with the coordination plane 3, so the whole ligand itself is not planar.

There are some differences in complex (II), and in each ligand the chelate (1 and 3 planes) and the naphthalene rings (2 and 4 planes) form an angle of about 10°. The angle between the two coordination planes is 153·9°.

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Structure of 2,4:3,5-Di-*O*-isopropylidene-D-mannitol

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Abstract. $C_{12}H_{22}O_6$, M_r = 262·302, monoclinic, $P2_1$, a = 11·389 (3), b = 11·617 (3), c = 12·061 (3) Å, β = 115·33 (2)°, V = 1442·3 Å^3, Z = 4, D_x = 1·208 g cm^{-3}, m.p. = 407–409 K, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·90 cm^{-1}, $F(000)$ = 568, T = 292 K, final R = 0·039 for 1458 observed reflections. There are no strong conformational differences between the two symmetry-independent molecules, the dioxane rings are all in the twist-boat conformation. Each of the molecules is nearly symmetric with respect to a pseudo-twofold axis perpendicular to the C(3)—C(4) bond. The molecules are linked into sheets parallel to crystal plane (100) by hydrogen bonds between the hydroxyl groups. There are four symmetry-independent hydrogen bonds in this structure [2·758 (8), 2·699 (5), 2·644 (7), 2·640 (8) Å] and they form one eight-membered ring. Intramolecular hydrogen bonds also occur between the hydroxyl groups and dioxane oxygens in accordance with the results of a ^{13}C NMR study of this compound in solution.

Introduction. 2,4:3,5-Di-*O*-isopropylidene-D-mannitol [hereinafter referred to as (I)] was obtained, together with the 2,3:4,5-derivative, from 1,6-di-*O*-benzoyl-D-mannitol by Gawrońska (1988). The structures of both were established by ^{13}C NMR spectroscopy. Small $\Delta\delta$ values, of 1 p.p.m., for the ^{13}C NMR reso-

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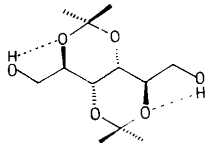
nances of the *gem*-dimethyl carbons of the 1,3-dioxane rings in (I) indicated a skew conformation of these rings, as a chair conformation would require $\Delta\delta$ of about 10 p.p.m. (Gawrońska, 1988; Buchman, Edgar, Rawson, Shahidi & Wightman, 1982). The IR spectra of a 10^{-3} *M* solution of (I) in CCl_4 showed a single band at 3602 cm^{-1} characteristic of an intramolecular hydrogen bond. An intramolecular hydrogen bond in a five-membered ring was postulated (see the scheme below), as the alternative hydrogen bond forming a six-membered ring would be disfavoured owing to electrostatic forces between the close C—O dipoles (Gawrońska, 1988).



The main aim of this X-ray study was to compare the information concerning the solution and crystalline states of (I), in particular the conformation of the 1,3-dioxane rings, the symmetry of the molecule in the crystal lattice and the hydrogen-bonding pattern.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for* (I)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}(Å^2)
Molecule *A*				
O(1)	0·1957 (5)	0·7668	1·0036 (4)	0·078
C(1)	0·2722 (7)	0·6702 (5)	1·0616 (5)	0·064
C(2)	0·2380 (6)	0·5642 (5)	0·9804 (5)	0·052
O(2)	0·2848 (4)	0·5857 (4)	0·8892 (3)	0·056
C(3)	0·3034 (6)	0·4578 (5)	1·0529 (5)	0·047
O(3)	0·2144 (4)	0·4100 (4)	1·0931 (4)	0·069
C(4)	0·3250 (6)	0·3731 (5)	0·9667 (5)	0·047
O(4)	0·2333 (4)	0·3907 (4)	0·8426 (3)	0·053
C(5)	0·3015 (6)	0·2485 (6)	0·9980 (5)	0·053
O(5)	0·3433 (4)	0·2436 (4)	1·1273 (3)	0·053
C(6)	0·3835 (7)	0·1635 (5)	0·9679 (5)	0·071
O(6)	0·3476 (5)	0·0454 (4)	0·9771 (4)	0·085
C(7)	0·2455 (7)	0·5008 (7)	0·7957 (5)	0·058
C(8)	0·1091 (6)	0·5270 (6)	0·6942 (5)	0·064
C(9)	0·3485 (6)	0·4997 (6)	0·7481 (5)	0·075
C(10)	0·2627 (7)	0·3085 (6)	1·1692 (6)	0·065
C(11)	0·1418 (7)	0·2417 (7)	1·1499 (6)	0·093
C(12)	0·3486 (7)	0·3392 (6)	1·3010 (5)	0·080
Molecule *B*				
O(1*B*)	0·2060 (6)	0·8910 (5)	0·8131 (4)	0·129
C(1*B*)	0·2586 (8)	0·8472 (7)	0·7388 (6)	0·099
C(2*B*)	0·2302 (7)	0·9232 (6)	0·6273 (5)	0·067
O(2*B*)	0·3122 (5)	1·0227 (4)	0·6733 (3)	0·071
C(3*B*)	0·2699 (7)	0·8658 (6)	0·5354 (6)	0·057
O(3*B*)	0·1591 (4)	0·8033 (4)	0·4525 (4)	0·063
C(4*B*)	0·3094 (6)	0·9546 (5)	0·4658 (5)	0·052
O(4*B*)	0·2466 (4)	1·0625 (3)	0·4639 (3)	0·052
C(5*B*)	0·2622 (6)	0·9191 (5)	0·3315 (5)	0·055
O(5*B*)	0·2744 (4)	0·7955 (4)	0·3320 (3)	0·064
C(6*B*)	0·3446 (7)	0·9693 (6)	0·2735 (6)	0·080
O(6*B*)	0·2809 (5)	0·9571 (4)	0·1436 (4)	0·112
C(7*B*)	0·2862 (7)	1·1134 (6)	0·5839 (5)	0·059
C(8*B*)	0·1727 (7)	1·1856 (7)	0·5746 (6)	0·084
C(9*B*)	0·4099 (6)	1·1801 (6)	0·6211 (5)	0·068
C(10*B*)	0·1818 (7)	0·7366 (6)	0·3629 (6)	0·066
C(11*B*)	0·0520 (6)	0·7228 (7)	0·2516 (5)	0·075
C(12*B*)	0·2444 (8)	0·6215 (6)	0·4153 (7)	0·091

Experimental. Colourless crystals of (I) were obtained by a slow evaporation of its ethanol solution. Dimensions of the crystal 0·3 × 0·3 × 0·3 mm. Refined unit-cell dimensions obtained from setting angles of 15 reflections with $8{\cdot}3 < \theta < 14{\cdot}2^\circ$. Syntex $P2_1$ diffractometer. 2θ–θ scan, Mo $K\alpha$ radiation (graphite monochromator). 1521 independent reflections ($2\theta < 45^\circ$), index range h 0/12, k 0/12, l −13/13; 1458 reflections considered observed [$I \geq 1{\cdot}96\sigma(I)$]; two control reflections monitored every 200 reflections showed no systematic or significant changes in net counts. Background and integrated intensity for each reflection was obtained by the profile analysis method of Lehmann & Larsen (1974); Lorentz and polarization corrections only. Structure solved routinely by direct methods. Absolute configuration of the crystal was assigned according to the chemical information. Non-H atoms were refined anisotropically, the positions of the carbon-H atoms were idealized after each cycle of least-squares refinement (C—H 1·06 Å) and assigned isotropic thermal factors, the hydroxyl-H atoms could not be located owing to the strong vibrations of the hydroxyl groups. Function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = \sigma^{-2}(F_o)$. Final $R = 0{\cdot}039$, $wR = 0{\cdot}039$, $S = 1{\cdot}4$, $(\Delta/\sigma)_{max} = 0{\cdot}02$; the highest and the lowest densities in the final ΔF map were 0·17 and −0·17 e Å^{-3}, respectively. The refined coordinates of the atoms are listed in Table 1.* Most of the calculations were performed with *SHELX76* (Sheldrick, 1976) on an IBM XT computer. Drawings from *PLUTO* (Motherwell & Clegg, 1978) and *ORTEP* (Johnson, 1965); atomic scattering factors were those incorporated in *SHELX76*.

Discussion. Fig. 1 shows the two symmetry-independent molecules of (I) as present in the crystal lattice. The molecular skeleton has an internal twofold axis perpendicular to the C(3)—C(4) bond; however no crystallographic twofold axis is present in space group $P2_1$. It must be noted that both molecules *A* and *B* have their pseudo-twofold axes similarly directed perpendicular to the (100) plane (see Fig. 2). There are no significant differences in the corresponding bond lengths and valency angles of molecules *A* and *B* or between the values related by the pseudo-symmetry in each of the molecules, except for differences in the bond lengths of the

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* Lists of anisotropic thermal parameters, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54395 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

terminal hydroxyl groups, which may result from errors caused by their vibrations (Table 2). The two independent molecules also have a very similar conformation. An analogous conformation and molecular symmetry of the C-atom chain are observed in the molecules of D-mannitol. D-Mannitol crystallizes in several forms, but the general molecular geometry is the same: the molecules have a planar zigzag chain of C atoms with a pseudo-twofold symmetry axis, except for the *α* form in which the molecules have exact twofold symmetry as a crystallographic requirement (Berman, Jeffrey & Rosenstein, 1968; Kim, Jeffrey & Rosenstein, 1968). A planar zigzag chain was also observed in galactitol (Berman & Rosenstein, 1968). In the molecules of (I) the C-atom chain is not planar owing to the strains caused by the dioxane rings, but the pseudo-twofold symmetry of the chain and of the molecule is maintained. The 1,3-dioxane rings are all in the flexible twist-boat conformation, with very small differences between the corresponding torsion angles (Table 3). An analysis of the possible conformations of the flexible molecule skeleton using a Dreiding model indicates that the conformation present in the crystal is the only conformation of (I) which avoids steric hindrances: between the methyl groups [C(8), C(9), C(10), C(11)] and the H atoms at C(3) and C(4), between the methyl groups and the H atoms at methylenes C(1) and C(6), and between the methyl groups themselves. Thus, the conformation of the flexible skeleton is maintained by these steric hindrances, rather than frozen by intermolecular forces in the crystal lattice. This is confirmed by the unusually small differences in conformation of the two symmetry-independent molecules. It indicates that this conformation of (I) should be also present in solution, which is supported by the ^{13}C NMR results (Gawrońska, 1988).

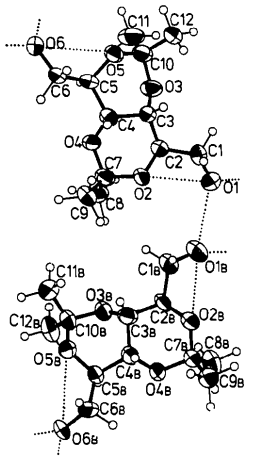

Fig. 1. Two symmetry-independent molecules in (I). The thermal ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles, the hydroxyl-H atoms were not located and are not included in this drawing. Hydrogen bonds are represented as dotted lines.

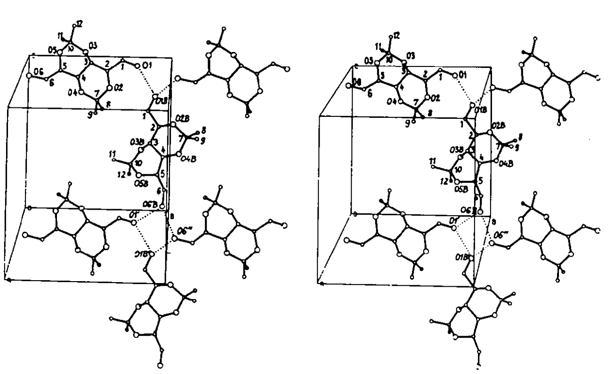

Fig. 2. Arrangement of the molecules in the crystal lattice viewed down **a**. In this drawing only half of the unit-cell contents have been plotted to avoid overlap of the molecules, H atoms have been omitted for clarity. The intermolecular hydrogen bonds are shown as dotted lines.

Table 2. *Bond lengths* (Å) *and valency angles* (°) *for* (I)

	Molecule *A*	Molecule *B*
O(1)—C(1)	1·408 (6)	1·371 (12)
C(1)—C(2)	1·519 (8)	1·524 (10)
C(2)—O(2)	1·434 (9)	1·440 (8)
C(2)—C(3)	1·513 (8)	1·518 (12)
O(2)—C(7)	1·419 (8)	1·446 (8)
C(3)—O(3)	1·411 (9)	1·428 (7)
C(3)—C(4)	1·527 (9)	1·515 (10)
O(3)—C(10)	1·451 (8)	1·440 (10)
C(4)—O(4)	1·426 (6)	1·438 (7)
C(4)—C(5)	1·548 (9)	1·531 (8)
O(4)—C(7)	1·430 (9)	1·446 (7)
C(5)—O(5)	1·425 (7)	1·443 (8)
C(5)—C(6)	1·507 (10)	1·508 (12)
O(5)—C(10)	1·435 (10)	1·434 (10)
C(6)—O(6)	1·451 (8)	1·424 (8)
C(7)—C(8)	1·542 (8)	1·505 (11)
C(7)—C(9)	1·510 (12)	1·498 (10)
C(10)—C(11)	1·509 (11)	1·523 (8)
C(10)—C(12)	1·510 (8)	1·520 (10)
O(1)—C(1)—C(2)	113·2 (5)	112·2 (7)
C(1)—C(2)—C(3)	111·4 (5)	112·0 (6)
C(1)—C(2)—O(2)	106·2 (5)	105·5 (5)
O(2)—C(2)—C(3)	109·6 (5)	106·8 (6)
C(2)—O(2)—C(7)	113·3 (5)	114·1 (4)
C(2)—C(3)—C(4)	108·5 (5)	110·9 (6)
C(2)—C(3)—O(3)	105·9 (5)	106·6 (6)
O(3)—C(3)—C(4)	109·3 (5)	110·0 (5)
C(3)—O(3)—C(10)	113·9 (5)	114·3 (6)
C(3)—C(4)—C(5)	110·0 (5)	110·5 (5)
C(3)—C(4)—O(4)	111·3 (5)	110·0 (5)
O(4)—C(4)—C(5)	105·4 (5)	105·8 (5)
C(4)—O(4)—C(7)	113·2 (5)	113·6 (4)
C(4)—C(5)—C(6)	111·4 (6)	112·4 (5)
C(4)—C(5)—O(5)	107·1 (5)	106·0 (4)
O(5)—C(5)—C(6)	106·7 (5)	108·1 (5)
C(5)—O(5)—C(10)	113·7 (5)	113·4 (5)
C(5)—C(6)—O(6)	112·3 (6)	110·3 (6)
O(2)—C(7)—O(4)	111·0 (4)	109·0 (5)
O(4)—C(7)—C(9)	111·5 (6)	111·3 (5)
O(4)—C(7)—C(8)	104·8 (6)	105·4 (5)
O(2)—C(7)—C(9)	106·1 (5)	106·8 (5)
O(2)—C(7)—C(8)	111·2 (6)	110·9 (6)
C(8)—C(7)—C(9)	112·6 (5)	113·7 (6)
O(3)—C(10)—O(5)	109·3 (5)	109·1 (5)
O(5)—C(10)—C(12)	105·8 (6)	105·2 (7)
O(5)—C(10)—C(11)	110·8 (6)	111·5 (5)
O(3)—C(10)—C(12)	112·0 (6)	111·5 (6)
O(3)—C(10)—C(11)	104·3 (6)	107·5 (6)
C(11)—C(10)—C(12)	114·9 (6)	112·4 (6)

Table 3. *Selected torsion angles* (°) *for* (I)

	Molecule *A*	Molecule *B*
D-Mannitol chain		
O(1)—C(1)—C(2)—C(3)	−168·6 (10)	−170·6 (11)
C(1)—C(2)—C(3)—C(4)	−151·0 (10)	−149·4 (11)
C(2)—C(3)—C(4)—C(5)	−141·6 (10)	−141·3 (10)
C(3)—C(4)—C(5)—C(6)	−149·8 (10)	−153·5 (10)
C(4)—C(5)—C(6)—O(6)	−169·9 (10)	−165·8 (10)
2,4-Dioxane ring		
C(2)—C(3)—C(4)—O(4)	−25·3 (11)	−25·1 (12)
C(3)—C(2)—O(2)—C(7)	68·1 (11)	69·6 (11)
C(2)—O(2)—C(7)—O(4)	−30·9 (12)	−33·1 (12)
O(2)—C(2)—C(3)—C(4)	−34·0 (11)	−34·4 (12)
C(3)—C(4)—O(4)—C(7)	64·4 (11)	64·9 (11)
C(4)—O(4)—C(7)—O(2)	−33·2 (12)	−33·3 (11)
3,5-Dioxane ring		
O(3)—C(3)—C(4)—C(5)	−26·6 (11)	−23·7 (12)
C(4)—C(3)—O(3)—C(10)	66·3 (11)	64·2 (12)
C(3)—O(3)—C(10)—O(5)	−34·3 (12)	−32·6 (12)
C(3)—C(4)—C(5)—O(5)	−33·6 (11)	−35·8 (11)
C(4)—C(5)—O(5)—C(10)	69·2 (11)	71·2 (11)
C(5)—O(5)—C(10)—O(3)	−33·6 (12)	−34·6 (12)

While it was evident from IR spectra that in the solution there is an intramolecular hydrogen bond enclosing a five-membered ring (Gawrońska, 1988), several patterns of hydrogen bonding were possible in the crystal. The molecular packing in the crystal lattice is shown in Fig. 2. Table 4 lists the shortest intra- and intermolecular contacts of the non-H atoms. Each of the four hydroxyl groups act as a donor and as an acceptor in two intermolecular, and as a donor in one intramolecular, hydrogen bond. Although the H atoms at the hydroxyl groups could not be located, it appears that the five-membered rings observed in solution are still preserved in the crystal. This is deduced from short O(1)···O(2) and O(5)···O(6) intramolecular distances of 2·9 Å in molecules *A* and *B* – see Table 4. The shortest are the intermolecular hydrogen bonds which link the molecules into sheets along (100) and enclose an eight-membered ring, this pattern often being observed in crystals where hydroxyl groups form the hydrogen bonds linking the molecules [*e.g.* in the pentaerythritol crystal (Eilerman & Rudman, 1979)]. The pattern of the hydrogen bonds in (I) is indicated in Figs. 1 and 2. No other intramolecular contacts shorter than 3·40 Å between non-H atoms are present in this structure.

Table 4. *Selected intramolecular distances between* O *atoms and intermolecular distances shorter than* 3·5 Å *between the non-*H *atoms for* (I)

Intramolecular distances			
O(1)···O(2)	2·926 (6)	O(1*B*)···O(2*B*)	2·896 (8)
O(3)···O(4)	3·128 (7)	O(3*B*)···O(4*B*)	3·157 (6)
O(5)···O(6)	2·945 (7)	O(5*B*)···O(6*B*)	2·973 (7)
Intermolecular contacts			
O(1)···O(1*B*)	2·758 (8)	O(5)···O($6B^{ii}$)	3·427 (7)
O(1)···O($6B^{i}$)	2·699 (5)	O(6)···O($1B^{iii}$)	2·644 (7)
O(2)···O(1*B*)	3·485 (9)	O(6)···O($6B^{ii}$)	2·640 (8)

Symmetry code: (i) $x, y, z+1$; (ii) $x, y-1, z+1$; (iii) $x, y-1, z$.

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Structure of Photostable 1-(Hydroxyalkyl)-2(1*H*)-pyrazinones

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Abstract. 1-(2-Hydroxyethyl)-5,6-diphenyl-2(1*H*)-pyrazinone (I), $C_{18}H_{16}N_2O_2$, $M_r = 292.32$, orthorhombic, *Pbca*, $a = 13.320$ (4), $b = 31.654$ (15), $c = 6.991$ (3) Å, $V = 2948$ (2) Å^3, $Z = 8$, $D_x = 1.317$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71068 Å, $\mu = 0.81$ cm^{-1}, $F(000) = 1232$, $T = 294$ K, $R = 0.057$ for

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