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Structure of D-Perseitol (D-*glycero*-D-*galacto*-Heptitol)

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Abstract. $C_7H_{16}O_7$, $M_r = 212{\cdot}20$, monoclinic, $P2_1$, $a = 9{\cdot}748\,(4)$, $b = 4{\cdot}851\,(2)$, $c = 20{\cdot}013\,(1)$ Å, $\beta = 99{\cdot}61\,(2)°$, $V = 933{\cdot}1\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}510$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 11{\cdot}4$ cm^{-1}, $F(000) = 456$, $T = 295$ K, final $R = 0{\cdot}039$ for 1713 unique observed data. The asymmetric unit consists of two independent molecules which have identical planar extended carbon-chain conformations. All 14 hydroxyl groups are involved as donors and acceptors in intermolecular hydrogen bonds. There are three bifurcated hydrogen bonds involving one of the independent molecules; each consists of a strong intermolecular and a weaker intramolecular interaction. Some of the C—C—O—H torsion angles deviate substantially from the ideal staggered conformations.

Introduction. In the class of alditols the crystal structures of all possible pentitols and hexitols are known (Jeffrey & Kim, 1970). Of the class of heptitols, which consists of 16 members, three occur in plants, D-volemitol (D-*glycero*-D-*manno*-heptitol) in the mushroom *Lactarius volemus*, β-sedoheptitol (D-*glycero*-D-*gluco*-heptitol) in the stem and leaves of *Sedum spectabile*, and D-perseitol (D-*glycero*-D-*galacto*-heptitol) in *Laureus persea* L. (avocado) (Hough & Richardson, 1967; Ferrier, 1983). Of the 16 possible heptitols four are *meso* forms and 12 consist of six enantiomeric pairs. Thirteen heptitols are known in the literature.

So far only the crystal structures of *meso*-L-*glycero*-L-*gulo*-heptitol (GGH) (Nimgirawath, James & Mills, 1976) and of D-*glycero*-L-*allo*-heptitol (GAH) (Angyal, Saunders, Grainger, Le Fur & Williams, 1986) have been determined. GGH crystallizes in space group $P2_1/c$ as an assembly of equal numbers of enantiomorphous non-planar conformers and GAH in space group $P2_12_12$ with non-planar conformers of one handedness. Two other *meso*-alditols have been shown to crystallize as enantiomorphous conformers: ribitol crystallizes in space group $P2_1/c$ where both enantiomers are present (Kim, Jeffrey & Rosenstein, 1969) and xylitol has space group $P2_12_12_1$ and contains conformers of only one handedness (Kim & Jeffrey, 1969). We now report the crystal structure of D-perseitol (D-*glycero*-D-*galacto*-heptitol).

Experimental. Crystals suitable for X-ray analysis were obtained from a commercial sample of D-perseitol by crystallization from 1-propanol/water. A colourless thin plate of dimensions $0{\cdot}2 \times 0{\cdot}1 \times 0{\cdot}05$ mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation. Lattice parameters were determined from the setting angles of 25 reflections with $13{\cdot}9 < \theta < 18{\cdot}4°$. The intensity data of 1790 reflections were collected in one quarter of the reflection sphere ($-11 \le h \le 0$, $-5 \le k \le 0$, $-24 \le l \le 24$) with $2\theta_{\max} = 140°$, of which 1713 were above the $2{\cdot}5\sigma(I)$ level. The ω–θ scan mode was applied with $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)°$. Two reference reflections, $\bar{1}\bar{1}\bar{6}$ and $2\bar{1}5$, measured every hour showed no significant variations during 11 h of X-ray exposure time. The data were corrected for Lorentz–polarization effects, but not for absorption. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). The solution with the highest figure of merit showed all non-H atoms. The structure obtained was inverted so as to comply with the known chirality. The aliphatic H atoms were placed at calculated C(sp^3) positions with C—H 1·00 Å and refined riding on their carrier C atoms. The hydroxyl-group H atoms were located from a difference map. Blocked-matrix refinement on F of 296 parameters including scale factor, positional and anisotropic thermal parameters for non-H atoms, positions of the hydroxyl-group H atoms and a general isotropic thermal parameter for all H atoms resulted in $R = 0{\cdot}039$, $wR = 0{\cdot}052$, $w = 1/\sigma^2(F_o)$, $S = 1{\cdot}19$ and $U_{\rm iso}(\rm H) = 0{\cdot}043\ (4)$ Å^2. Minimum and maximum residual electron densities in the final Fourier map were $-0{\cdot}22$ and $0{\cdot}26$ e Å^{-3} respectively. The average and max. shift-to-e.s.d. ratios in the final stage were 0·007 and 0·025 respectively. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Programs used include *SHELX76* (refinement) (Sheldrick, 1976) and the *EUCLID* package (geometry calculations and refinement) (Spek, 1982).

Discussion. The final fractional coordinates and (equivalent) isotropic thermal parameters of C and O atoms and the hydroxyl-group H atoms are listed in Table 1.* Bond distances and angles of the independent molecules are listed in Table 2. A perspective view of the two independent molecules with the adopted numbering scheme is shown in Fig. 1. The corresponding distances and angles of the two molecules compare very well; the average deviations are 0·005 (4) Å and 0·08 (6)° respectively. The C—C—C angles are consistently larger than the tetrahedral value, which was also observed for GGH (Nimgirawath, James & Mills, 1976) and GAH (Angyal *et al.*, 1986). The corresponding torsion angles display larger deviations; the range is 0·2–6·6° with an average of 4 (2)°. The carbon chains have extended planar conformations; the $\sigma_{\rm plane}$ values are 0·030 (2) and 0·025 (3) Å, for molecules I and II respectively, and the two planes are perpendicular [angle of 89·4 (2)°].

The conformation of the six O—C—C—O fragments of both molecules is $ag^-ag^+ag^-$, implying three planar O—C—C—O fragments in each molecule.

Table 1. *Final fractional coordinates of* C *and* O *atoms with equivalent isotropic thermal parameters* (Å^2) *and of hydroxyl-group* H *atoms with general isotropic thermal parameters* (Å^2)

E.s.d.'s are given in parentheses.

	x	y	z	$U_{\rm eq}$†
Molecule I				
O(1)	0·2345 (3)	0·0743 (6)	0·4574 (1)	0·0407 (8)
O(2)	0·4348 (2)	−0·4111 (5)	0·3770 (1)	0·0286 (6)
O(3)	0·3542 (2)	0·1126 (5)	0·3103 (1)	0·0239 (6)
O(4)	0·1226 (2)	−0·4808 (4)	0·2693 (1)	0·0217 (6)
O(5)	0·0552 (2)	0·0485 (5)	0·2012 (1)	0·0250 (6)
O(6)	0·2446 (2)	−0·4680 (5)	0·1244 (1)	0·0318 (6)
O(7)	0·1796 (3)	0·0565 (6)	0·0539 (1)	0·0390 (8)
C(1)	0·3507 (3)	−0·0861 (7)	0·4474 (1)	0·027 (1)
C(2)	0·3109 (3)	−0·2644 (6)	0·3845 (1)	0·0211 (6)
C(3)	0·2578 (3)	−0·0962 (6)	0·3212 (1)	0·0188 (6)
C(4)	0·2233 (3)	−0·2815 (6)	0·2580 (1)	0·0180 (6)
C(5)	0·1734 (3)	−0·1129 (6)	0·1939 (1)	0·0203 (6)
C(6)	0·1287 (3)	−0·2994 (6)	0·1323 (1)	0·0215 (6)
C(7)	0·0776 (4)	−0·1346 (8)	0·0685 (1)	0·031 (1)
H(O1)	0·244 (3)	0·114 (8)	0·498 (1)	0·043 (4)
H(O2)	0·410 (4)	−0·530 (9)	0·355 (2)	0·043 (4)
H(O3)	0·422 (3)	0·056 (9)	0·295 (2)	0·043 (4)
H(O4)	0·051 (3)	−0·402 (9)	0·277 (2)	0·043 (4)
H(O5)	0·074 (4)	0·165 (9)	0·220 (2)	0·043 (4)
H(O6)	0·222 (4)	−0·596 (9)	0·095 (2)	0·043 (4)
H(O7)	0·210 (4)	0·01 (1)	0·018 (2)	0·043 (4)
Molecule II				
O(11)	0·9648 (2)	0·2604 (5)	0·4081 (1)	0·0350 (6)
O(12)	0·7062 (2)	−0·2725 (6)	0·4081 (1)	0·0316 (6)
O(13)	0·8974 (2)	−0·3260 (5)	0·3158 (1)	0·0315 (6)
O(14)	0·5772 (2)	0·0477 (6)	0·2541 (1)	0·0312 (6)
O(15)	0·7732 (2)	0·0181 (5)	0·1631 (1)	0·0290 (6)
O(16)	0·5251 (3)	−0·5370 (5)	0·1593 (1)	0·0346 (6)
O(17)	0·7257 (4)	−0·5583 (6)	0·0644 (1)	0·049 (1)
C(11)	0·8894 (3)	0·0401 (8)	0·4325 (1)	0·031 (1)
C(12)	0·7633 (3)	−0·0364 (7)	0·3801 (1)	0·0251 (8)
C(13)	0·8018 (3)	−0·1028 (7)	0·3110 (1)	0·0225 (6)
C(14)	0·6759 (3)	−0·1709 (7)	0·2572 (1)	0·0218 (6)
C(15)	0·7152 (3)	−0·2239 (7)	0·1877 (1)	0·0241 (6)
C(16)	0·5877 (3)	−0·2985 (7)	0·1354 (1)	0·0263 (8)
C(17)	0·6209 (4)	−0·3544 (8)	0·0648 (1)	0·038 (1)
H(O11)	1·049 (4)	0·20 (1)	0·419 (2)	0·043 (4)
H(O12)	0·613 (4)	−0·288 (9)	0·394 (2)	0·043 (4)
H(O13)	0·893 (4)	−0·395 (9)	0·352 (2)	0·043 (4)
H(O14)	0·581 (4)	0·156 (9)	0·225 (2)	0·043 (4)
H(O15)	0·859 (4)	0·02 (1)	0·176 (2)	0·043 (4)
H(O16)	0·438 (4)	−0·517 (9)	0·151 (2)	0·043 (4)
H(O17)	0·706 (4)	−0·675 (9)	0·085 (2)	0·043 (4)

† $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

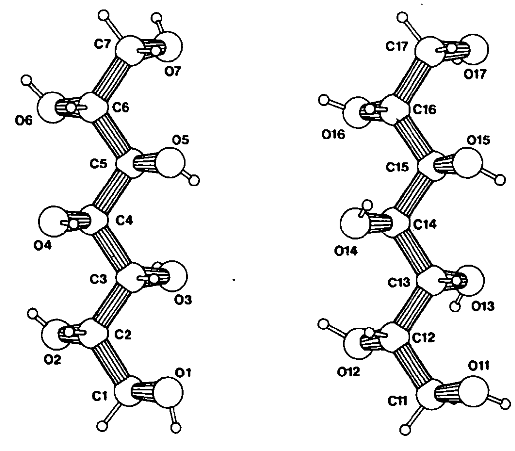

Fig. 1. Molecular conformations with atom numbering of the two independent molecules of D-perseitol as viewed perpendicular to the plane of the C atoms. Note the large difference between the corresponding C—C—O—H torsion angles.

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* Lists of structure amplitudes, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52178 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and bond angles* (°)

	Molecule I	Molecule II
O(1)—C(1)	1·416 (4)	1·428 (4)
O(2)—C(2)	1·431 (4)	1·428 (4)
O(3)—C(3)	1·423 (4)	1·421 (4)
O(4)—C(4)	1·423 (4)	1·426 (4)
O(5)—C(5)	1·421 (4)	1·426 (4)
O(6)—C(6)	1·425 (4)	1·427 (4)
O(7)—C(7)	1·425 (5)	1·423 (5)
C(1)—C(2)	1·523 (3)	1·523 (4)
C(2)—C(3)	1·523 (3)	1·527 (3)
C(3)—C(4)	1·542 (3)	1·528 (4)
C(4)—C(5)	1·531 (3)	1·525 (3)
C(5)—C(6)	1·533 (3)	1·529 (4)
C(6)—C(7)	1·518 (4)	1·526 (3)
O(1)—C(1)—C(2)	109·2 (2)	110·4 (2)
O(2)—C(2)—C(1)	105·4 (2)	104·4 (2)
O(2)—C(2)—C(3)	110·8 (2)	110·9 (3)
C(1)—C(2)—C(3)	112·8 (2)	112·5 (2)
O(3)—C(3)—C(2)	111·6 (2)	110·9 (2)
O(3)—C(3)—C(4)	110·4 (2)	108·9 (2)
C(2)—C(3)—C(4)	111·5 (2)	113·2 (2)
O(4)—C(4)—C(3)	109·1 (2)	109·1 (2)
O(4)—C(4)—C(5)	111·4 (2)	110·9 (2)
C(3)—C(4)—C(5)	111·8 (2)	112·6 (2)
O(5)—C(5)—C(4)	111·0 (2)	110·5 (3)
O(5)—C(5)—C(6)	106·3 (2)	106·4 (2)
C(4)—C(5)—C(6)	111·5 (2)	111·4 (2)
O(6)—C(6)—C(5)	107·7 (2)	107·9 (2)
O(6)—C(6)—C(7)	111·3 (2)	109·6 (3)
C(5)—C(6)—C(7)	112·0 (2)	113·7 (3)
O(7)—C(7)—C(6)	111·9 (3)	113·1 (2)

Table 3. *Geometry of the hydrogen bonds* (Å,°)

D—H···*A*	*D*···H	*D*···*A*	H···*A*	*D*—H···*A*	Symmetry operation*
O(1)—H(O1)···O(12)	0·83 (4)	2·759 (3)	1·94 (2)	171 (3)	2·656
O(2)—H(O2)···O(3)	0·74 (4)	2·718 (3)	1·98 (4)	170 (4)	1·545
O(3)—H(O3)···O(14)	0·82 (3)	2·628 (3)	1·84 (3)	161 (4)	1·555
O(4)—H(O4)···O(13)	0·83 (3)	2·633 (3)	1·84 (3)	160 (4)	1·455
O(5)—H(O5)···O(4)	0·69 (4)	2·684 (3)	2·00 (4)	176 (5)	1·565
O(6)—H(O6)···O(7)	0·86 (4)	2·723 (4)	1·89 (4)	163 (4)	1·545
O(7)—H(O7)···O(17)	0·85 (4)	2·738 (4)	1·89 (4)	175 (5)	2·655
O(11)—H(O11)···O(1)	0·86 (4)	2·799 (4)	1·94 (4)	171 (4)	1·655
O(12)—H(O12)···O(2)	0·91 (4)	2·700 (3)	1·81 (4)	164 (4)	1·555
O(13)—H(O13)···O(12)	0·81 (4)	2·847 (3)	2·37 (4)	119 (4)	1·555
O(13)—H(O13)···O(11)	0·81 (4)	2·732 (3)	2·07 (4)	139 (4)	1·545
O(14)—H(O14)···O(15)	0·79 (4)	2·855 (3)	2·50 (4)	109 (3)	1·555
O(14)—H(O14)···O(16)	0·79 (4)	2·756 (4)	2·00 (4)	160 (4)	1·565
O(15)—H(O15)···O(5)	0·83 (4)	2·731 (3)	1·90 (4)	176 (4)	1·655
O(16)—H(O16)···O(6)	0·84 (4)	2·727 (4)	1·89 (4)	175 (4)	1·555
O(17)—H(O17)···O(15)	0·74 (4)	2·834 (4)	2·18 (4)	147 (4)	1·545
O(17)—H(O17)···O(16)	0·74 (4)	2·946 (4)	2·58 (4)	113 (4)	1·555

* The symmetry operation is performed on the acceptor O atoms. The first digit indicates one of the following symmetry operations: (1) *x*, *y*, *z*; (2) $-x$, $\frac{1}{2}+y$, $-z$. The last set of numbers specifies the lattice translations, *e.g.* 2·656 is **a** + **c** from 2·555.

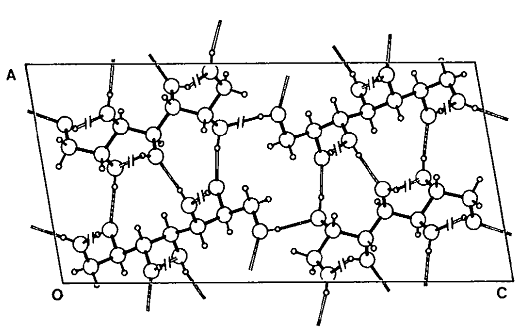

Fig. 2. View of the contents of the unit cell along **b**, showing the intermolecular hydrogen bonds. Only the hydroxyl-group H atoms are shown. Hydrogen bonds connecting molecules translated in the + or − **b** directions are denoted by the symbol —||—.

Table 4. *Comparison of corresponding* C—C—O—H *torsion angles* (°)

	Molecule I	Molecule II
C(2)—C(1)—O(1)—H(O1)	155 (3)	−135 (3)
C(1)—C(2)—O(2)—H(O2)	160 (3)	152 (3)
C(2)—C(3)—O(3)—H(O3)	76 (3)	16 (3)
C(3)—C(4)—O(4)—H(O4)	59 (3)	97 (3)
C(4)—C(5)—O(5)—H(O5)	79 (4)	89 (3)
C(5)—C(6)—O(6)—H(O6)	170 (3)	140 (3)
C(6)—C(7)—O(7)—H(O7)	112 (3)	47 (3)

All 14 hydroxyl-group H atoms are involved in intermolecular hydrogen bonds (Table 3), each O atom accepting one intermolecular bond. The two independent molecules form a regular hydrogen-bond pattern (Fig. 2) in which each molecule is bonded to six neighbours. The short *b* axis [4·851 (2) Å] causes most of the hydrogen bonds to connect molecules that are related by translations along *b*. Three O—H donors [O(13), O(14) and O(17) of molecule II] are involved in asymmetric bifurcated hydrogen bonds according to the classification of Newton, Jeffrey & Takagi (1979), the weaker interaction of each bifurcated bond being intramolecular. These four-atom configurations approach planarity as follows from the sums of angles around the H atom, which amount to 356 (6), 360 (5) and 345 (6)° respectively.

Although the geometries involving non-H atoms of molecules I and II do not differ appreciably (see Table 2), the corresponding C—C—O—H conformational angles not only display remarkable differences, but some of these also deviate substantially from the ideal staggered conformation and approach unfavourable eclipsed conformations (Table 4). These effects must be ascribed to the demands of the hydrogen-bond network which clearly overrule those of the intramolecular *X*—C—O—H torsional energy.

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Acta Cryst. (1990). C**46**, 74–78

Structure of a 1:1 Complex between *N*-Boc-L-Pro-L-Val-OCH$_3$ (I) and *N*-Boc-L-Pro-C$^\beta$-Methylated-L-Val-OCH$_3$ (II)

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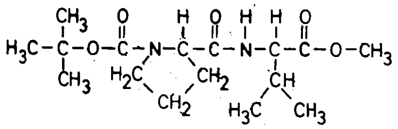
Abstract. $C_{16}H_{28}N_2O_5.C_{17}H_{30}N_2O_5$, $M_r = 670{\cdot}84$, triclinic, $P1$, $a = 9{\cdot}698\,(4)$, $b = 10{\cdot}173\,(3)$, $c = 10{\cdot}557\,(2)$ Å, $\alpha = 110{\cdot}19\,(2)$, $\beta = 97{\cdot}49\,(2)$, $\gamma = 90{\cdot}64\,(2)°$, $V = 967{\cdot}4\,(5)$ Å^3, $Z = 1$, $D_m = 1{\cdot}104\,(5)$, $D_x = 1{\cdot}151\,(5)$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}63$ cm^{-1}, $F(000) = 364$, $T = 298$ K, final $R = 0{\cdot}061$ for 2772 unique observed reflections. The peptides were synthesized by combination of *N*-Boc-L-Pro and valine methyl ester (I) and *N*-Boc-L-Pro and C$^\beta$-methylated valine methyl ester (II). The molecules of the 1:1 complex are hydrogen-bonded together *via* the carbonyl O atoms of the Pro residues of (I) and (II) and the amino N atoms of valine and C$^\beta$-methylated valine respectively. The molecular dimensions in the methyl ester moieties of (I) and (II) show significant departures from the standard values. The backbone conformations of the two peptides are similar. The Boc groups adopt *trans–cis* conformations. The pyrrolidine rings of the Pro residues exist in C$^\gamma$-*endo* conformations. The torsion angles that define the conformations of the valine residues are close to −60 and 60° respectively. The crystal structure is essentially stablilized by a network of van der Waals interactions and hydrogen bonds.

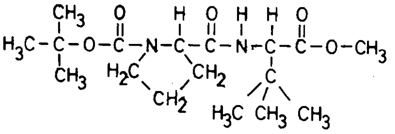
Introduction. The conformational preferences of amino acid side chains are of fundamental importance in determining the interactions that govern the preferred conformations of polypeptides and proteins (Chandrasekharan & Ramachandran, 1970; Janin, Wodak, Levitt & Maigret, 1978; Bhat, Sasisekharan & Vijayan, 1979) and of oligopeptides (Benedetti, Morelli, Nemethy & Scheraga, 1983). Conformational preferences may be determined by conformational-energy computations (Zimmerman, Pottle, Nemethy & Scheraga, 1977; Vasquez, Nemethy & Scheraga, 1983). They are basically governed to a large extent by interactions of a given side chain with atoms of the two neighbouring units. It is therefore worthwhile to examine the backbone and side-chain conformations of peptides with only marginal differences in the size of the side chains. We report here the crystal structure of a 1:1 complex between *N*-Boc-L-Pro-L-Val-OCH$_3$ (I) and *N*-Boc-L-Pro-C$^\beta$-methylated-L-Val-OCH$_3$ (II). The peptide (I) has been synthesized earlier (Crisma, Fasman, Balaram & Balaram, 1984).

(I)



+

(II)



Experimental. The amino acids valine and *tert*-butylglycine (C$^\beta$-methylated valine) were obtained from Sigma Chemical Company. The valine methyl ester and C$^\beta$-methylated valine methyl ester were synthesized by reacting valine and C$^\beta$-methylated valine separately in methanol with thionyl chloride, which was added drop by drop. The mixture was stirred for 10 min at 263 K. *N*-Boc-L-Pro-L-Val-OCH$_3$ (I) and *N*-Boc-L-Pro-C$^\beta$-methylated-L-Val-OCH$_3$ (II) were synthesized by coupling *N*-Boc-L-Pro-OH with valine methyl ester and C$^\beta$-methylated valine methyl ester, respectively, using the procedure described by Poisel (1977). A 1:1 mixture of peptides (I) and (II) was crystallized from their solution in methanol at 298 K. Large, irregular and colourless crystals; one with

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