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The Crystal and Molecular Structure of a Heterocyclic Dithiepane Derivative of an Octopyranose: $C_{17}H_{27}N_3O_5S_2$

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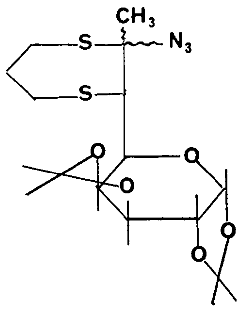
The crystal structure of 7-azido-8-deoxy-1,2,3,4-di-*O*-isopropylidene-6,7-*S*,*S*-trimethylene-6,7-dithio-α-D-erythro-D-*galacto*-octopyranose, $C_{17}H_{27}N_3O_5S_2$, has been determined. The crystals are orthorhombic, $P2_12_12_1$, with $a=9{\cdot}599$, $b=10{\cdot}835$, $c=19{\cdot}559$ Å, $Z=4$. The structure was solved by direct methods and refined by the full-matrix least-squares technique to an R value of 0·046 for 1929 independent reflexions collected with an automatic four-circle diffractometer. The configuration of carbon C(7) is *S*. The pyranose ring is boat shaped, the dithiepane ring is in a twist-chair conformation, and the conformations of the two dioxolane rings are envelope and twist-chair.

Introduction

Several new powerful antibiotics (lincomycin, aldgamicyn E, *etc.*) contain branched and extended-chain carbohydrate derivatives.

To synthesize the octose derivatives from the more abundant hexoses, M. Gero and his co-workers were led to prepare dithiane derivatives of octopyranoses (Sepulchre, Gateau-Olesker, Lukacs, Vass & Gero, 1972). One of these compounds was transformed into a new class of carbohydrate dithiepane derivative:



The configuration at C(7) could not be deduced from chemical considerations and so the crystal structure determination was undertaken. It was thought that the X-ray study could also produce some interesting results concerning the conformation of the still unknown dithiepane cycle, and that of the pyranose ring, substituted by the two isopropylidene groups. The preliminary results have been reported in a short note (Gateau-Olesker, Gero, Pascard-Billy, Riche, Sepulchre, Vass & Hughes, 1974).

Experimental

Crystal data

$C_{17}H_{27}N_3O_5S_2$, F.W. 417. Orthorhombic. Space group: $P2_12_12_1$; $a=9{\cdot}599(5)$, $b=10{\cdot}835(5)$, $c=19{\cdot}559(9)$ Å, $V=2034{\cdot}2$ Å^3, $D_x=1{\cdot}362$ g cm^{-3}, $Z=4$. Crystal dimensions: $0{\cdot}3\times0{\cdot}35\times0{\cdot}4$ mm.

Intensity recording

Automatic four-circle Philips PW1100 diffractometer, graphite monochromator, copper radiation ($\lambda=1{\cdot}5418$ Å), ω scan to $2\theta=60°$, ω–2θ scan for $60°<$

$2\theta < 136^\circ$, scan speed: 0·03° s^{-1}, scan width: 1·20°+ 0·16° tg θ. Two background measurements were made for 10 s at each end of the scan. Number of independent reflexions: 2148. Number of observed reflexions, $I > 2\sigma(I)$: 1929. For crystal decomposition (10%), a correction was made empirically with the aid of three standard reflexions, monitored every hour.

Structure determination

The structure was solved with the symbolic addition procedure and the phase function (Riche, 1973).

The phase function calculated with five symbols (three general and two equatorial phases) gave four maxima. For the best tangent refinement which coincided with the first maximum, the corresponding E map showed all the atoms, except the last two nitrogen atoms. These appeared on a subsequent E map after recycling over the known atoms.

The hydrogen atoms were located on difference syntheses.

Refinement

Full-matrix least-squares refinement was made with a local version of *ORFLS* (Busing, Martin & Levy, 1962). Atomic scattering factors for carbon, nitrogen and oxygen were taken from Doyle & Turner (1968) and the atomic form factor for hydrogen was that given by Stewart, Davidson & Simpson (1965).

All the heavy atoms were refined anisotropically while the hydrogen atoms were refined with the corresponding isotropic B values of the bonded carbon atoms. The final R value was 0·046 (0·051 including the non-zero reflexions).

Table 2. *Hydrogen atoms: coordinates* ($\times 10^3$) *and isotropic temperature factors* (Å^2)

	x	y	z	B
H(1)	915	417	639	3·38
H(2)	711	392	711	3·64
H(3)	540	569	696	3·54
H(4)	602	722	625	2·85
H(5)	638	563	549	2·60
H(6)	876	706	507	2·62
H(8*A*)	773	459	454	3·27
H(8*B*)	794	485	374	3·27
H(8*C*)	918	534	424	3·27
H(9*A*)	583	847	378	4·30
H(9*B*)	667	894	316	4·30
H(10*A*)	663	1044	400	4·41
H(10*B*)	808	1023	376	4·41
H(11*A*)	796	1033	497	3·78
H(11*B*)	881	922	468	3·78
H(13*A*)	594	189	502	6·00
H(13*B*)	552	336	501	6·00
H(13*C*)	447	240	543	6·00
H(14*A*)	692	178	684	5·38
H(14*B*)	692	105	613	5·38
H(14*C*)	561	139	651	5·38
H(16*A*)	655	815	807	4·93
H(16*B*)	669	914	746	4·93
H(16*C*)	572	820	728	4·93
H(17*A*)	889	740	821	5·39
H(17*B*)	926	833	764	5·39
H(17*C*)	991	685	748	5·39

Table 1. *Heavy atoms: atomic coordinates and anisotropic thermal parameters in the form* $\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right]$

The values are $\times 10^4$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B
S(6)	6838 (1)	8469 (1)	5214 (1)	117 (2)	64 (1)	18 (0)	12 (1)	3 (1)	1 (0)	3·4
S(7)	7899 (1)	7384 (1)	3621 (1)	113 (2)	109 (1)	15 (0)	−5 (2)	7 (1)	4 (1)	3·9
C(1)	8176 (5)	4238 (4)	6176 (2)	81 (6)	82 (5)	22 (1)	10 (5)	1 (3)	4 (2)	3·4
C(2)	6973 (6)	4379 (4)	6682 (2)	131 (7)	78 (5)	16 (1)	−15 (6)	1 (3)	6 (2)	3·7
C(3)	6499 (6)	5677 (4)	6857 (2)	119 (7)	76 (5)	16 (1)	−22 (5)	6 (3)	−4 (2)	3·5
C(4)	6836 (5)	6645 (4)	6309 (2)	100 (6)	65 (4)	15 (1)	−1 (5)	2 (2)	−2 (2)	3·0
C(5)	7249 (5)	6070 (4)	5640 (2)	71 (5)	71 (4)	14 (1)	8 (4)	2 (2)	−1 (2)	2·7
C(6)	7668 (5)	6974 (4)	5064 (2)	76 (5)	66 (4)	14 (1)	−6 (4)	1 (2)	1 (2)	2·7
C(7)	7327 (5)	6412 (4)	4346 (2)	84 (6)	80 (5)	13 (1)	−5 (5)	−1 (2)	1 (2)	2·9
C(8)	8086 (6)	5208 (5)	4227 (2)	123 (8)	90 (5)	19 (1)	14 (6)	3 (3)	−8 (2)	3·9
C(9)	6779 (6)	8732 (5)	3631 (2)	132 (8)	119 (6)	20 (1)	12 (6)	−2 (3)	16 (2)	4·5
C(10)	7339 (6)	9815 (5)	4032 (3)	132 (8)	89 (5)	28 (2)	−4 (6)	−1 (3)	17 (2)	4·5
C(11)	7915 (6)	9489 (4)	4715 (2)	113 (7)	85 (5)	26 (1)	−4 (6)	−2 (3)	7 (2)	4·1
C(12)	6404 (6)	2809 (5)	5951 (3)	119 (8)	66 (5)	31 (2)	−5 (5)	5 (3)	−9 (2)	4·1
C(13)	5530 (7)	2607 (5)	5321 (3)	213 (11)	107 (6)	37 (2)	−1 (8)	−23 (4)	−16 (3)	6·2
C(14)	6525 (7)	1661 (5)	6397 (3)	194 (11)	68 (5)	43 (2)	−5 (7)	8 (4)	4 (3)	5·6
C(15)	7729 (6)	7306 (5)	7316 (2)	167 (9)	87 (5)	14 (1)	−15 (6)	1 (3)	−7 (2)	4·1
C(16)	6647 (8)	8241 (5)	7516 (3)	211 (12)	135 (7)	31 (2)	−9 (9)	13 (4)	−32 (3)	6·3
C(17)	9110 (7)	7485 (6)	7681 (3)	193 (10)	132 (7)	24 (1)	−23 (8)	−16 (3)	−12 (3)	5·6
O	8436 (3)	5273 (3)	5758 (1)	77 (4)	71 (3)	19 (1)	13 (3)	4 (2)	4 (1)	3·0
O(1)	7766 (4)	3222 (3)	5765 (2)	126 (2)	79 (3)	36 (1)	5 (4)	22 (2)	−12 (2)	4·6
O(2)	5826 (4)	3803 (3)	6326 (2)	107 (5)	68 (5)	29 (1)	−12 (3)	6 (2)	−7 (2)	3·9
O(3)	7271 (5)	6075 (3)	7448 (1)	254 (8)	101 (4)	13 (1)	−52 (5)	−5 (2)	2 (1)	5·3
O(4)	7982 (4)	7326 (3)	6598 (1)	114 (5)	82 (3)	14 (1)	−25 (4)	1 (2)	−2 (1)	3·4
N(1)	5784 (4)	6292 (4)	4345 (2)	81 (5)	105 (5)	17 (1)	−9 (4)	−2 (2)	−10 (2)	3·5
N(2)	5303 (5)	5659 (4)	3874 (2)	101 (6)	92 (4)	25 (1)	0 (5)	−2 (2)	−4 (2)	4·0
N(3)	4733 (6)	5109 (5)	3470 (3)	169 (9)	154 (7)	48 (2)	−7 (7)	−28 (4)	−39 (3)	6·9

The positional coordinates and thermal parameters of the heavy atoms are listed in Table 1; the coordinates of the hydrogen atoms and their *B* values are given in Table 2.*

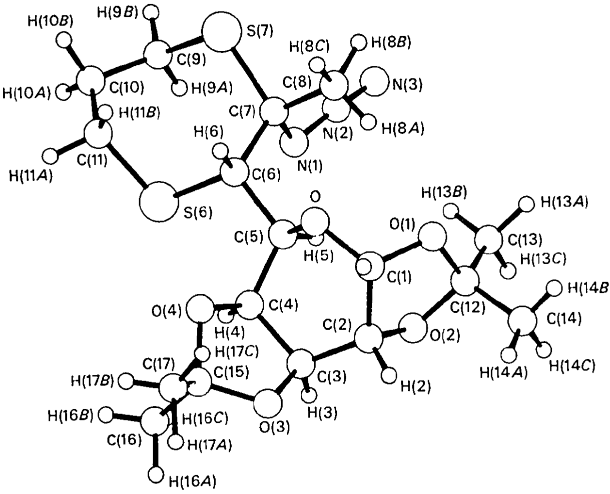


Fig. 1. *ORTEP* drawing of the molecule.

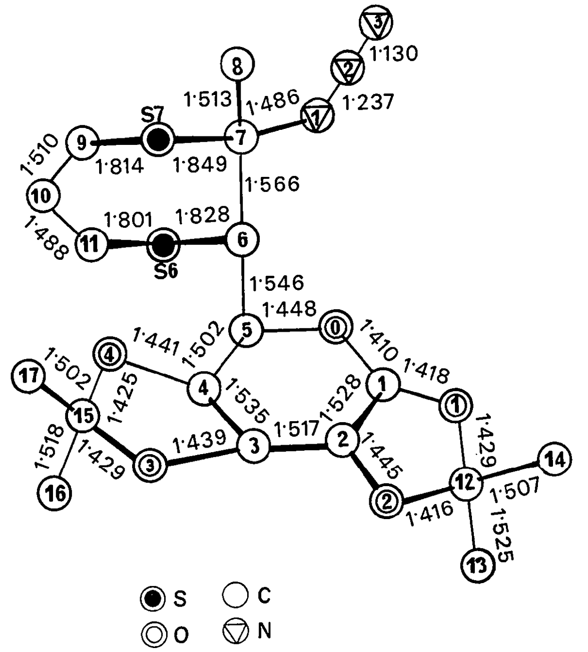


Fig. 2. Bond distances. $\bar{\sigma}$(S–C) = 0·005 Å, $\bar{\sigma}$(C–O) = 0·005 to 0·006 Å, $\bar{\sigma}$(C–C) = 0·006 to 0·009 Å, $\bar{\sigma}$(C–N) and $\bar{\sigma}$(N–N): 0·006 Å.

Discussion of the structure

The overall shape of the molecule is illustrated in Fig. 1. It appears as a galactose ring, with the two isopropylidene rings *trans*. The configuration of C(7) is *S*, and the azido group, bonded to C(7) is *trans* with respect to H(6).

The bond lengths are given in Fig. 2, the valency angles in Fig. 3, and the endocyclic torsion angles in Fig. 4. The C–H distances are listed in Table 3.

Table 3. C–H *distances* (Å)

C(1)—H(1)	1·03 (3)	C(13)-H(13*A*)	1·05 (5)
C(2)—H(2)	0·98 (4)	-H(13*B*)	1·01 (5)
C(3)—H(3)	1·07 (4)	-H(13*C*)	1·06 (5)
C(4)—H(4)	1·00 (4)	C(14)-H(14*A*)	0·96 (4)
C(5)—H(5)	1·01 (4)	-H(14*B*)	0·92 (4)
C(6)—H(6)	1·05 (4)	-H(14*C*)	0·95 (5)
C(8)—H(8*A*)	0·97 (4)	C(16)-H(16*A*)	1·09 (4)
-H(8*B*)	1·04 (3)	-H(16*B*)	0·98 (4)
—H(8*C*)	1·06 (4)	-H(16*C*)	1·01 (4)
C(9)—H(9*A*)	1·00 (4)	C(17)-H(17*A*)	1·05 (4)
—H(9*B*)	0·96 (4)	-H(17*B*)	0·92 (4)
C(10)-H(10*A*)	0·97 (4)	-H(17*C*)	1·11 (4)
-H(10*B*)	1·00 (4)		
C(11)-H(11*A*)	1·04 (4)		
-H(11*B*)	0·91 (4)		

(*a*) *The pyranose ring and the carbohydrate chain*

This cycle is boat shaped, puckered along the C(2)–C(5) line, with a puckering angle of 45·9°. This boat shape is distorted towards a twist-boat conformation. The flattening of the boat is due to the strain of the bonding of the two isopropylidene rings (see the mean planes in Table 4). As can be seen from Table 5, the bond lengths agree well with similar bonds in comparable rings. The bonds where the five- and six-membered rings are fused are slightly longer than average.

C(5)–C(6) is noticeably longer than usual, the close approach of S(6) and O(4) (3·173 Å) being probably responsible for the lengthening of this bond. The same effect appears in the length of C(6)–C(7) (1·566 Å), the S(6)–S(7) distance being 3·482 Å.

C(7)–C(8) has the length usually found for carbohydrate-chain terminal groups.

The C–O distances are in perfect agreement with those observed in pyranose rings: again there is a marked difference between the C(5)–O and C(1)–O lengths, the former being nearly 8σ longer.

The endocyclic angles are larger than the tetrahedral value. The smallest ring angle involves C(5). The angle C(4)–C(5)–C(6) is consistently larger (116·1) than O–C(5)–C(6) (106·8°), which is quite common in sugar rings: 112·0° against 106·8° (reference 2, Table 5), 115·0° against 106·3° in α-galactose (reference 1, Table 5).

(*b*) *Isopropylidene cycles*

In Table 6, the bond lengths and valency angles obtained in this investigation are compared with the

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* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31062 (14 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

values for isopropylidene rings bonded to various sugar rings, and it has been possible to average those very similar values.

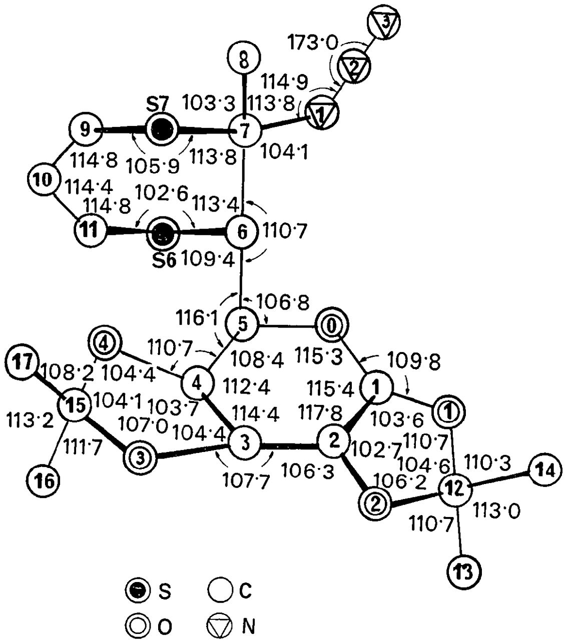


Fig. 3. Bond angles. C(17)–C(15)–O(3) 107·6, C(16)–C(15)–O(4) 111·3, C(13)–C(12)–O(1) 108·4, C(14)–C(12)–O(2) 109·3, S(7)–C(7)–N(1) 110·2, C(6)–C(7)–C(8) 111·8°. $\bar{\sigma}$(C–S–C) = 0·2°, $\bar{\sigma}$(C–C–O) = 0·3 to 0·4°, $\bar{\sigma}$(N–N–N) = 0·5°, $\bar{\sigma}$(C–O–C) = 0·3 to 0·4°.

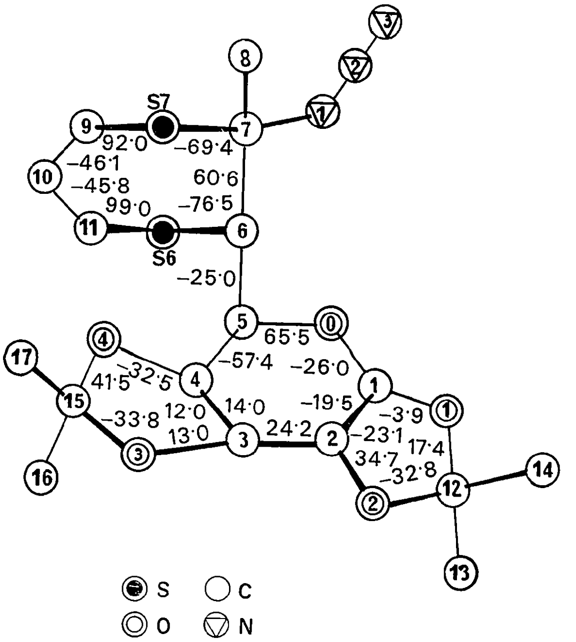


Fig. 4. Endocyclic torsion angles.

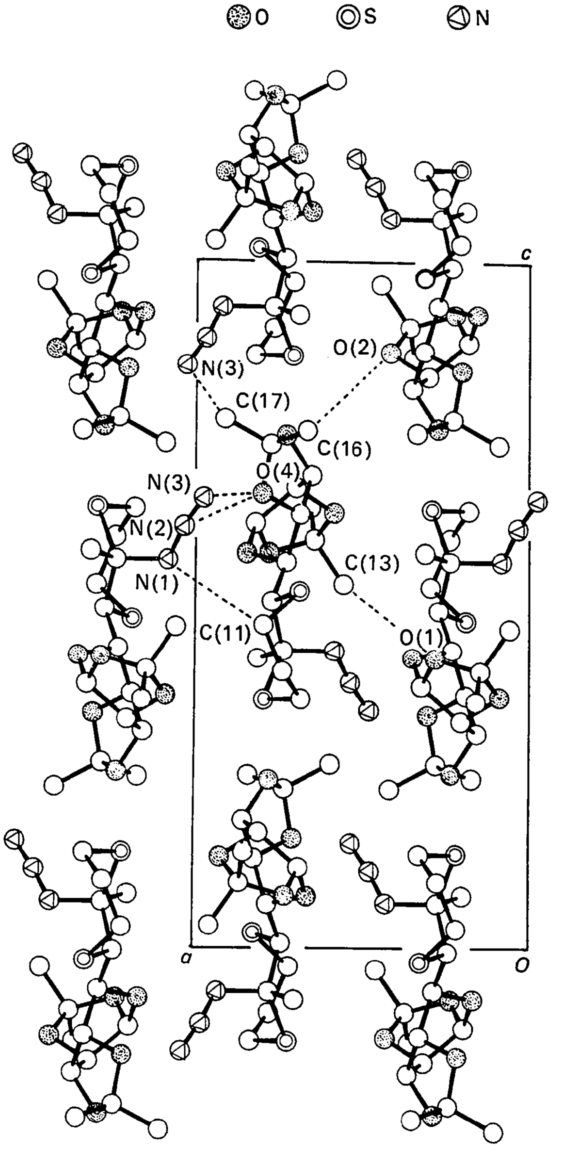


Fig. 5. *ORTEP* view of the unit cell projected along **b**. .... shortest intermolecular distances.

The best four-atom plane of ring *B* ($\bar{\sigma}$ = 0·02) is given in Table 4, with O(2) displaced 0·49 Å out of the plane. If we consider the C(12)–O(1)–C(1) plane, the deviation of C(2) is −0·10 Å whereas that of O(2) is +0·41 Å. Thus ring *B* has a slightly distorted envelope conformation.

As for the dioxolane ring C, the O(3)–C(3)–C(4) plane shows that C(15) and O(4) are symmetrically displaced out of the plane. This ring has a twist-chair shape. There are two best four-atom planes: plane VI, with C(15) out of the plane, whose orientation draws C(17) into the plane (0·02 Å), and plane VII with O(4) out of the plane.

(*c*) *Dithiepane ring*

The dithiepane ring adopts a regular twist-chair conformation. The sulphur atoms are 3·482 Å apart.

The S–C bond lengths are of the same order as those observed in dithiane rings (Spek, 1973; Kalff & Romers, 1965). The valency angles of S(6) and S(7), 102·0° and 105·9° respectively, are larger than the average (100°) in dithiane.

The C(9)–C(10), C(10)–C(11) distances and the mean C–C–C angle value of 114·3° agree well with the values encountered in seven-membered carbon rings.

(*d*) *Particular configurations*

The dihedral angle H(5)–C(5)–C(6)–H(6) is −149·2°. The disposition around the C(6)–C(7) bond is perfectly staggered. As for the azido group, its direction makes a dihedral angle of −167·4° with the octose chain C(6)–C(7).

Molecular packing

The following short intermolecular contacts are shown as dotted lines on the projection in Fig. 5.

O(4) $(x,y,z)\cdots$N(2)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	3·253 Å
O(4) $(x,y,z)\cdots$N(3)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	3·250
C(11) $(x,y,z)\cdots$N(1)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	3·419
C(13) $(x,y,z)\cdots$O(1)	$(-\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	3·516
C(16) $(x,y,z)\cdots$O(2)	$(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$	3·337
C(17) $(x,y,z)\cdots$N(3)	$(\frac{3}{2}-x, 1-y, \frac{1}{2}+z)$	3·394

Table 4. *Mean planes*

The asterisks refer to the atoms defining the plane.

I $-0{\cdot}6350x-0{\cdot}3571y-0{\cdot}6850z+14{\cdot}8972=0$
II $-0{\cdot}7453x-0{\cdot}3104y-0{\cdot}5901z+14{\cdot}9332=0$
III $-0{\cdot}4002x+0{\cdot}6290y-0{\cdot}6665z+8{\cdot}3034=0$
IV $-0{\cdot}4239x+0{\cdot}6476y-0{\cdot}6332z+8{\cdot}0251=0$
V $0{\cdot}7829x-0{\cdot}5460y-0{\cdot}2982z+2{\cdot}4734=0$
VI $0{\cdot}6853x-0{\cdot}6653y-0{\cdot}2962z+3{\cdot}8758=0$
VII $0{\cdot}8619x-0{\cdot}4258y-0{\cdot}2755z+0{\cdot}8540=0$

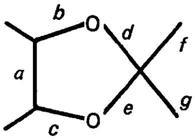
	Pyranose		Isopropyl *B*		Isopropyl *C*			Dithiepane
	I	II	III	IV	V	VI	VII	VIII
S(6)								−1·17
S(7)								1·18
O	*0	*−0·02	+1·15	+1·16				
C(1)	*0	*−0·04	*0	*0·02				
C(2)	*0	+0·27	−0·10	*−0·01				
C(3)	−0·45	*−0·03			*0	*0·09	*−0·08	
C(4)	−0·29	*+0·03			*0	*−0·07	*+0·04	
C(5)	0·57	+0·70			+1·04	+1·00		−0·80
C(6)		+0·76						−0·11
C(7)								+0·24
C(8)								+1·18
C(9)								*0
C(10)								*0
C(11)								*0
C(12)			*0	*0·02				
C(13)			+1·02	+1·01				
C(14)			−1·41	−1·38				
C(15)					−0·31	−0·54	*−0·07	
C(16)					−1·79	−2·04	−1·50	
C(17)					+0·41	+0·02	+0·80	
O(1)			*0	*−0·01				
O(2)			+0·41	+0·49				
O(3)					*0	*−0·03	*+0·05	
O(4)					+0·29	*+0·02	+0·52	
N(1)								
N(2)								
N(3)								

Table 5. *Pyranose ring*: *comparison of equivalent bond lengths in different rings*

Ref.	C(1)–C(2)	C(2)–C(3)	C(3)–C(4)	C(4)–C(5)	C(5)–C(6)	C(5)–O	C(1)–O	σ
1	1·504	1·507	1·495	1·525	1·491	1·449	1·407	0·008
2	1·517	1·523	1·533	1·536	1·514	1·448	1·427	0·005
3	1·518	1·519	1·524	1·511	–	1·422	1·409	0·004
4	1·527	1·503	1·510	1·494	–	1·426	1·409	0·004
5	1·528	1·510	1·526	1·519	–	1·425	1·426	0·004
6	1·528	1·517	1·535	1·502	1·546	1·448	1·410	0·006

(1) α-Galactose (Cook & Bugg, 1973). (2) 4-*O*-β-D-Galactopyranosyl-α-D-glucopyranose (Fries, Rao & Sundaralingam, 1971). (3) 1,2,3,4-Tetra-*O*-acetyl-α-D-arabinopyranose (James & Stevens, 1974). (4) Methyl-2,3,4-tri-*O*-acetyl-α-D-xylopyranose (James & Stevens, 1974). (5) 1,2,4,5-Di-*O*-isopropylidene-β-D-fructopyranose (Takagi, Shiono & Rosenstein, 1973). (6) This investigation.

Table 6. *Isopropylidene rings fused to sugar rings*: *comparison of bond lengths and angles*



Ref.	*a*	*b*	*c*	*d*	*e*	*f*	*g*	σ
1	1·528	1·418	1·445	1·429	1·429	1·507	1·525	6
	1·535	1·439	1·441	1·416	1·425	1·502	1·518	6
2	1·527	1·401	1·409	1·441	1·420	1·490	1·508	3
3	1·528	1·400	1·413	1·428	1·434	1·490	1·534	4
	1·526	1·441	1·423	1·441	1·433	1·507	1·512	4
4	1·552	1·426	1·429	1·437	1·405	1·510	1·516	3
	1·536	1·421	1·416	1·428	1·421	1·488	1·510	3
Mean	1·533	1·421	1·425	1·431	1·424	1·499	1·518	
Mean (dev)	(0·008)	1·425 (0·012)				1·509 (0·013)		

Ref.	*ab*	*ac*	*bd*	*ce*	*de*	*gf*	*gd*	*gf*	*fd*	*fe*	σ
1	103·6	102·7	110·7	106·2	104·6	113·0	110·3	110·7	108·4	109·3	0·4
	104·4	103·7	107·0	104·4	104·1	113·3	111·7	108·2	107·6	111·3	0·4
2	104·0	103·5	110·0	106·7	104·2	112·4	109·5	109·3	111·1	110·0	0·2
3	105·0	104·7	106·4	108·6	105·4	113·3	109·1	108·5	110·9	109·2	0·3
	102·7	102·1	109·1	107·0	105·3	113·6	109·7	109·2	108·7	109·9	0·3
4	104·7	102·1	107·2	107·4	104·7	113·1	109·0	108·4	109·6	111·6	0·2
	103·9	103·5	110·4	108·3	105·4	113·2	110·3	108·3	109·5	109·8	0·2
Mean	104·0	103·4	108·7	106·9	104·8	113·1	109·9	108·9	109·4	110·1	
Mean (dev)	103·7 (0·9)		107·8 (1·7)		(0·5)	(0·3)	109·6 (1·0)				

(1) This investigation. (2) 4,5-Di-*O*-acetyl-1,2-*O*-isopropylidene-3-*O*-methyl-α-D-guloseptanose (McConnell & Stevens, 1973). (3) 1,2,4,5-Di-*O*-isopropylidene-β-D-fructopyranose (Takagi, Shiono & Rosenstein, 1973). (4) Methyl-2,3,4,5-di-*O*-isopropylidene-α-D-*allo*-septanoside (Craig, Stephenson & Stevens, 1974).

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