The authors are grateful to Professors J. Toussaint and H. Brasseur for their continuous interest in this work.

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The Crystal Structures of Allitol and D-Iditol

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(*Received* 29 *June* 1971)

The crystal structures of two hexitols, allitol and D-iditol, $C_6H_{14}O_6$, have been determined with Cu $K\alpha$ radiation, using direct methods and the tangent formula for phase determination. The crystal data are: for allitol, $P2_1/c$ with $a=4{\cdot}708$ (3), $b=13{\cdot}408$ (8), $c=6{\cdot}616$ (4) Å; $\beta=100{\cdot}1°$; $Z=2$; $D_m=1{\cdot}482$, $D_x=1{\cdot}472$ g.cm^{-3}; for D-iditol, $P2_1$ with $a=8{\cdot}124$ (2), $b=8{\cdot}386$ (3), $c=5{\cdot}870$ (3) Å; $\beta=93{\cdot}82°$, $Z=2$; $D_m=1{\cdot}516$, $D_x=1{\cdot}510$ g.cm^{-3}. Both molecules have bent chain conformations as predicted from previous studies of alditol structures. That of allitol has a crystallographic center of symmetry. The bond lengths and angles are normal, with mean values over both structures of C–C = 1·523, C–O = 1·422 Å, C–C–C = 113·3, C–C–O = 109·8°. All hydrogen bonding is intermolecular. That of allitol is normal with each hydroxyl acting as donor and acceptor to form infinite chains. That of D-iditol is unusual for the alditol series in that the hydrogen bonding consists of infinite chains with branched chains. This requires that one hydroxyl is a donor only and that another is a double acceptor as well as a donor.

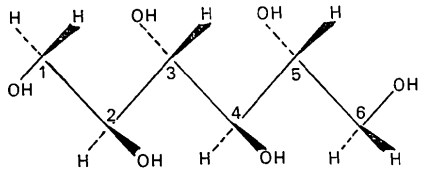
Introduction

These two structure determinations are part of a study of the pentitols and hexitols which has included D,L-arabinitol (Hunter & Rosenstein, 1968), ribitol (Kim, Jeffrey & Rosenstein, 1969), xylitol (Kim & Jeffrey, 1969), D-mannitol (Berman, Jeffrey & Rosenstein, 1968; Kim, Jeffrey & Rosenstein, 1968) galactitol (Berman & Rosenstein, 1968), and D-glucitol (Park, Jeffrey & Hamilton, 1971).

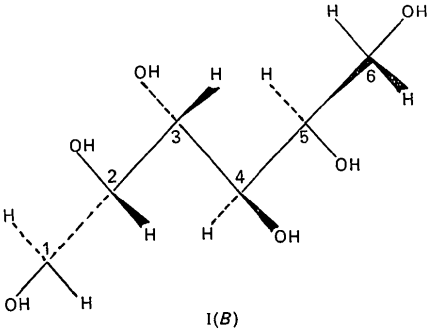
All the stereoisomers, three pentitols and six hexitols, have now been examined except D-altritol. Several are reported to exist in different polymorphic modifications, of which only those of D-mannitol have been studied in detail. Allitol occurs naturally in certain plants. D-iditol is a synthetic product. The naturally occurring form is the L stereoisomer which is found with D-glucitol in mountain ash berries. No polymorphs have been reported for either of these hexitols.

From previous work it has been predicted by Jeffrey & Kim (1970) that in the crystalline state neither allitol nor D-iditol will have the straight-chain conformations shown in I(*A*) and II(*A*), respectively. In allitol, the unique bent chain centrosymmetrical conformation I(*B*) was predicted, while in D-iditol, it was postulated that the conformation II(*B*) would be preferred over II(*C*).

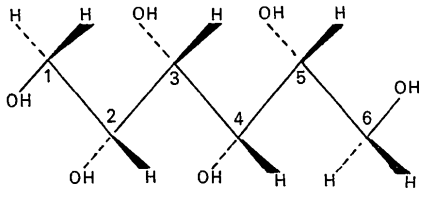
The primary purpose of these structure determinations was to verify these predictions and to study the intermolecular hydrogen bonding and molecular packing.



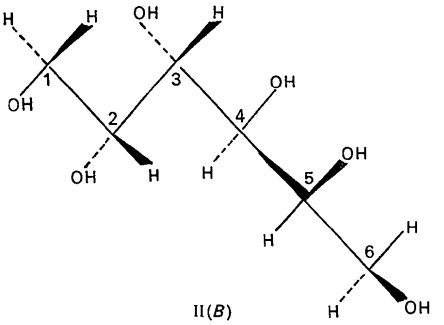
I(*A*)



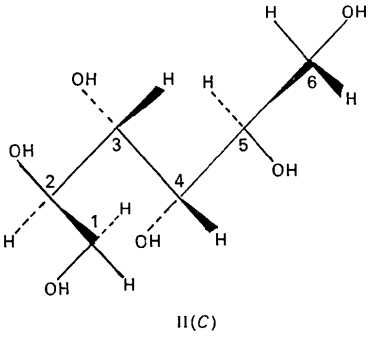
I(*B*)



II(*A*)



II(*B*)



II(*C*)

Crystal data

Good single crystals of allitol were obtained from a specimen supplied by Dr B. E. Stacey, Sir John Cass College, London, EC3. The crystals of D-iditol were obtained from a specimen provided by Dr N. K. Richtmyer of the National Institutes of Health. They were more difficult to obtain in good quality since they are deliquescent. The crystal data reported below were obtained with Ni-filtered Cu $K\alpha$ radiation with the D-iditol crystal enclosed in a thin-walled glass capillary. D-Iditol is not only the most deliquescent of the pentitols and hexitols, but is also the one with the lowest melting point, the highest being that of galactitol at 188 °C.

$C_6H_{14}O_6$, M.W. 184·16

Allitol, m.p. 150–151 °C.	D-Iditol, m.p. 73·5–75·0 °C.
Monoclinic, space group $P2_1/c$, from systematic absences: $0k0$ absent, k odd; $h0l$ absent, l odd.	Monoclinic, space group $P2_1$, from systematic absences: $0k0$ absent, k odd.
$a=4{\cdot}708$ (3) Å	$a=8{\cdot}124$ (2) Å
$b=13{\cdot}408$ (8)	$b=8{\cdot}386$ (3)
$c=6{\cdot}616$ (4)	$c=5{\cdot}870$ (3)
$\beta=100{\cdot}1$ (1)°	$\beta=93{\cdot}8$ (1)°
$V=411{\cdot}15$ Å^3	$V=400{\cdot}44$ Å^3
$Z=2$	$Z=2$
$D_m=1{\cdot}482$ g.cm^{-3}	$D_m=1{\cdot}516$ g.cm^{-3}
$D_x=1{\cdot}472$	$D_x=1{\cdot}510$

Experimental

For both crystal structures, the X-ray data were measured on a Picker FACS I diffractometer with Ni-filtered Cu $K\alpha$ radiation in the $\theta/2\theta$ scanning mode with varying intervals up to $2\theta=130°$. The allitol crystal was $0{\cdot}20\times0{\cdot}25\times0{\cdot}50$ mm and gave 587 observed data out of 701 recorded measurements. The D-iditol crystal was $0{\cdot}30\times0{\cdot}20\times0{\cdot}20$ mm and gave 697 observed data out of 724 recorded. The iditol crystal was mounted in a thin-walled glass capillary to prevent deliquescence. The intensity data were reduced to structure amplitudes without absorption corrections (Shiono, 1969). The density measurements were by flotation in n-hexane and bromoform for allitol and in cyclohexane and bromobenzene for D-iditol.

Determination and Refinement of the structures

For the allitol structure, the phases of 47 reflections with $E>1{\cdot}90$ were generated from the phases of three origin-defining reflections (102; 025; 1,13,3) by application of the Sayre equation. The phase determination was then extended to 105 reflections with $E>1{\cdot}50$ by application of the tangent formula (Karle & Hauptman, 1956), using an IBM version of the Hall (1967) direct phasing program. The six highest peaks on the resulting E-map corresponded to the carbon and oxygen atoms of the asymmetric unit of the molecule. For the D-iditol structure, the phases of three origin-determining reflections (105, 10$\bar{2}$, 115), and two

symbolic phases (for 541 and 170) gave five starting phases for reflections with E's $>2{\cdot}27$ and different parities. The initial R value for allitol was 0·24 for 701 reflections using uniform isotropic temperature factors. The parameters were refined isotropically by the block-diagonal least-squares method to $R=0{\cdot}15$ and thence anisotropically. The seven symmetry-independent hydrogen atoms were located unambiguously on a difference synthesis at $R=0{\cdot}08$. The initial R value for D-iditol was 0·24. The structure was refined first isotropically, then anisotropically to $R=0{\cdot}10$, at which stage the fourteen hydrogen atom peaks were located on the difference synthesis in reasonable spatial relationships to the carbon and oxygen atoms. There were, however, background variations of comparable magnitude, and the location of these atoms directly from the difference synthesis was ambiguous. Both structures were refined by the anisotropic full-matrix least-squares method for the carbon and oxygen atoms (Shiono, 1966). For allitol, the hydrogen parameters were refined isotropically, starting with a uniform value of $B=4{\cdot}0$ Å^2. For D-iditol, the hydrogens were assigned isotropic thermal parameters according to the atoms

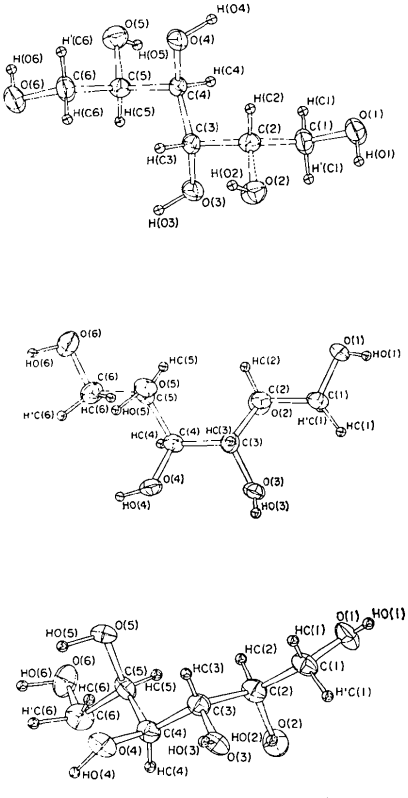


Fig. 1. *ORTEP* plots of the thermal ellipsoids at the 50 % probability level, showing the atomic numbering scheme. Top: allitol, viewed in the direction of the a axis; the center of symmetry is at the midpoint of C(3)–C(4). Middle: D-iditol, viewed with the pseudo-twofold symmetry axis vertical through the midpoint of C(3)–C(4). Bottom: D-iditol, viewed with the principal plane through O(1), C(1), C(2), C(3), C(4), and O(4) normal to the plane of the diagram.

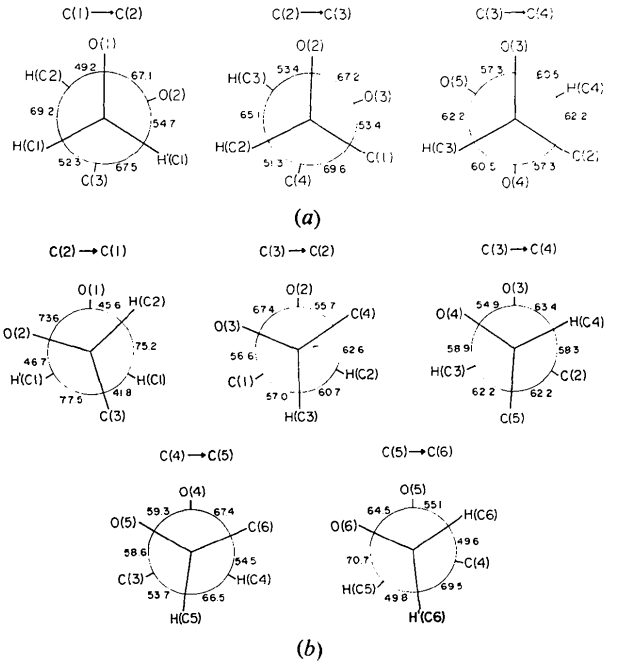


Fig 2. (*a*) Conformational angles about C–C bonds in allitol. (*b*) Conformational angles about C–C bonds in D-iditol.

to which they were attached and neither these nor the positional parameters were refined.

Table 1. *Allitol. Fractional atomic coordinates* ($\times 10^3$) *and anisotropic temperature factors* ($\times 10^4$)

The key to atomic numbering, given in Fig. 1, is consistent with that of other hexitols. The atoms symmetrically related through the molecular and crystallographic center at the origin are (1) and (6), (2) and (5), (3) and (4). The temperature factor expression used was

$$\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)].$$

	x	y	z
C(1)	−74·4 (5)	119·1 (2)	149·3 (3)
C(2)	164·2 (5)	96·2 (2)	328·0 (3)
C(3)	60·4 (4)	53·3 (2)	516·6 (3)
O(1)	41·2 (4)	143·3 (1)	−28·8 (2)
O(2)	318·0 (4)	187·0 (1)	380·7 (3)
O(3)	−153·4 (3)	117·7 (1)	574·4 (2)
Hl(Cl)	−200 (6)	63 (2)	113 (4)
H2(Cl)	−195 (6)	175 (2)	184 (4)
H(C2)	294 (5)	49 (2)	281 (4)
H(C3)	227 (5)	48 (2)	627 (3)
H(O1)	140 (8)	199 (3)	0 (6)
H(O2)	488 (11)	170 (3)	443 (7)
H(O3)	−102 (8)	129 (3)	711 (7)

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	368 (12)	40 (2)	89 (5)	−15 (3)	33 (6)	6 (2)
C(2)	289 (11)	25 (1)	118 (5)	10 (3)	50 (6)	−1 (2)
C(3)	243 (10)	26 (1)	90 (5)	20 (3)	8 (5)	1 (2)
O(1)	571 (11)	42 (1)	92 (4)	−53 (3)	38 (5)	−3 (2)
O(2)	309 (9)	31 (1)	173 (5)	−3 (2)	27 (5)	−2 (2)
O(3)	310 (8)	35 (1)	111 (4)	33 (2)	19 (4)	−6 (1)

In allitol, the Cruickshank (1961) weighting scheme was used ,with $A=0{\cdot}95$, $B=0{\cdot}0$, $C=0{\cdot}03$. In D-iditol

Table 2. D-*Iditol. Fractional atomic coordinates and anisotropic temperature factors*

The temperature factor expression is as in Table 1. Temperature factor for all hydrogens = 2·0.

Atoms other than hydrogen (all values × 10^4)

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	1278 (8)	2986 (10)	5124 (10)	113 (10)	57 (10)	164 (17)	2 (8)	−2 (10)	11 (11)
C(2)	2748 (8)	3267 (10)	6869 (11)	103 (10)	55 (9)	154 (18)	−2 (8)	−4 (10)	−4 (10)
C(3)	3593 (7)	4836 (10)	6458 (9)	80 (9)	79 (10)	130 (15)	−0 (8)	2 (9)	−1 (10)
C(4)	4927 (8)	136 (10)	1879 (10)	94 (9)	61 (9)	148 (16)	0 (7)	6 (10)	−5 (10)
C(5)	6436 (8)	3881 (10)	7924 (10)	98 (10)	74 (9)	137 (16)	20 (8)	−7 (10)	6 (11)
C(6)	7863 (9)	4093 (11)	−296 (12)	114 (10)	100 (12)	180 (19)	−14 (9)	−21 (11)	−10 (13)
O(1)	879 (5)	1338 (9)	5236 (9)	97 (7)	61 (8)	287 (16)	5 (6)	−35 (8)	31 (9)
O(2)	2261 (5)	3167 (8)	9182 (7)	118 (7)	101 (8)	139 (13)	16 (6)	−7 (7)	−32 (9)
O(3)	7588 (6)	1082 (0)	3404 (8)	107 (7)	52 (6)	177 (13)	−5 (5)	−9 (7)	18 (7)
O(4)	4346 (6)	1681 (8)	2407 (7)	110 (7)	60 (7)	200 (14)	−14 (6)	−30 (7)	7 (8)
O(5)	6964 (5)	3860 (8)	5686 (7)	112 (7)	74 (6)	147 (13)	−4 (6)	7 (7)	26 (7)
O(6)	8929 (5)	2778 (9)	9760 (8)	84 (7)	136 (10)	268 (16)	−14 (7)	9 (9)	−46 (10)

Table 2 (*cont.*)

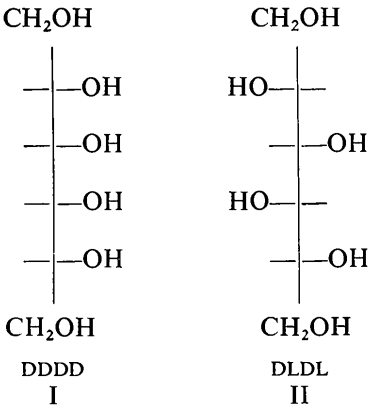
Hydrogen atoms (values × 10^3)

	x	y	z
HC(1)	142	330	389
H*C(1)	020	373	552
HC(2)	360	240	660
HC(3)	390	490	510
HC(4)	510	510	019
HC(5)	600	290	800
HC(6)	740	420	860
H*C(6)	869	505	950
HO(1)	018	132	433
HO(2)	120	280	920
HO(3)	240	680	580
HO(4)	633	710	880
HO(5)	779	478	570
HO(6)	860	200	100

the Hughes (1941) scheme was used, with $F_m = 2{\cdot}0$. In both structure determinations, the atomic scattering factors were those of Cromer & Waber (1965) for carbon and oxygen and of Stewart, Davidson & Simpson (1965) for hydrogen. The final R values for all measured and all observed reflections were 0·04 and 0·05 for allitol, 0·09 and 0·08 for D-iditol, respectively. The final parameters and structure factors are given in Tables 1, 2, and 3. An *ORTEP* (Johnson, 1965) plot of the thermal ellipsoids giving the atomic numbering scheme is shown in Fig. 1.

Discussion

The molecules have the bent-chain conformations I(B) and II(B) as predicted and shown in Fig. 1. In allitol, the molecular configuration, I, is such that there is only one conformation that does not incur a parallel C(n)–O/C($n+2$)–O interaction, and it is the one found in the crystal structure. This conformer I(B) has a center of symmetry which is also a crystallographic center. This contrasts with galactitol (Berman & Rosenstein, 1968) for which the conformational center is not observed in the crystal and the molecules show small but significant distortions from centric symmetry arising from the asymmetric crystal field.



The conformation possibilities for D-iditol are more complex. The configuration II permits two carbon chain conformations without parallel C(n)–O/C($n+2$)–O interaction, II(B) and II(C), and for each of these there are four possible orientations for the terminal CH_2OH groups with respect to the carbon chain. Thus there are eight rotamers which might be expected to have a significant population in solution. The one observed in the crystal structure II(B) has a pseudo-twofold axis of symmetry normal to the bond C(3)–C(4). With the exception of the hydroxyl hydrogens, the positions of which are determined by the intermolecular hydrogen bonding, the deviations from this noncrystallographic molecular axis are less than 0·024 Å. Using a nomenclature such that A = *anti*, P = +*synclinal*, M = −*synclinal*, as viewed down the C(n) → C($n+1$) bond, the observed conformation for allitol is *AMAPA* and for D-iditol it is *AAMAA* (*c.f.* Jeffrey & Kim, 1970).

In allitol, O(1), C(1), C(2), and C(3) lie within 0·06 Å of one plane, and the centrosymmetrically related atoms C(4), C(5), C(6), and O(6) lie near a parallel plane separated from the first by 1·49 Å. The four central carbon atoms [C(2), C(3), C(4), and C(5)] and the two oxygens O(2) and O(5) lie within ±0·1 Å of one plane. In D-iditol, O(1), C(1), C(2), C(3), C(4), and O(4) lie within 0·053 Å of one plane, and C(3), C(4), C(5), C(6), and O(6) lie within 0·05 Å of a second plane, inclined

at 49° from the first. The conformational angles for both molecules are shown in the Boeseken projections along the C–C bonds of Fig. 2.

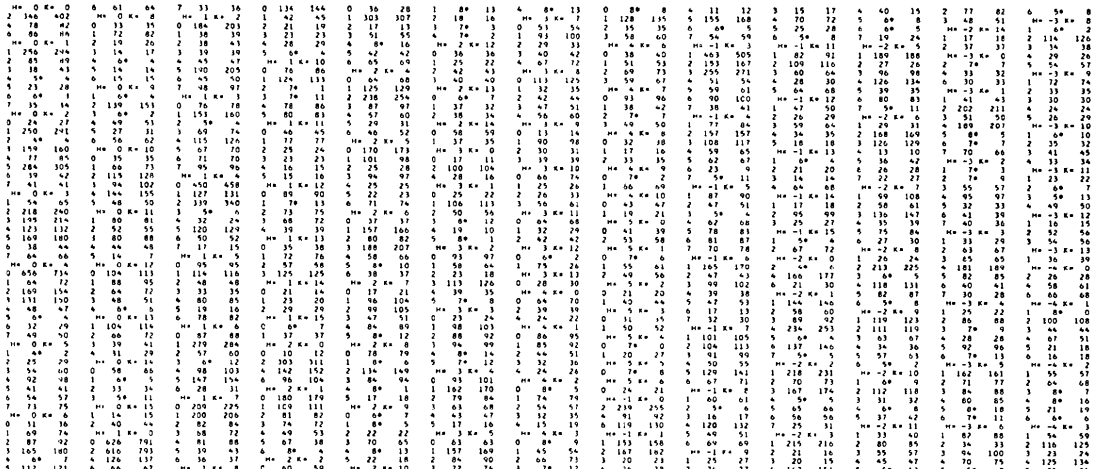
The bond distances and angles for the two molecules are given in Table 4. The mean values of C–C= 1·523 Å, C–O=1·422 Å, C–C–C=113·3°, C–C–O= 109·8° are in good agreement with those from other alditols, and no significance is attached to the small variations, with the possible exception of the C(3)–C(4)–O(4) angle which is about 4° less than the mean. The observed C–H distances vary from 0·96 to 0·99 Å. The hydrogen-bonding distances and angles are given in Table 5.

The hydrogen bonding and molecular packing are illustrated in Fig. 3. In all nine alditols previously studied, each hydroxyl is involved in two bonds, in one as donor and in one as acceptor. This is also true for allitol, but the D-iditol structure is an exception in that one hydroxyl, O(4)H, is a donor only, and one, O(3)H, is a donor for one and an acceptor for three hydrogen bonds. Allitol has two centrosymmetrically related infinite chains, → O(1)H → O(2)H → O(3)H → and → O(6)H → O(5)H → O(4) →, extending in the *b* axis direction. The mean O(H)···O distance is 2·714 Å, and the spread is from 2·645 to 2·755 Å. In D-iditol, the hydrogen-bonding scheme is more complex, with an infinite main chain and a branched chain shown schematically below.

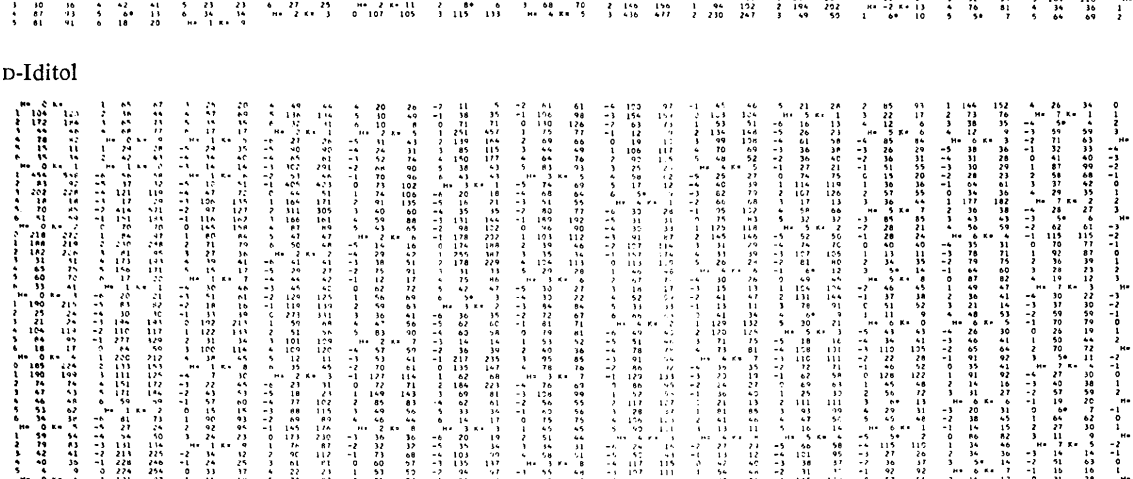
Table 3. *Observed and calculated structure factors*

Columns are *l*, index, 10|F_{obs}|, 10|F_{calc}|. Asterisks indicate unobserved reflections.

(*a*) Allitol



(*b*) D-Iditol



$$\rightarrow O(5)H \rightarrow O(1)H \searrow$$
$$O(3)H \rightarrow$$
$$O(4)H \rightarrow O(2)H \rightarrow O(6)H \nearrow$$

The mean O(H)···O distance is 2·786 Å, and the spread is from 2·742 to 2·837 Å.

Of the ten alditol crystal structures that have been studied, D-iditol is the only structure in which the hydrogen bonding does not consist of one (as in D,L-

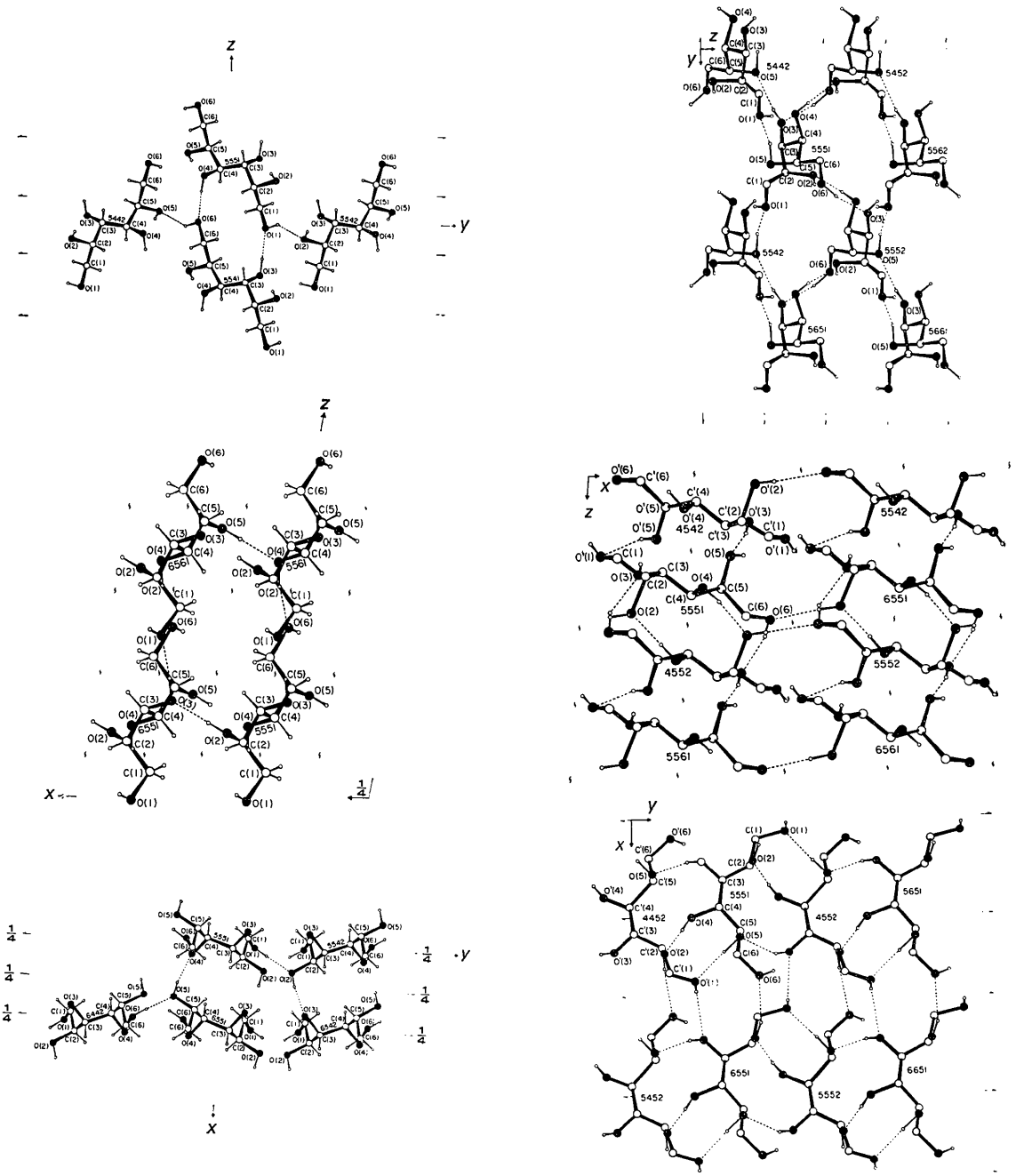


Fig. 3. Left: Allitol crystal structure
top, down *a* axis
middle, down *b* axis
bottom, down *c* axis

Right: *D*-iditol crystal structure
top, down *a* axis
middle, down *b* axis
bottom, down *c* axis.

Dotted lines represent hydrogen bonds. The four-digit numbers code the symmetry operation. The first three refer to the *abc* translations with reference to an origin cell at 555; the last digit specifies one of the following symmetry operations: 1 is x,y,z; 2 is $-x,\frac{1}{2}-y,\frac{1}{2}+z$ for allitol, and $-x,\frac{1}{2}+y,z$ for D-iditol.

arabinitol), two (as in xylitol, D-glucitol, ribitol, galactitol, allitol, and two forms of D-mannitol), or three (as in the meso-erythritol) infinite chains, with each hydroxyl involved in two hydrogen bonds, in one as donor and in one as acceptor.

Table 4. *Bond lengths and bond angles in allitol and* D-*iditol*

		Allitol (e.s.d.'s = 0·003 Å)	D-Iditol (e.s.d.'s = 0·009 Å)
C(1)–C(2)		1·512 Å	1·515 Å
C(2)–C(3)		1·529	1·509
C(3)–C(4)		1·540	1·530
C(4)–C(5)			1·533
C(5)–C(6)			1·522
C(1)–O(1)		1·419	1·420
C(2)–O(2)		1·428	1'438
C(3)–O(3)		1·428	1·425
C(4)–O(4)			1·417
C(5)–O(5)			1·412
C(6)–O(6)			1·401
Mean values:	C–C	1·524	1·522
	C–O	1·425	1·419
		(e.s.d.'s = 0·2°)	(e.s.d.'s = 0·5°)
C(1)–C(2)–C(3)		114·4°	112·4°
C(2)–C(3)–C(4)		113·3	113·0
C(3)–C(4)–C(5)			112·6
C(4)–C(5)–C(6)			113·1
C(2)–C(1)–O(1)		110·7	108·5
C(1)–C(2)–O(2)		107·0	110·3
C(3)–C(2)–O(2)		110·0	110·6
C(2)–C(3)–O(3)		109·3	108·4
C(4)–C(3)–O(3)		109·8	110·8
C(3)–C(4)–O(4)			105·6
C(5)–C(4)–O(4)			111·3
C(4)–C(5)–O(5)			110·3
C(6)–C(5)–O(5)			111·3
C(5)–C(6)–O(6)			111·9
Mean values:	C–C–C	113·9°	112·8°
	C–C–O	109·4	110·0

This research is supported by Grant No. GM-11293 of the U. S. Public Health Service, National Institutes of Health. We are grateful to Dr R. Rosenstein for his assistance in the structure determination of D-iditol.

Table 5. *Hydrogen bonding distances and angles*

i *j*	*l*	D_{ij}	D_{il}	D_{jl}	$\angle iil$
Allitol					
O(1)–H	O(2)	0·88 Å	1·97 Å	2·739 Å	144°
O(2)–H	O(3)	0·86	1·89	2·752	173
O(3)–H	O(1)	0·91	1·74	2·646	172
D-Iditol					
O(5)–H	O(1)	1·01	1·81	2·77	158
O(1)–H	O(3)	0·89	2·15	2·83	150
O(3)–H	O(5)	0·90	2·01	2·74	160
O(4)–H	O(2)	1·00	1·79	2·77	174
O(2)–H	O(6)	0·92	1·89	2·76	159
O(6)–H	O(3)	1·02	1·84	2·83	163

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