

Fig. 2. Unit-cell contents projected down *b*. Hydrogen bonds are indicated by dashed lines.

hydrogen bonding. The O...N separation compares well with those observed for compounds with similar molecular fragments. The C=O...H and O...H—N angles are typical at 122 (1) and 172 (3)°. Contact between dimers corresponds to normal van der Waals interactions.

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2,3:5,6-Di-*O*-isopropylidene- α -D-mannofuranose, C₁₂H₂₀O₆

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Abstract. $M_r = 260.29$, orthorhombic, $P2_12_12_1$, $a = 6.665$ (1), $b = 10.816$ (1), $c = 18.891$ (3) Å, $V = 1362$ Å³, $Z = 4$, $D_x = 1.269$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.766$ mm⁻¹, $F(000) = 560$, $T = 293$ K,

final $R = 0.0437$ for 1353 unique observed reflections. The fused five-membered rings have envelope configurations with maximum distances from the best planes of -0.205 (4) Å [C(7)] and 0.247 (2) Å [O(4)]

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and the angle between the planes is 115.9 (4)°. The only hydrogen bond [O(1) to O(5')] has an O...O distance of 3.014 (7) Å.

Introduction. The compound was prepared by refluxing D-mannose in acetone containing an acidic catalyst (conc. HCl), for comparison with 5-hydroxy-1,2-*O*-isopropylidene- α -D-xylo-hexofuranurono-6,3-lactone (Sheldrick, Mackie & Akrigg, 1983) and for investigation of the single hydrogen bond.

Experimental. Material prepared by WM, tabular, 0.32 × 0.26 × 0.14 mm, Enraf-Nonius CAD-4F diffractometer, Ni-filtered Cu K α ; cell parameters from 40 θ measurements in range 18–29°; reflections measured for four octants to 2 θ = 140° for the ranges of *h*, *k* and *l* of –8 to 8, 0 to 13 and –23 to 23, respectively, 3887 reflections measured plus 1563 flagged as <0; intensity control (84 measurements): average count of 1375 with a standard deviation (of the distribution) = 54 (3.9%) and no systematic trend; no absorption correction; data merged using *SHELX76* (Sheldrick, 1976) to give 1357 unique reflections with R_{int} = 0.02; *h*, *k*, *l* range 0 to 8, 13, 22; four reflections with high F_c/F_o ratios, possibly due to extinction, removed; structure solved by direct methods with *MULTAN80* (Main *et al.*, 1980), non-H atoms refined by least squares (F magnitudes) with anisotropic thermal parameters; H atoms found from difference Fourier syntheses and refined isotropically; R = 0.0437, wR = 0.0515; for final cycle, maximum shift/error = 0.032, average = 0.006; $w = 0.6255[\sigma^2(F) + 0.000885F^2]^{-1}$; difference Fourier synthesis showed a maximum value of 0.13 e Å⁻³ and a minimum of –0.12 e Å⁻³; atom scattering factors from *International Tables for X-ray Crystallography* (1974).

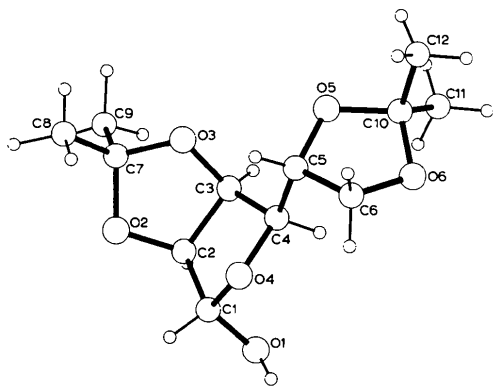


Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO78* (Motherwell & Clegg, 1978).

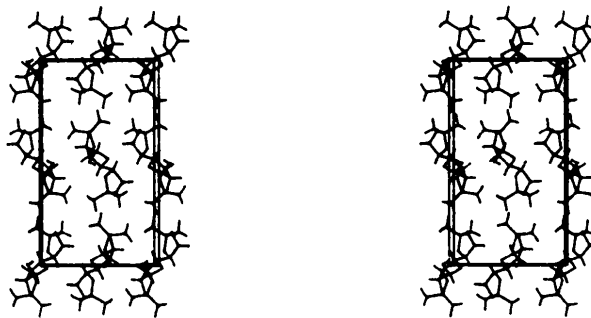


Fig. 2. Stereoscopic view of the crystal structure along *a*, with *b* horizontal and *c* vertical. The origin is at the bottom left-hand corner.

Table 1. Atom coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$)

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	–685 (5)	–373 (3)	–524 (2)	792
C(2)	991 (5)	–1221 (3)	–772 (2)	758
C(3)	2789 (4)	–873 (3)	–307 (2)	724
C(4)	1943 (4)	127 (3)	182 (2)	685
C(5)	3371 (5)	1152 (3)	375 (2)	739
C(6)	2580 (6)	1982 (4)	945 (2)	860
C(7)	3753 (5)	–820 (3)	–1469 (2)	809
C(8)	4395 (8)	118 (5)	–2005 (3)	984
C(9)	4667 (9)	–2074 (5)	–1589 (3)	1083
C(10)	5023 (5)	820 (4)	1445 (2)	854
C(11)	5165 (9)	–385 (5)	1836 (3)	1115
C(12)	6669 (10)	1745 (8)	1629 (4)	1251
O(1)	–1859 (4)	–1027 (2)	–33 (2)	911
O(2)	1597 (3)	–893 (3)	–1469 (1)	910
O(3)	4208 (3)	–366 (2)	–784 (1)	779
O(4)	290 (3)	643 (2)	–205 (1)	747
O(5)	5110 (3)	604 (2)	694 (1)	850
O(6)	3121 (4)	1343 (2)	1574 (1)	859

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

C(1)–C(2)	1.520 (5)	C(1)–O(1)	1.403 (4)
C(2)–C(3)	1.533 (4)	C(1)–O(4)	1.412 (4)
C(3)–C(4)	1.529 (5)	C(4)–O(4)	1.435 (3)
C(4)–C(5)	1.506 (4)	C(2)–O(2)	1.422 (4)
C(5)–C(6)	1.497 (5)	C(7)–O(2)	1.439 (4)
C(7)–C(8)	1.495 (6)	C(3)–O(3)	1.416 (4)
C(7)–C(9)	1.504 (6)	C(7)–O(3)	1.419 (4)
C(10)–C(11)	1.500 (6)	C(5)–O(5)	1.434 (4)
C(10)–C(12)	1.525 (6)	C(10)–O(5)	1.440 (4)
		C(6)–O(6)	1.422 (5)
		C(10)–O(6)	1.410 (4)
O(1)–C(1)–C(2)	108.1 (3)	C(8)–C(7)–C(9)	113.3 (4)
O(1)–C(1)–O(4)	111.5 (3)	C(8)–C(7)–O(2)	108.8 (3)
C(2)–C(1)–O(4)	105.3 (2)	C(8)–C(7)–O(3)	108.8 (3)
C(1)–C(2)–C(3)	104.4 (3)	C(9)–C(7)–O(2)	110.8 (4)
C(1)–C(2)–O(2)	110.2 (3)	C(9)–C(7)–O(3)	111.2 (4)
C(3)–C(2)–O(2)	104.3 (2)	O(2)–C(7)–O(3)	103.4 (3)
C(2)–C(3)–C(4)	103.4 (2)	C(11)–C(10)–C(12)	114.4 (4)
C(2)–C(3)–O(3)	104.6 (2)	C(11)–C(10)–O(5)	110.0 (3)
C(4)–C(3)–O(3)	110.9 (3)	C(11)–C(10)–O(6)	108.7 (3)
C(3)–C(4)–C(5)	115.7 (2)	C(12)–C(10)–O(5)	107.6 (4)
C(3)–C(4)–O(4)	104.6 (3)	C(12)–C(10)–O(6)	110.2 (4)
C(5)–C(4)–O(4)	108.8 (2)	O(5)–C(10)–O(6)	105.8 (3)
C(4)–C(5)–C(6)	113.1 (3)	C(2)–O(2)–C(7)	107.3 (2)
C(4)–C(5)–O(5)	108.0 (3)	C(3)–O(3)–C(7)	107.7 (2)
C(6)–C(5)–O(5)	103.3 (3)	C(1)–O(4)–C(4)	105.5 (2)
C(5)–C(6)–O(6)	102.8 (3)	C(5)–O(5)–C(10)	108.3 (3)
		C(6)–O(6)–C(10)	106.2 (3)

Discussion. Fig. 1 shows the molecule and numbering scheme and Fig. 2 a stereoscopic view of the unit cell. Table 1* lists atom parameters; Table 2 gives bond distances and angles.

The fused five-membered rings, C(2)–C(3)–O(3)–C(7)–O(2) and C(2)–C(3)–C(4)–O(4)–C(1), both have the envelope conformation with maximum distances from the best least-squares plane for each ring of -0.205 (4) Å for C(7) and 0.247 (2) Å for O(4), while the largest deviation from the best plane in the third ring, C(5)–C(6)–O(6)–C(10)–O(5), is 0.210 (4) Å for C(6). The obtuse angle between the fused rings is 115.9 (4)°, calculated as the angle between the best planes formed by C(2), C(3) and the atoms bonded directly to them [*cf.* 5-hydroxy-1,2-*O*-isopropylidene- α -D-xylo-hexofuranurono-6,3-lactone (Sheldrick *et al.*, 1983)]. The single hydroxyl group forms a weak

hydrogen bond, O(1) to O(5)($x-1, y, z$) with an O...O distance of 3.014 (7) Å, linking the molecules in the *a*-axis direction only.

C–O bond distances in the isopropylidene rings are nearly equal, only O(2)–C(7), at 1.439 (4) Å, having a value greater than 3σ from the average.

We wish to thank the University Computing Service of the University of Leeds for the provision of facilities.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and least-squares-planes' details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39870 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Diterpene from the *Austroepatorium inulaefolia* (HBK) K et R: 2,18-Diacetoxy-15,16-epoxy-3-hydroxy-12-oxo-19-norlabdane-8(17),13(16),14-triene Monohydrate, C₂₃H₃₀O₇·H₂O

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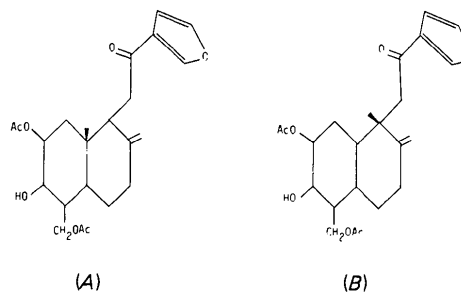
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Abstract. $M_r = 436.50$, orthorhombic, $P2_12_12_1$, $a = 14.076$ (3), $b = 21.088$ (5), $c = 7.822$ (2) Å, $V = 2321.8$ Å³, $Z = 4$, $D_x = 1.249$, $D_m = 1.246$ (5) g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.54$ cm⁻¹, $F(000) = 936$, $T = 296$ K. Final $R = 0.067$ for 1235 unique observed reflections. The crystalline structure consists of discrete molecules situated almost parallel to the *xz* plane forming layers at $y = 0$ and $\frac{1}{2}$, and channels running parallel to *c* where the water molecules are trapped by hydrogen bonds with three different diterpene molecules. The bicyclic system has the chair conformation with all substituents in *cis* positions.

Introduction. *Austroepatorium inulaefolia* (HBK) K et R was found to be rich in a new series of diterpenes

with norlabdane skeletons (Bahsas, 1979). Chemical and spectroscopic studies suggested two possible structures (*A* and *B*) for one of these compounds. The present crystallographic study was undertaken to provide information on its structure and to determine if structure *B* was present.



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