79 (5)
O(5′)	6696 (2)	335 (2)	−1541 (4)	H*A*(N6′)	753 (3)	168 (3)	−215 (5)
$O_w(1)$	6405 (3)	2867 (3)	3338 (6)	H*B*(N6′)	804 (3)	179 (3)	−67 (5)
$O_w(2)$	7885 (4)	2855 (3)	1284 (6)	H(O2)	456 (3)	290 (3)	243 (5)
$O_w(3)$	9836 (4)	2682 (4)	1891 (6)	H(O5)	426 (3)	−87 (3)	−8 (5)
$O_w(4)$	1425 (10)	2883 (9)	3546 (16)	H*A*(O_w1)	567 (4)	271 (3)	307 (6)
H(C1)	539 (3)	109 (3)	227 (5)	H*B*(O_w1)	633 (4)	324 (3)	302 (6)
H(C2)	395 (3)	188 (3)	107 (5)	H*A*(O_w2)	738 (4)	281 (3)	185 (6)
H(C3)	305 (3)	147 (3)	302 (5)	H*B*(O_w2)	781 (4)	321 (3)	62 (7)
H(C4)	271 (3)	4 (3)	223 (5)	H*A*(O_w3)	926 (4)	267 (4)	129 (7)
H(C5)	428 (3)	−24 (3)	194 (5)	H*B*(O_w3)	1036 (4)	258 (4)	285 (7)

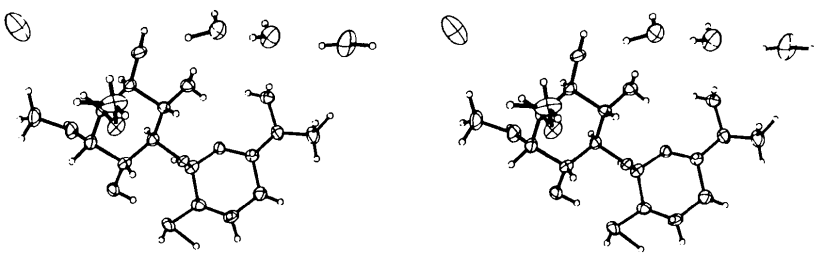

Fig. 1. Stereoscopic view with thermal ellipsoids at 30% probability.

1·2 were derived with *MULTAN* (Germain, Main & Woolfson, 1971). An *E* map computed from the solution with the best consistency revealed the positions of the 24 non-hydrogen atoms. The four non-hydrogen atoms of the solvent molecules were located on a difference map. Hydrogen atoms were found from the subsequent difference synthesis. One of the water oxygen atoms, $O_w(4)$, was refined successfully with an occupancy of 0·5. The final *R* value was 0·078. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The positional parameters are given in Table 1.*

Discussion. A stereoscopic view of the molecule is shown in Fig. 1. The numbering system is given in Fig.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33560 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

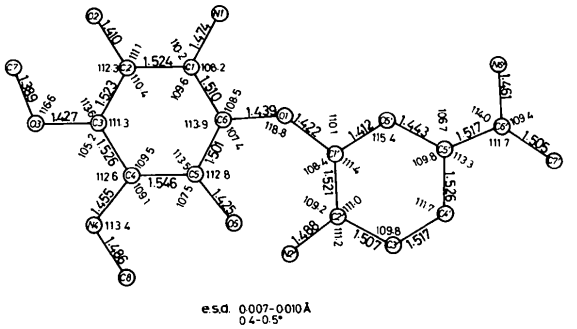

Fig. 2. The numbering system, with bond lengths (Å) and angles (°).

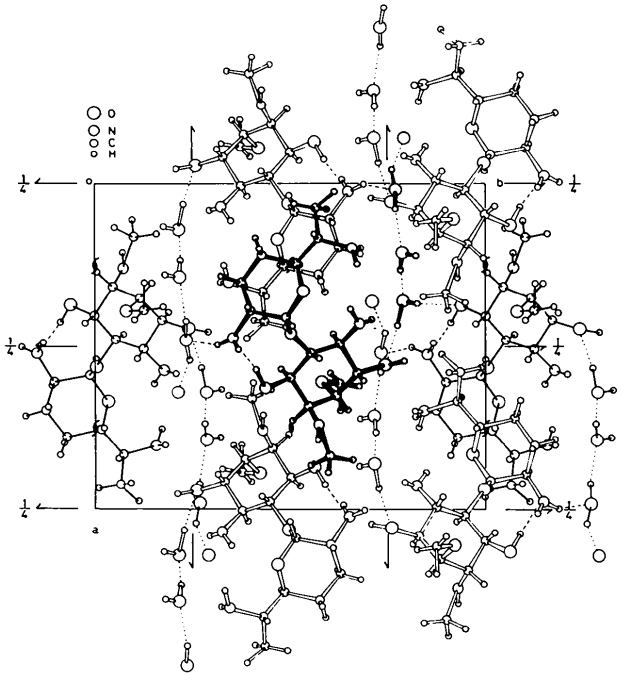

Fig. 3. The crystal structure projected along *c*.

2. The molecule consists of purpurosamine and 1,4-diaminocyclitol moieties, the linkage between them being α. The absolute configuration of the molecule can be deduced by correlating the X-ray structure with the absolute configuration of the 1,4-diaminocyclitol moiety, which had been determined by CD measurements. The two six-membered rings are in the chair conformation. The pyranose ring has the normal $^4C_1$ conformation. The ring torsion angles in purpurosamine and 1,4-diaminocyclitol moieties are between 53 and 57, and 50 and 60° respectively. The torsion angles of O(5′)–C(1′)–O(1)–C(6) and C(1)–C(6)–O(1)–C(1′) are 105 and 124° respectively. The bond lengths and angles in Fig. 2 show no unexpected variations. There is an intramolecular hydrogen bond between O(5)–H and N(2′); the O···N distance is 2·76 Å. The linking oxygen atom is not included in any hydrogen bonds. The crystal structure viewed along the *c* axis is shown in Fig. 3. The packing of the molecules in the crystal is stabilized by a system of hydrogen bonds. Hydrogen bonds N(2′)–H···O_w(3), O_w(1)–H···O(5), and O_w(1)–H···O(2) link the fortimicin B molecule to the water molecules. The N···O and two O···O distances are 2·96, 2·83, and 2·93 Å respectively. Four water molecules in an asymmetric unit are hydrogen bonded successively to each other to form a short row.

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