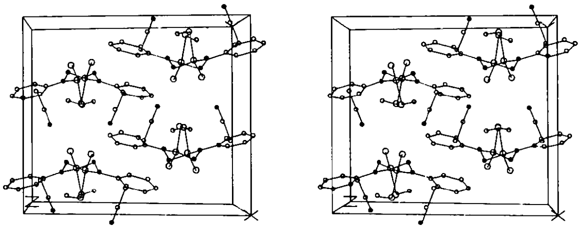

Fig. 2. Arrangement of the molecules (H atoms deleted) in the unit cell, viewed along *x*. Stereoplot drawn with *CELLGRAPH* (Reck & Kretschmar, 1989).

and two times +33·2° (Hendrickson, 1961). The pseudoaxial substituents at the P atoms are arranged in *cis* positions with respect to the P_2N_4 ring. The angle between the planar phenyl ring and the least-squares plane through the P_2N_4 ring is 23·6°.

The unit cell (Fig. 2) contains two pairs of molecules which are symmetry related by a glide plane. The shortest intermolecular distances exist between phenyl groups [C(12)···C(14^{ii}) 3·521 Å] and between a phenyl group and the methyl group of the acetonitrile molecule [C(12)···C(2^{iii}) 3·524 Å] [(ii) $-x$, $1-y$, $-z$; (iii) $\frac{1}{2}-x$, $\frac{1}{2}-y$, $-z$]. These distances do not indicate interactions stronger than van der Waals forces.

The comparatively short N—N bond length together with the sum of the bond angles around N(1) (356·5°) shows that the bonding state at the N(1) atom tends to sp^2 hybridization. The shortest N—N distance in a phosphorus hydrazine heterocycle (1.400 Å) has been found in *cis*-3,6-dithioxo-3,6-diphenoxy-1,2,4,5-tetraaza-3λ^5,6λ^5-diphosphacyclohexane (Engelhardt & Hartl, 1976), which has an angular sum of 359·3°.

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Structure of 1,2,3,4,5,6-Hexa-*O*-acetyl-D-glucitol (Sorbitol Hexaacetate)

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Dedicated to Professor Dr E. Weiß on the occasion of his 65th birthday

Abstract. $C_{18}H_{26}O_{12}$, M_r = 434·40, monoclinic, $P2_1$, a = 10·253 (1), b = 8·370 (1), c = 12·548 (1) Å, β = 95·98 (5)°, V = 1071·0 (2) Å^3, Z = 2, D_x = 1·347 g cm^{-3}, λ(Mo $K\alpha_1$) = 0·709261 Å, μ = 1·1 cm^{-1}, F(000) = 460, T = 293 K, R = 0·053 for 1681 observed reflexions. The molecules of the title compound have a planar zigzag carbon-chain conformation which aligns O(2) 1,3-parallel to O(4). The primary O(1) is found in the same O//O relation to O(3).

Introduction. The X-ray structures of many alditols (sugar alcohols) up to a chain length of C_7 have been determined. Generally, planar (zigzag) conformations are expected but in most cases they are found in bent (sickle) carbon-chain conformations in order to avoid unfavorable 1,3-parallel interactions of C and O atoms (designated C//O and O//O). The

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general avoidance of O//O interactions (which resemble 1,3-diaxial interactions in the cyclic case) was accepted as a rule by Jeffrey (1990) and incorrectly assigned to Hassel and Ottar who, indeed, were very cautious in speculations about the steric influence of such a geometry (Hassel & Ottar, 1947).

Recently we reported structural examples where significant violations of this rule in alditol derivatives are observed (Kopf, Brandenburg, Seelhorst & Köll, 1990; Köll, Malzahn & Kopf, 1990). Up to now no example is reported where such an interaction in a simple alditol is tolerated. This situation is different for C//O interactions, and in D-altritol (Kopf, Bischoff & Köll, 1991), D-*glycero*-L-*allo*-heptitol (Angyal, Saunders, Grainger, Le Fur & Williams, 1986) and D-*glycero*-D-*manno*-heptitol (Köll, Komander, Angyal, Morf, Zimmer & Kopf, 1991) such relations are accepted.

Since it is known that the steric demands of acetylated O atoms are less than those of free hydroxyl groups (Lemieux & Pavia, 1969; Paulsen & Friedmann, 1972), we initiated a project to study the solid-state conformations of alditol acetates by X-ray crystallography. No crystal structure determinations of acetylated alditols have been published previously.

Experimental. Crystals of 1,2,3,4,5,6-hexa-*O*-acetyl-D-glucitol (Zäch, 1929) suitable for X-ray analysis were obtained by cooling a saturated hot solution of the substance in ethanol. The crystals obtained melted at 372 K. Colorless transparent crystal of dimensions 0·3 × 0·3 × 0·2 mm used for data collection. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell parameters determined by least-squares refinement of the setting angles of 25 reflexions within $16{\cdot}2 \leq 2\theta \leq 21{\cdot}3°$. Space group and lattice parameters in accordance with the literature (French, 1954). Intensity data measured by $\theta/2\theta$ scans ($4{\cdot}5 \leq 2\theta \leq 50°$, $0 \leq h \leq 12$, $0 \leq k \leq 9$, $-14 \leq l \leq 14$). No significant decay in the intensities for three standards monitored every 2 h. 2168 data measured, of which 1906 were symmetry independent ($R_{\rm int} = 0{\cdot}021$). 1681 intensities considered observed [$|F_o| > 3\sigma(F_o)$]. Data corrected for Lorentz and polarization effects, but not for absorption.

Structure was solved by direct methods. The solution with the highest figure of merit showed all non-H atoms. The structure obtained was in accordance with the known chirality. All H atoms were localized in theoretical positions (sp^3 hybridization) with a C—H distance of 0·96 Å. Blocked-matrix least-squares refinement on F of 375 parameters including scale factor, positional and anisotropic thermal parameters for all non-H atoms, and positional and isotropic parameters for all H atoms

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

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

$$U_{\rm eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2)
O(1)	1·1176 (3)	0·1826 (4)	0·1624 (2)	0·0508 (7)
O(2)	1·0596 (3)	0·4008 (4)	0·3199 (2)	0·0414 (7)
O(3)	0·8653 (3)	0·1725	0·2405 (2)	0·0395 (6)
O(4)	0·7990 (3)	0·4016 (4)	0·3889 (2)	0·0445 (7)
O(5)	0·5845 (3)	0·3631 (4)	0·1446 (2)	0·0454 (7)
O(6)	0·5222 (3)	0·5975 (4)	0·2962 (3)	0·0588 (7)
O(11)	1·1456 (4)	0·1249 (5)	−0·0074 (2)	0·0704 (7)
O(21)	1·1738 (4)	0·6235 (5)	0·2924 (3)	0·0795 (8)
O(31)	0·8112 (4)	0·0960 (5)	0·0701 (3)	0·0744 (7)
O(41)	0·8714 (4)	0·6546 (5)	0·4041 (3)	0·0707 (7)
O(51)	0·4711 (4)	0·1386 (6)	0·1500 (3)	0·0965 (8)
O(61)	0·5250 (5)	0·6607 (6)	0·4686 (3)	0·0921 (8)
C(1)	1·0782 (5)	0·3446 (6)	0·1317 (3)	0·0502 (8)
C(2)	0·9942 (4)	0·4114 (6)	0·2123 (3)	0·0391 (7)
C(3)	0·8594 (4)	0·3385 (5)	0·2097 (3)	0·0370 (8)
C(4)	0·7629 (4)	0·4217 (6)	0·2756 (3)	0·0383 (7)
C(5)	0·6246 (5)	0·3504 (6)	0·2573 (3)	0·0435 (8)
C(6)	0·5275 (5)	0·4292 (6)	0·3222 (4)	0·0577 (8)
C(11)	1·1488 (4)	0·0859 (6)	0·0842 (4)	0·0475 (8)
C(12)	1·1887 (5)	−0·0764 (7)	0·1253 (4)	0·0633 (8)
C(21)	1·1474 (5)	0·5185 (6)	0·3513 (4)	0·0533 (8)
C(22)	1·2023 (5)	0·4963 (7)	0·4622 (4)	0·0666 (8)
C(31)	0·8385 (4)	0·0615 (5)	0·1628 (4)	0·0442 (8)
C(32)	0·8438 (5)	−0·1027 (6)	0·2060 (4)	0·0608 (8)
C(41)	0·8476 (5)	0·5306 (6)	0·4456 (3)	0·0506 (8)
C(42)	0·8673 (5)	0·4974 (7)	0·5621 (4)	0·0669 (8)
C(51)	0·5076 (5)	0·2469 (6)	0·0990 (4)	0·0526 (8)
C(52)	0·4749 (6)	0·2673 (8)	−0·0174 (4)	0·0795 (8)
C(61)	0·5191 (5)	0·7001 (7)	0·3778 (4)	0·0539 (8)
C(62)	0·5031 (6)	0·8654 (7)	0·3379 (4)	0·0748 (8)

resulted in $R = 0{\cdot}053$ and $wR = 0{\cdot}051$. Owing to the polar space group $P2_1$ the y coordinate of O(3) was fixed. The ratio of observations to number of variables is 4·5. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with weights $w = 1/\sigma^2(|F_o|)$. Max. shift/e.s.d. was 0·68 in the final cycle; max. and min. heights in the final $\Delta\rho$ map were 0·26 and $-0{\cdot}25$ e Å^{-3}, respectively. The complex neutral-atom scattering factors were taken from *SHELX76*. Programs used were *SHELXS90* (Sheldrick, 1990), *SHELX76* (Sheldrick, 1976), *PLATON88* (Spek, 1982) and *SCHAKAL88* (Keller, 1986) on MicroVAX II and VAX 3200 computers.

Discussion. The final fractional coordinates and equivalent isotropic thermal parameters of C and O atoms are listed in Table 1.* Bond distances and angles between C and O atoms and some selected torsion angles of the title compound are given in Table 2. A perspective view of the molecule including the numbering scheme is presented in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, fractional coordinates and isotropic thermal parameters of H atoms, bond distances and angles involving H atoms, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54191 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *between* C *and* O *atoms and selected torsion angles* (°)

O(1)—C(1)	1·455 (6)	O(1)—C(11)	1·336 (6)
O(2)—C(2)	1·446 (5)	O(2)—C(21)	1·365 (6)
O(3)—C(3)	1·442 (4)	O(3)—C(31)	1·354 (5)
O(4)—C(4)	1·441 (5)	O(4)—C(41)	1·359 (6)
O(5)—C(5)	1·435 (5)	O(5)—C(51)	1·342 (6)
O(6)—C(6)	1·446 (6)	O(6)—C(61)	1·339 (6)
O(11)—C(11)	1·192 (6)	O(21)—C(21)	1·197 (6)
O(31)—C(31)	1·203 (6)	O(41)—C(41)	1·198 (6)
O(51)—C(51)	1·192 (7)	O(61)—C(61)	1·182 (6)
C(1)—C(2)	1·503 (6)	C(2)—C(3)	1·508 (6)
C(3)—C(4)	1·523 (6)	C(4)—C(5)	1·534 (7)
C(5)—C(6)	1·503 (7)	C(11)—C(12)	1·495 (8)
C(21)—C(22)	1·458 (7)	C(31)—C(32)	1·476 (7)
C(41)—C(42)	1·481 (6)	C(51)—C(52)	1·474 (7)
C(61)—C(62)	1·475 (8)		

C(1)—O(1)—C(11)	116·8 (3)	C(2)—O(2)—C(21)	116·9 (4)
C(3)—O(3)—C(31)	117·9 (3)	C(4)—O(4)—C(41)	117·8 (3)
C(5)—O(5)—C(51)	117·5 (4)	C(6)—O(6)—C(61)	117·1 (4)
O(1)—C(1)—C(2)	109·3 (3)	O(2)—C(2)—C(1)	111·3 (3)
O(2)—C(2)—C(3)	109·3 (3)	C(1)—C(2)—C(3)	115·0 (4)
O(3)—C(3)—C(2)	111·8 (3)	O(3)—C(3)—C(4)	107·9 (3)
C(2)—C(3)—C(4)	116·7 (4)	O(4)—C(4)—C(3)	111·6 (3)
O(4)—C(4)—C(5)	103·9 (3)	C(3)—C(4)—C(5)	112·6 (4)
O(5)—C(5)—C(4)	106·5 (3)	O(5)—C(5)—C(6)	111 3 (4)
C(4)—C(5)—C(6)	113·7 (4)	O(6)—C(6)—C(5)	108·5 (4)
O(1)—C(11)—O(11)	123·9 (5)	O(1)—C(11)—C(12)	112·0 (4)
O(11)—C(11)—C(12)	124·1 (5)	O(2)—C(21)—O(21)	122·4 (5)
O(2)—C(21)—C(22)	111·2 (4)	O(21)—C(21)—C(22)	126·4 (5)
O(3)—C(31)—O(31)	122·7 (4)	O(3)—C(31)—C(32)	112·2 (4)
O(31)—C(31)—C(32)	125·1 (4)	O(4)—C(41)—O(41)	122·8 (4)
O(4)—C(41)—C(42)	111·9 (4)	O(41)—C(41)—C(42)	125·4 (5)
O(5)—C(51)—O(51)	121·7 (5)	O(5)—C(51)—C(52)	113·7 (4)
O(51)—C(51)—C(52)	124·6 (5)	O(6)—C(61)—O(61)	123·7 (5)
O(6)—C(61)—C(62)	110·7 (4)	O(61)—C(61)—C(62)	125·6 (5)

O(1)—C(1)—C(2)—C(3)	− 70·7 (5)	H(3)—C(3)—C(4)—H(4)	− 63 (1)
C(1)—C(2)—C(3)—C(4)	− 169·5 (5)	H(4)—C(4)—C(5)—H(5)	178 (1)
C(2)—C(3)—C(4)—C(5)	173·3 (4)	H(5)—C(5)—C(6)—H(61)	− 70 (1)
C(3)—C(4)—C(5)—C(6)	179·4 (4)	H(5)—C(5)—C(6)—H(62)	47 (1)
C(4)—C(5)—C(6)—O(6)	58·0 (6)	H(11)—C(1)—O(1)—C(11)	− 76 (1)
O(1)—C(1)—C(2)—O(2)	54·3 (5)	H(12)—C(1)—O(1)—C(11)	36 (1)
O(2)—C(2)—C(3)—O(3)	− 60·3 (4)	H(4)—C(4)—O(4)—C(41)	− 12 (1)
O(3)—C(3)—C(4)—O(4)	56·5 (4)	H(61)—C(6)—O(6)—C(61)	102 (1)
O(4)—C(4)—C(5)—O(5)	− 178·5 (3)	H(2)—C(2)—O(2)—C(21)	− 34 (1)
O(5)—C(5)—C(6)—O(6)	− 62·4 (5)	H(3)—C(3)—O(3)—C(31)	3 (1)
H(11)—C(1)—C(2)—H(2)	59 (1)	H(5)—C(5)—O(5)—C(51)	41 (1)
H(12)—C(1)—C(2)—H(2)	− 67 (1)	H(62)—C(6)—O(6)—C(61)	− 18 (1)
H(2)—C(2)—C(3)—H(3)	63 (1)		

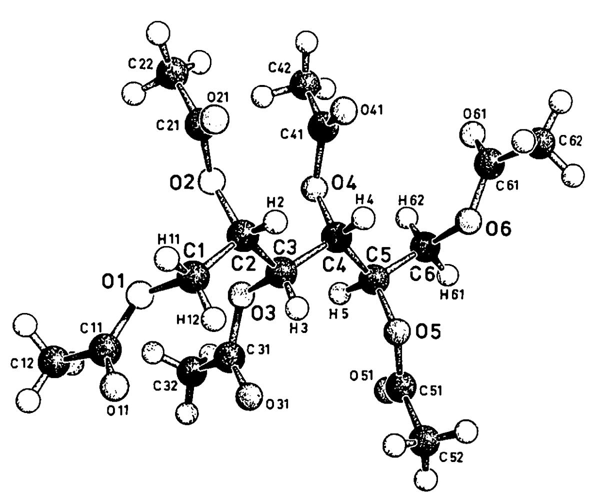

Fig. 1. *SCHAKAL*88 (Keller, 1986) drawing of the title compound. The numbering of the H atoms at the acetyl groups is not given, but is systematic. The H atom with lowest number is aligned parallel to the carbonyl O. The others follow clockwise, when looking in direction of the carbonyl C atom.

The molecules of the title compound are found in a planar zigzag conformation of the carbon chain. This situation was unexpected, because in this conformation a 1,3-parallel interaction between O(2) and O(4) has to be tolerated. The parent D-glucitol (*A* form) shows a sickle conformation and avoids such an unfavorable interaction by torsion around the C(2)—C(3) bond (Park, Jeffrey & Hamilton, 1971). Even more surprising is the fact that O(1) and O(3) are also found in such an O//O arrangement, because primary acetoxy groups have a much higher degree of conformational freedom than secondary ones. Therefore, the solid-state conformation of the title compound is an exceptionally dramatic example of a violation of the above-mentioned rule (Jeffrey, 1990). Distances O(1)—O(3) and O(2)—O(4) are 2·861 (4) and 2·893 (4) Å, respectively. The orientation of the acetoxy substituents is as usual, such that the carbonyl O atoms are more or less 1,3-parallel to the C—H bonds of the carbon chain (Table 2). No unusual features of the geometry of the acetoxy groups are observed.

The X-ray structure determination of the title compound strongly supports the previously reported evidence that the steric demands of acetylated hydroxyl groups are significantly lower than those of free ones (Lemieux & Pavia, 1969; Paulsen & Friedmann, 1972). This implies that the solid-state conformations of acetylated open-chain carbohydrates should be much less predictable than those of the parent compounds. This has been already realized for alditol acetate conformations in solution (Angyal & Le Fur, 1984).

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Structure of 3-Chlorotropolone

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Abstract. $C_7H_5ClO_2$, $M_r = 156·6$, monoclinic, $P2_1/c$, $a = 8·476$ (2), $b = 12·241$ (2), $c = 8·170$ (2) Å, $\beta = 126·47°$, $V = 681·7$ Å^3, $Z = 4$, $D_x = 1·525$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 44·63$ cm^{-1}, $F(000) = 320$, $T = 293$ K, $R = 0·046$ for 833 reflections with $I > 3\sigma(I)$. The hydroxylic proton forms a bifurcated hydrogen bond with carbonyl O atoms, one branch intramolecular and the other intermolecular. The latter intermolecular branches form a hydrogen-bonded dimer, which is roughly planar.

Introduction. The structures of tropolone and some tropolone derivatives have been studied. It has been shown that 5-isopropyltropolone (Berg, Karlasson, Pilotti & Wiehager, 1976) as well as tropolone (Shimanouchi & Sasada, 1973) forms a bifurcated hydrogen bond with the carbonyl O atoms, one branch being intramolecular and other intermolecular. On the other hand, 4-isopropyltropone does not form a dimer, but O···O hydrogen bonds of 2·8 Å link the molecules in a chain (Derry & Hamor, 1972).

Very recently, we have measured the electronic spectra of the isolated 3-chlorotropolone (Tsuji, Sekiya, Nishimura, Mori & Takeshita, 1991) and 3-bromotropolone (Sekiya, Sasaki, Nishimura, Mori & Takeshita, 1990). The observation of tunnel splitting provides conclusive evidence for the delocalization of the proton. We have found that the hydroxylic proton is delocalized in 3-chlorotropolone, whereas the proton is localized in 3-bromotropolone. It has been suggested that the localization of the hydroxylic proton strongly depends on the planarity of the molecule. In order to examine the effect of the substitution of a Cl atom on the molecular and crystal structure, an X-ray analysis has been performed for crystalline 3-chlorotropolone.

Experimental. 3-Chlorotropolone was synthesized following a known method (Nozoe, Seto, Ito, Sato & Katano, 1953). 3-Chlorotropolone crystals were prepared in a sealed Pyrex tube by heating at about 393 K with a coiled heater followed by gradual cooling. A crystal of 0·12 × 0·12 × 0·12 mm was sealed in a thin-walled Lindemann glass tube to minimize loss by sublimation. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation, lattice parameters from setting of 15 reflections with $13·52 \leq \theta \leq 17·57°$. ω–2θ scan technique used to collect intensities of 1107 independent reflections with $2 \leq \theta \leq 60°$ ($-9 \leq h \leq 9$, $0 \leq k \leq 13$, $0 \leq l \leq 9$), 833 of which were considered as observed [$I > 3\sigma(I)$]. Three standard reflections monitored every 3600 s, no significant variation in intensity during data collection; intensities not corrected for absorption. Structure solved by direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); H-atom positions determined by difference Fourier synthesis. Refinement by full-matrix least-squares method on F. C, O and Cl atoms anisotropic, H atoms isotropic and fixed at 4·0 Å^2. Final conventional $R = 0·046$, $wR = 0·084$, $w = 4F_o^2/[\sigma(F_o^2)]^2$; $(\Delta/\sigma)_{max}$ in final least-squares cycle 0·60; final difference Fourier height maximum (absolute value) 0·21 e Å^{-3}, refined secondary extinction value $g = 2·9931 \times 10^{-5}$. Atomic scattering factors from Cromer & Waber (1974). Computation on PDP11/23 computer using Enraf–Nonius *SDP-Plus* (Frenz, 1985) and *ORTEPII* (Johnson, 1976) programs.

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