Stereochemistry of Carbohydrate Derivatives. Part IV.[1] Crystal Structure of 3,6-(Acetylepimino)-3,6-dideoxy-1,2-*O*-isopropylidene-β-L-idofuranose

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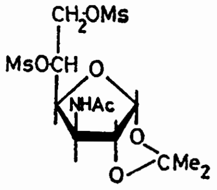
X-Ray crystallographic analysis, by a direct method, has confirmed the structure of title compound (2), the product of solvolysis of 3-acetamido-3-deoxy-1,2-*O*-isopropylidene-5,6-di-*O*-methylsulphonyl-α-D-glucofuranose (1) in 95% 2-methoxyethanol. Compound (2) crystallises in the monoclinic space group $P2_1$, with $Z = 2$ in a unit cell of dimensions $a = 12{\cdot}13$, $b = 9{\cdot}51$, $c = 5{\cdot}32$ Å, $\beta = 93{\cdot}74°$: the structure was refined to R 0·078 for 946 independent reflections.

In an earlier paper,[2] we reported that the solvolysis of 3-acetamido-3-deoxy-1,2-*O*-isopropylidene-5,6-di-*O*-methylsulphonyl-α-D-glucofuranose (1) in buffered 95% 2-methoxyethanol afforded a crystalline product, which was tentatively identified as 3,6-(acetylepimino)-3,6-dideoxy-1,2-*O*-isopropylidene-β-L-idofuranose (2). The L-*ido*-configuration was originally assigned on the assumption that the 3-acetamido-group participated in the solvolysis of the C-5 sulphonic ester group causing inversion of configuration at this centre. However, indisputable evidence for the structure (2) was not obtained. We now report details of an *X*-ray crystallographic analysis of the 3,6-epimine (2), which was undertaken concurrently with further chemical investigations.[3] This has demonstrated that the structure originally assigned is correct; it can be seen from Figure 1, for example, that the C-5 hydroxy-group has



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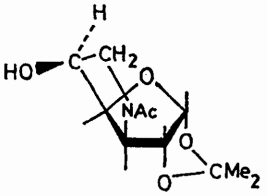

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[1] Part III, D. J. Watkin and T. A. Hamor, *J. Chem. Soc.* (*B*), 1971, 1692.

[2] J. S. Brimacombe and J. G. H. Bryan, *Carbohydrate Res.*, 1968, **6**, 423.

[3] J. S. Brimacombe and A. M. Mofti, *Carbohydrate Res.*, 1971, **18**, 157.

an *exo*-configuration with respect to the oxa-azabicyclo-[3,3,0]octane ring-system. Since only positions 5 and 6 of the dimethanesulphonate (1) are concerned in the solvolysis, the absolute configuration of the molecule is readily deduced from the stereochemistry at the other positions, which remains unaltered during the reaction.

EXPERIMENTAL AND RESULTS

3,6-(*Acetylepimino*)-3,6-*dideoxy*-1,2-O-*isopropylidene*-β-L-*idofuranose* (2).—This compound, m.p. 169—170 °C (from acetone–light petroleum, b.p. 60—80 °C), $[\alpha]_D$ −74° (*c* 1, $CHCl_3$), was obtained by solvolysis of 3-acetamido-3-deoxy-1,2-*O*-isopropylidene-5,6-di-*O*-methylsulphonyl-α-D-glucofuranose (1) as described previously.[2]

Crystal Data.—$C_{11}H_{17}NO_5$, $M = 243{\cdot}1$, Monoclinic, $a = 12{\cdot}131 \pm 0{\cdot}008$, $b = 9{\cdot}509 \pm 0{\cdot}008$, $c = 5{\cdot}321 \pm 0{\cdot}005$ Å, $\beta = 93{\cdot}74 \pm 0{\cdot}05°$, $U = 612{\cdot}4$ Å^3, $Z = 2$, $D_c = 1{\cdot}258$, $F(000) = 260$. Systematic absences, $0k0$ when k is odd, space group $P2_1$ or $P2_1/m$. The intensity statistics indicated a non-centrosymmetric space group, therefore $P2_1$ is the correct space group. Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å, for intensity measurements, Cu-K_α radiation $\lambda = 1{\cdot}5418$ Å, for unit-cell dimensions, μ(Mo-K_α) = 1·179 cm^{-1}.

Crystallographic Measurements.—Crystals were obtained as short prisms flattened on the (100) face and elongated along the *c* axis. The intensities were collected on a Hilger and Watts linear diffractometer from crystals mounted about the *b* and *c* axes. No corrections were made for absorption. The total number of reflections available for measurement with this instrument was 1470 and of these 946 gave values which were acceptable having regard to the background measurements.

Structure Analysis.—The intensities were placed on a common scale and converted to structure factors in the usual way; a scale factor and an overall isotropic temperature factor were then obtained from a Wilson plot. The F_0 values were then converted to E values by means of a computer programme supplied by Dr. P. Main of York University. Other programmes supplied by Dr. Main were used on the Elliott 4130 computer to determine the structure by direct methods. These were as follows: (i) 'Sigma 2,' which set up all phase relationships of the form $\phi_h \approx \phi_{h'} + \phi_{h-h'}$ where ϕ_h is the phase of F_h; (ii) 'Converge' which determined the signs of centric reflections, fixed the origin and enantiomorph by choosing the most suitable reflections. It then assigned phases of $\pm\pi/4$, $\pm 3\pi/4$ to a small number of other reflections of high E value and with a large number of triple relationships. Thus, there were multiple starting points for the next programme 'Fastan' which applied the tangent formula. (iii) 'Fastan' developed, by means of the tangent formula, sets of phases for the reflections with E values above a certain minimum value. These were assessed for reliability and output in a form suitable for carrying out a Fourier summation with the E values properly weighted. Figures of merit were assigned to each set of phases. In the case of compound (2), the reflections defining the origin were $20\bar{7}$, $10\bar{1}$, and $11\bar{1}$, which were assigned phases of 360°. Reflections 329 and 2,4,11 were given values of $\pm\pi/4$ and $\pm 3\pi/4$ in turn, thus producing eight sets of phases for 384 reflections with the largest values of E. The most useful figures of merit were as follows, 1·058, 1·029, 1·010, 0·986, 0·974, 0·898, 0·894, and 0·756 (this value is zero for random phases and should be >1·0 for a correct set). An E-map was computed with the set of phased E values having the highest figure of merit and contours were drawn at arbitrary intervals. Figure 2 shows a projection of the three-dimensional Fourier on the *ab* face of the unit cell. The positions of 14 peaks were accepted as representing the atomic positions of a ring system consisting of three five-membered rings and a COMe group. A structure-factor calculation was then carried out assuming all 14 atoms to be nitrogen and with an overall temperature factor (*B*) of 4·0. All the observed reflections were included and *R* was 0·295. A bond-length, bond-angle calculation was performed which gave reasonable values. An F_0 three-dimensional Fourier was now computed with all the F_0 values included and phases given by F_c values. The 14 atoms used for the F_c calculation appeared as well established peaks, but at the positions where the other three atoms were expected to appear the peak values were very much lower. At this point, the atomic positions of the

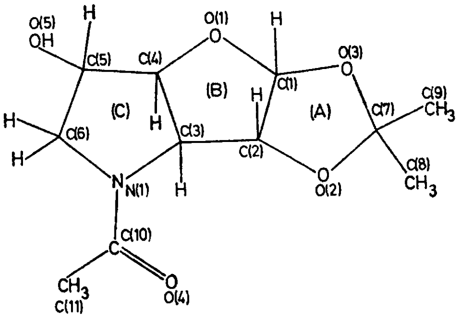

FIGURE 1 Drawing showing the stereochemistry of the epimine and the numbering system used

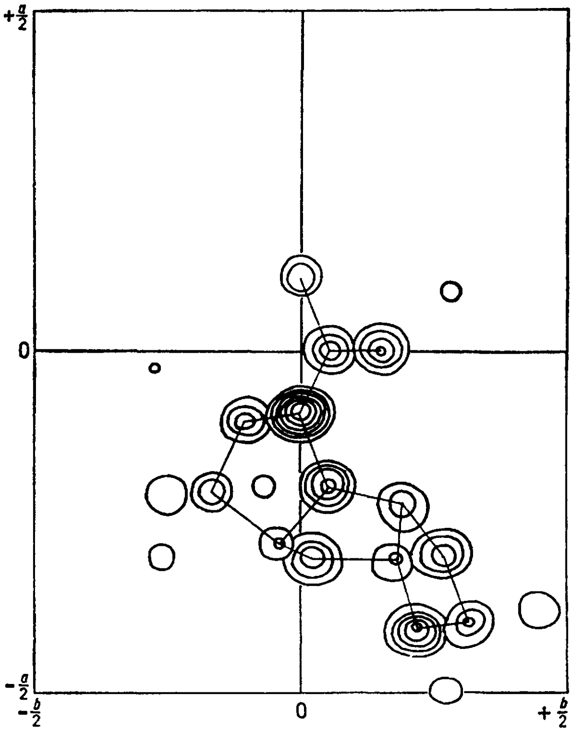

FIGURE 2 E-map projected on to the (001) plane; the linked atoms were accepted at this stage but the peak positions correspond to the mirror image of the true structure

14 well established peaks were named according to the chemical evidence, and two of the other peaks were accepted as being carbon and oxygen atoms, respectively. A structure-factor calculation with these atoms gave R 0·243.

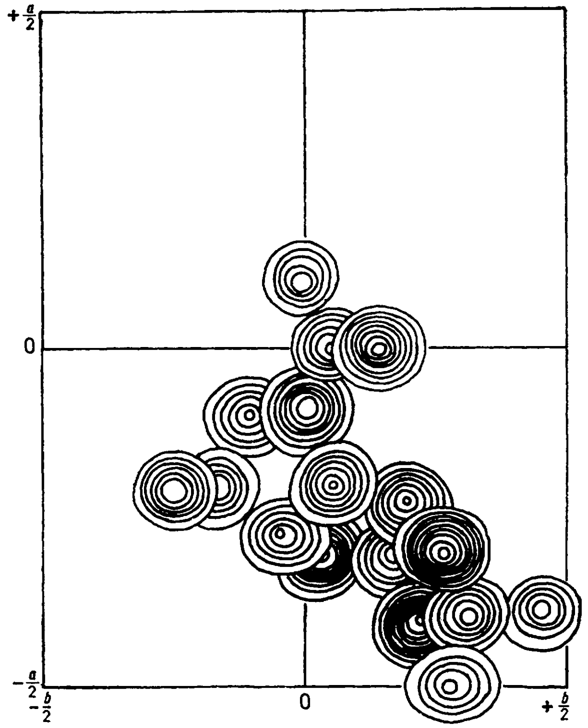

FIGURE 3 Third three-dimensional Fourier projected on to the (001) plane; the peak positions correspond to the mirror image of the true structure

A second, three-dimensional Fourier synthesis was computed and this gave the 14 peaks corresponding to the input co-ordinates and an extra peak, which was only slightly higher than in the first Fourier. However, it was now accepted and a third Fourier (Figure 3) was computed; all 17 peaks were now normal. This series shows the effect, in a non-centrosymmetric space group, of the close correlation between the input information and the phases output by the structure-factor calculation. A structure-factor calculation with all 17 atomic positions derived from the third Fourier and with a temperature factor (B) of 4·5 gave R 0·222.

Block-diagonal least-squares refinement of atomic co-ordinates and individual isotropic temperature factors reduced R to 0·194 in three cycles. Further isotropic refinement with assumed hydrogen co-ordinates reduced R to 0·149. The refinement was continued with anisotropic temperature parameters for carbon, nitrogen, and oxygen atoms, and fixed isotropic temperature parameters for hydrogen atoms to give the final R of 0·078. Difference Fourier syntheses were carried out in order to determine the hydrogen co-ordinates, but did not give satisfactory positions for the hydrogens of the methyl groups.

Table 1 gives the atomic co-ordinates and Table 2 the temperature parameters used in the final calculations of structure factors. The hydrogens of the methyl groups are not included in the Tables as their values are uncertain. In order to make a reasonable allowance for these hydrogen atoms, they were assumed to be situated as half-atoms at six equally spaced positions on a circle formed by rotating the methyl pyramid about the axis through the carbon atom. Table 3 gives the bond lengths and bond angles. Figure 4 shows a projection on to the ab face of the unit cell while Table 4 gives the intermolecular distances of $<$3·7 Å.

TABLE 1

Atomic co-ordinates with the estimated standard deviations in parentheses

	x/a	y/b	z/c
C(1)	−0·3023(6)	−0·1710(9)	−0·5235(12)
C(2)	−0·2262(5)	−0·1983(8)	−0·2854(12)
C(3)	−0·1945(5)	−0·0520(8)	−0·1895(12)
C(4)	−0·2780(6)	0·0445(9)	−0·3311(17)
C(5)	−0·2122(7)	0·1734(10)	−0·3884(22)
C(6)	−0·0987(7)	0·1146(10)	−0·4427(16)
C(7)	−0·3946(6)	−0·3111(8)	−0·2523(14)
C(8)	−0·3830(8)	−0·4639(11)	−0·3320(25)
C(9)	−0·4905(8)	−0·2872(15)	−0·0891(17)
C(10)	0·0063(5)	−0·0505(9)	−0·1573(14)
C(11)	0·1130(6)	0·0113(10)	−0·2428(19)
O(1)	−0·3060(4)	−0·0268(7)	−0·5619(10)
O(2)	−0·2946(4)	−0·2647(6)	−0·1157(9)
O(3)	−0·4070(4)	−0·2206(7)	−0·4596(10)
O(4)	0·0043(5)	−0·1420(8)	0·0060(14)
O(5)	−0·2076(5)	0·2589(8)	−0·1685(17)
N(1)	−0·0880(4)	−0·0047(7)	−0·2685(10)
H(1)[C(1)]	−0·2812	0·2255	−0·6835
H(2)[C(2)]	−0·1546	0·2562	−0·3097
H(3)[C(3)]	−0·1908	0·0500	0·0103
H(4)[C(4)]	−0·3509	−0·0618	−0·2418
H(5)[C(5)]	−0·2393	−0·2376	−0·5399
H(6)[C(6)]	−0·0391	−0·1885	−0·4098
H(7)[C(6)]	−0·1072	−0·0721	−0·6335
H(8)[O(5)]	−0·1261	−0·2986	−0·1040

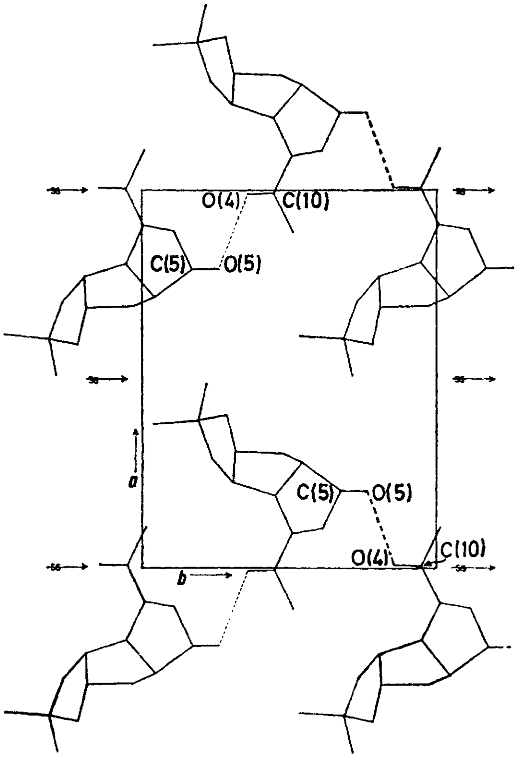

FIGURE 4 Projection of the unit cell on to the (001) plane; hydrogen bonds are shown as broken lines

The initial determination of the structure from the E-map gave the opposite absolute configuration according to

TABLE 2

Anisotropic temperature parameters * estimated standard deviations in parentheses

	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
C(1)	0·00814(56)	−0·00266(140)	−0·00120(197)	0·01273(109)	−0·00554(291)	0·02231(221)
C(2)	0·00470(39)	−0·00177(108)	0·00544(157)	0·00964(87)	0·00189(259)	0·02525(203)
C(3)	0·00519(42)	−0·00118(111)	−0·00079(172)	0·00984(88)	−0·00201(272)	0·02679(222)
C(4)	0·00402(44)	−0·00100(127)	−0·00724(237)	0·01180(98)	−0·00518(386)	0·06745(418)
C(5)	0·00839(64)	0·00347(152)	−0·01539(313)	0·00981(102)	−0·00498(462)	0·09015(540)
C(6)	0·00922(60)	−0·00636(148)	−0·00925(259)	0·01048(95)	0·00510(359)	0·05175(356)
C(7)	0·00743(54)	−0·00473(136)	−0·00345(208)	0·01271(108)	0·00092(310)	0·03138(261)
C(8)	0·00859(70)	−0·00499(182)	−0·01425(400)	0·01338(132)	−0·01034(599)	0·12571(781)
C(9)	0·00850(69)	−0·00811(254)	0·00957(288)	0·04098(282)	−0·01658(591)	0·04489(368)
C(10)	0·00423(42)	−0·00001(123)	0·00092(198)	0·01311(107)	0·00352(343)	0·04428(298)
C(11)	0·00508(48)	−0·00144(143)	0·00887(260)	0·01481(130)	0·00642(431)	0·07616(474)
O(1)	0·00922(38)	−0·00394(97)	−0·02076(145)	0·01140(65)	0·00139(239)	0·04575(202)
O(2)	0·00761(37)	−0·00664(96)	−0·00526(134)	0·01483(71)	0·00994(206)	0·02679(158)
O(3)	0·00880(39)	−0·00949(103)	−0·01221(144)	0·01787(82)	0·01237(240)	0·03521(178)
O(4)	0·00609(33)	0·00003(114)	−0·00894(171)	0·02254(92)	0·03254(308)	0·07511(277)
O(5)	0·00819(49)	0·00053(117)	−0·00387(273)	0·01503(82)	−0·06274(347)	0·16231(526)
N(1)	0·00510(35)	−0·00089(90)	0·00309(146)	0·00883(70)	0·00105(221)	0·03058(197)
H(1)[C(1)]	5·70 †					
H(2)[C(2)]	4·20					
H(3)[C(3)]	4·80					
H(4)[C(4)]	6·00					
H(5)[C(5)]	7·55					
H(6)[C(6)]	7·125					
H(7)[C(6)]	7·125					
H(8)[O(5)]	9·00					

* In the form: $T = \exp[-(h^2b_{11} + k^2b_{22} + l^2b_{33} + 2hkb_{12} + 2hlb_{13} + 2klb_{23})]$. † Hydrogen atoms were given a fixed isotropic temperature factor.

the known chemical derivation. Therefore, when the refinement was nearly complete, the model was changed to the correct configuration. Each model was refined and the final R was marginally lower for the correct configuration.

TABLE 3

Molecular dimensions

(*a*) Bond lengths (Å), with standard deviations in parentheses

C(1)–C(2)	1·548(9)	C(2)–O(2)	1·413(9)
C(2)–C(3)	1·517(10)	C(4)–O(1)	1·429(10)
C(3)–C(4)	1·523(10)	C(5)–O(5)	1·421(14)
C(4)–C(5)	1·512(13)	C(7)–O(2)	1·442(9)
C(5)–C(6)	1·527(13)	C(7)–O(3)	1·396(10)
C(7)–C(8)	1·520(14)	C(10)–O(4)	1·231(11)
C(7)–C(9)	1·513(13)	C(3)–N(1)	1·458(9)
C(10)–C(11)	1·518(11)	C(6)–N(1)	1·464(11)
C(1)–O(1)	1·389(10)	C(10)–N(1)	1·325(9)
C(1)–O(3)	1·416(10)		

(*b*) Bond angles (deg.)

C(1)–C(2)–C(3)	104·0	C(3)–N(1)–C(10)	121·9
C(2)–C(3)–C(4)	104·5	N(1)–C(10)–C(11)	118·0
C(3)–C(4)–C(5)	104·7	C(11)–C(10)–O(4)	122·7
C(4)–C(5)–C(6)	103·3	O(4)–C(10)–N(1)	119·3
C(5)–C(6)–N(1)	102·5	C(10)–N(1)–C(6)	125·0
C(6)–N(1)–C(3)	112·1	C(6)–C(5)–O(5)	112·0
N(1)–C(3)–C(4)	104·2	O(5)–C(5)–C(4)	106·2
C(3)–C(4)–O(1)	104·6	C(5)–C(4)–O(1)	107·4
C(4)–O(1)–C(1)	109·5	O(1)–C(1)–O(3)	110·2
O(1)–C(1)–C(2)	107·3	O(2)–C(2)–C(3)	110·2
C(1)–C(2)–O(2)	104·3	O(3)–C(7)–C(8)	111·4
C(2)–O(2)–C(7)	109·1	O(3)–C(7)–C(9)	108·3
O(2)–C(7)–O(3)	105·0	O(2)–C(7)–C(8)	110·2
C(7)–O(3)–C(1)	109·8	O(2)–C(7)–C(9)	109·0
O(3)–C(1)–C(2)	104·3	C(8)–C(7)–C(9)	112·6
C(2)–C(3)–N(1)	113·4		

DISCUSSION

The numbering system used (see Figure 1) is such that the carbon atoms of the parent hexose are numbered by the normal carbohydrate convention while the remaining atoms are numbered arbitrarily. Carbon–carbon and carbon–oxygen bond lengths are in good agreement with the values expected [4] in carbohydrate derivatives of this type and no individual bond length or angle deviates significantly from the expected value.

Several points of interest emerge from the crystallographic study of the epimine (2). In the course of examining the structure of (2) by n.m.r. spectroscopy two conformers were observed [2] in deuteriochloroform solution at ambient temperature as a consequence of restricted rotation about the *N*-acetyl bond.[5,6] However, only one of the two conformers is present in the crystals and it is significant that the amide carbonyl group of this conformer is involved in intermolecular hydrogen bonding with the C(5) hydroxy-group of the molecule related to it by the screw axis (Figure 4). Careful analysis of the n.m.r. spectrum of the epimine,[2]

TABLE 4

Intermolecular distances $<3{\cdot}7$ Å

C(8) ··· O(5^{I})	3·474	C(11) ··· O(5^{IV})	3·397
C(3) ··· O(1^{II})	3·688	C(10) ··· O(5^{IV})	3·420
O(2) ··· C(1^{II})	3·279	O(4) ··· C(5^{IV})	3·589
C(9) ··· O(3^{II})	3·506	O(4) ··· C(6^{IV})	3·419
C(11) ··· O(2^{III})	3·535	O(4) ··· O(5^{IV})	2·723 *

* Hydrogen bond.

Roman numerals as superscripts refer to the following equivalent positions relative to the reference molecule at x, y, z:

I	$x, 1 + y, z$	III	$-x, y - \frac{1}{2}, -z$
II	$x, y, 1 + z$	IV	$-x, \frac{1}{2} + y, -z$

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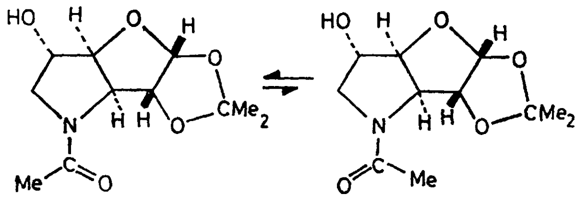
[4] G. A. Jeffrey and R. D. Rosenstein, *Adv. Carbohydrate Chem.*, 1964, **19**, 7; G. Strahs, *ibid.*, 1970, **25**, 53; M. Sundaralingam, *J. Amer Chem. Soc.*, 1965, **87**, 599.

[5] H. Paulsen and K. Todt, *Adv. Carbohydrate Chem.*, 1968, **23**, 115.

[6] C. H. Bushweller, J. W. O'Neil, M. H. Halford, and F. H. Bissett, *J. Amer. Chem. Soc.*, 1971, **93**, 1471.

particularly with regard to the magnetic anisotropy of the amide group, led us to conclude that the major conformer in solution at ambient temperature corresponded to that (3) present in the crystals. It was possible to support this conclusion by examining the n.m.r. spectrum (90 MHz) of the epimine at low temperatures. It has been shown that, in many instances, the crystalline form of a compound exists as a conformationally pure species [7] and that dissolution of the crystalline material at temperatures where equilibration to other conformers is very slow results in a solution of the pure conformer. Dissolution of the epimine in deuteriochloroform at *ca.* −50 °C and examination of the n.m.r. spectrum showed a singlet ascribable to the *N*-acetyl group at δ 2·05 p.p.m. indicating a solution of conformationally pure (3). Upon warming the sample to higher temperatures, a second singlet appeared downfield at δ 2·15 p.p.m. and increased in intensity demonstrating the equilibration (3) $\rightleftharpoons$ (4). Integration over these signals gave the ratio of conformers (3) to (4) present at ambient temperature as 2·5 : 1. It was also possible to follow the equilibration (3) $\rightleftharpoons$ (4) with time at −40 °C, since at this temperature the equilibration occurred at a convenient rate.



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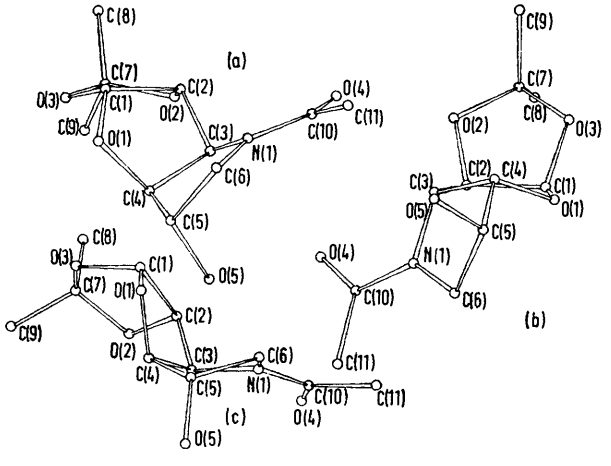


FIGURE 5 View of the molecule looking along the mean plane of (a) ring A, (b) ring B, and (c) ring C

The two *cis*-fused, five-membered rings of the trioxabicyclo[3,3,0]octane system (rings A and B) each adopt envelope-like conformations. The furanose ring (Figure 5b) has C(4) displaced by 0·459 Å in an *exo*-direction (with respect to the bicyclic system) from the mean plane through the other four atoms [Plane (B2) in Table 5] and the acetal carbon atom [C(7)] of the dioxolan ring is also displaced in an *exo*-direction by 0·38 Å [Plane (A2); see also Figure 5a]. The five-membered ring containing the nitrogen atom (ring C) likewise assumes an envelope conformation having C(5) displaced by 0·538 Å [Plane (C2) in Table 5] in an *endo*-direction with respect to the oxa-azabicyclo[3,3,0]octane ring-system. These values are comparable with out-of-plane displacements commonly found for furanose rings adopting envelope conformations, although in many other instances the out-of-plane atom of the furanose ring has been either C(2) or C(3).[4,8] The trioxabicyclo[3,3,0]octane ring-system of 6-azido-5,6-dideoxy-5-iodo-1,2-*O*-isopropylidene-β-L-idofuranose (5) assumes a 'twin-envelope' conformation having C(4) displaced by 0·70 Å in an *endo*-direction and the acetal carbon atom [C(7)]

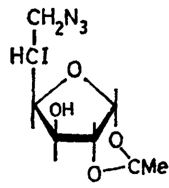
TABLE 5

Equations of mean planes in the form $lX + mY + nZ = d$ where X, Y, and Z are co-ordinates in Å relative to the orthogonal axes. The deviations of the atoms from the plane (Å) are given in square brackets. Atoms marked * are not included in the derivation of the plane

Atoms	*l*	*m*	*n*	*d*
Plane (A1)	0·344	−0·841	−0·417	1·275
[C(1) 0·061, C(2) 0·033, O(2) −0·113, O(3) −0·137, C(7) 0·156]				
Plane (A2)	0·245	−0·898	−0·365	1·613
[C(1) 0·015, C(2) −0·015, O(2) 0·010, O(3) −0·010, C(7)* 0·382]				
Plane (B1)	−0·844	−0·113	0·524	1·603
[C(1) 0·067, C(2) 0·047, C(3) −0·135, O(1) −0·166, C(4) 0·187]				
Plane (B2)	−0·841	0·035	0·538	1·364
[C(1) 0·020, C(2) −0·018, O(1) −0·013, C(3) 0·012, C(4)* 0·459]				
Plane (C1)	−0·088	−0·505	−0·859	1·388
[C(3) −0·076, C(4) 0·185, N(1) −0·056, C(6) 0·169. C(5) −0·22]				
Plane (C2)	−0·021	−0·637	−0·770	1·141
[C(3) −0·005, C(4) 0·003, N(1) 0·006, C(6) −0·003, C(5)* −0·538]				

Angles between planes:

(A1)–(B1)	114° 27′
(A2)–(B2)	115° 44′
(B1)–(C1)	108° 37′
(B2)–(C2)	114° 46′



(5)

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[7] F. R. Jensen and C. H. Bushweller, *J. Amer. Chem. Soc.*, 1969, **91**, 3223; C. H. Bushweller, J. Golini, G. U. Rao, and J. W. O'Neill, *ibid.*, 1970, **92**, 3055; H. S. Gutowsky, J. Jonas, and T. H. Siddall, *ibid.*, 1967, **89**, 4300; M. Oki and H. Iwamura, *Tetrahedron*, 1968, **24**, 2377.

[8] See, *e.g.*, P. Groth and H. Hammer, *Acta Chem. Scand.*, 1968, **22**, 2059; W. Saenger and K. H. Scheit, *Angew. Chem. Internat. Edn.*, 1969, **8**, 139; D. C. Rohrer and M. Sundaralingam, *J. Amer. Chem. Soc.*, 1970, **92**, 4956; S. T. Rao and M. Sundaralingam, *ibid.*, p. 4963.

displaced by 0·45 Å in an *exo*-direction.[9] Thus, *cis*-fusion of a third five-membered ring as in the epimine (2) has reversed the direction of displacement of C(4).

The conformation of the epimine (2) in the crystalline form differs from that suggested for a structurally related compound, 3,6-anhydro-1,2-*O*-isopropylidene-α-D-glucofuranose, in solution. A study[10] of the latter compound by n.m.r. spectroscopy suggested that the furanose ring adopts a twist conformation in which C(2) is below and C(3) is above the plane defined by the other three atoms, whereas the anhydro-ring adopts an envelope shape with C(4) displaced above the plane defined by the other atoms. Thus, the two rings adopt the alternative shapes.

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[9] J. S. Brimacombe, J. G. H. Bryan, and T. A. Hamor, *J. Chem. Soc.* (*B*), 1970, 514.

[10] R. J. Abraham, L. D. Hall, L. Hough, and K. A. McLauchlan, *J. Chem. Soc.*, 1962, 3699.