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5-Hydroxy-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranurono-6,3-lactone, $C_9H_{12}O_7$

By B. Sheldrick, W. Mackie and D. Akrigg

Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England

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Abstract. $M_r = 232{\cdot}19$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}621\,(1)$, $b = 11{\cdot}826\,(2)$, $c = 15{\cdot}598\,(2)$ Å, $V = 1036{\cdot}86$ Å^3, $Z = 4$, $D_x = 1{\cdot}49$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\,K\alpha) = 1{\cdot}024$ mm^{-1}, $F(000) = 488$, $T = 293$ K, final $R = 0{\cdot}0683$ for 841 unique observed reflections. Both hydroxyl groups of the *gem* diol show characteristic short O—H bond lengths, and both appear to be involved in hydrogen bonding.

Introduction. The title compound was prepared by Mackie & Perlin (1965) and NMR studies indicated the probable structure to be a *gem* diol. This investigation confirms the structure.

Experimental. Material prepared as described by Mackie & Perlin (1965), recrystallized from water/2-propanol, rectangular prism $0{\cdot}24 \times 0{\cdot}15 \times 0{\cdot}12$ mm, Enraf–Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; cell parameters from 73 θ measurements; reflections measured for four octants to $2\theta = 140°$ for the ranges of h, k and l of 0 to 6, −14 to 14 and −19 to 19 respectively, 4262 reflections measured; $01\bar{5}$ used as intensity control (74 measurements): average count of 10297 distributed with a standard deviation of 342·6 (= 3·3%) and no systematic trend; no absorption correction; data merged using *SHELX* (Sheldrick, 1976) to give 1021 unique reflections with a merging $R_{\mathrm{int}} = 0{\cdot}06$; 8 reflections with high F_c/F_o ratios, possibly due to extinction, removed and 172 reflections eliminated with $F < 3\sigma(F)$; structure solved with *MULTAN80* (Main *et al.*, 1980), non-H atoms refined by least squares, first with isotropic thermal parameters, then anisotropic; H atoms of the methyl groups calculated at each stage, the remaining six H atoms found from a difference Fourier synthesis and refined with thermal-vibration parameters set at the equivalent U_{iso} of the attached atom; for final cycle, maximum shift/error 0·021, average 0·003; unit weights; difference Fourier synthesis showed a maximum value of 0·23 e Å^{-3} and a minimum of −0·26 e Å^{-3}; atomic scattering factors from *MULTAN80* and *SHELX*.

Discussion. Fig. 1 shows the molecule and numbering scheme and Fig. 2 is an a-axis projection of the unit cell. Table 1* lists the atom coordinates and equivalent U_{iso}'s; Table 2 gives bond distances and angles.

The structure shows that the compound is a *gem* diol and not some form of hydrate. Both hydroxyl groups have short O—H bond distances [0·634 (77) and 0·694 (75) Å], as do most *gem* diols, *e.g.* 5,5-dihydroxybarbituric acid (Singh, 1965), ninhydrin (Medrud, 1969) and tetrahydrofuran-3,3,4,4-tetrol (Mighell & Jacobson, 1964). Both form hydrogen bonds, O(6) to O(4′)($2-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) and O(7) to O(5′)($x+1$, y, z), with distances of 2·897 (13) and 2·791 (13) Å.

There are small differences in the obtuse angles between the two outer *trans* rings and the central ring,

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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 38618 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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112·1 (0·86)° [O(2)—C(3)—C(7)—C(6) to O(1)—C(7)—C(3)—C(2)] and 116·0 (1·06)° [O(2)—C(4)—C(6)—C(7) to O(3)—C(4)—C(6)—O(4)], compared with 110·5 and 114·2° respectively for 1,2-*O*-isopropylidene-α-D-glucofuranurono-6,3-lactone (Sheldrick, Mackie & Akrigg, 1983).

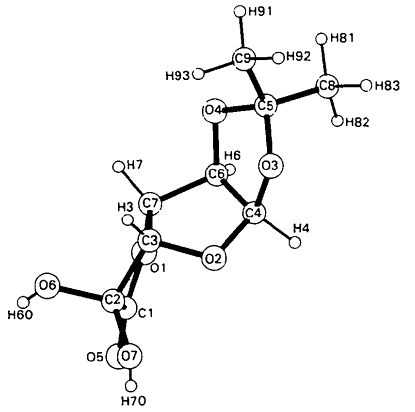

Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO*78 (Motherwell, 1978).

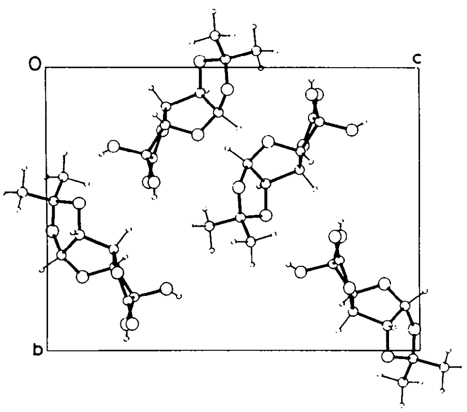

Fig. 2. Projection of the crystal structure along **a** (*PLUTO*78).

Table 1. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^4)

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

	x	y	z	U_{eq}
O(1)	7546 (9)	2244 (4)	3117 (4)	595
O(2)	12138 (10)	2380 (4)	4052 (3)	601
O(3)	12874 (11)	772 (5)	4856 (4)	701
O(4)	10119 (11)	−218 (4)	4122 (3)	590
O(5)	7223 (10)	4050 (5)	2729 (4)	634
O(6)	11398 (12)	2788 (5)	1808 (4)	649
O(7)	12274 (11)	4058 (5)	2870 (4)	629
C(1)	8429 (14)	3215 (7)	2845 (5)	523
C(2)	11118 (13)	3083 (6)	2677 (6)	494
C(3)	11716 (14)	2041 (7)	3191 (5)	531
C(4)	11111 (14)	1597 (6)	4615 (5)	544
C(5)	11842 (17)	−293 (7)	4812 (5)	614
C(6)	9267 (14)	901 (7)	4123 (5)	541
C(7)	9353 (14)	1386 (7)	3218 (5)	557
C(8)	10563 (22)	−598 (7)	5641 (5)	855
C(9)	13703 (20)	−1121 (10)	4538 (7)	1056

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

C(1)—C(2)	1·542 (11)	C(2)—O(6)	1·409 (10)
C(2)—C(3)	1·508 (11)	C(2)—O(7)	1·358 (9)
C(3)—C(7)	1·538 (11)	C(3)—O(2)	1·422 (9)
C(4)—C(6)	1·530 (10)	C(4)—O(2)	1·401 (9)
C(6)—C(7)	1·525 (11)	C(4)—O(3)	1·441 (9)
C(5)—C(8)	1·523 (12)	C(5)—O(3)	1·388 (10)
C(5)—C(9)	1·495 (13)	C(5)—O(4)	1·450 (9)
C(1)—O(1)	1·321 (9)	C(6)—O(4)	1·407 (10)
C(1)—O(5)	1·211 (9)	C(7)—O(1)	1·444 (9)

O(1)—C(1)—C(2)	109·6 (7)	O(3)—C(5)—O(4)	105·1 (6)
O(1)—C(1)—O(5)	123·1 (7)	O(3)—C(5)—C(8)	111·7 (7)
C(2)—C(1)—O(5)	127·3 (8)	O(3)—C(5)—C(9)	108·5 (8)
C(1)—C(2)—C(3)	102·2 (7)	C(8)—C(5)—C(9)	114·7 (8)
C(1)—C(2)—O(6)	107·3 (7)	C(8)—C(5)—O(4)	109·2 (7)
C(1)—C(2)—O(7)	110·3 (6)	C(9)—C(5)—O(4)	107·2 (7)
O(6)—C(2)—C(3)	106·5 (6)	O(4)—C(6)—C(7)	110·1 (7)
O(6)—C(2)—O(7)	111·7 (7)	C(4)—C(6)—O(4)	106·0 (6)
O(7)—C(2)—C(3)	118·0 (7)	C(4)—C(6)—C(7)	103·9 (7)
C(2)—C(3)—O(2)	107·9 (6)	C(6)—C(7)—C(3)	104·0 (7)
C(2)—C(3)—C(7)	103·5 (6)	C(6)—C(7)—O(1)	110·1 (7)
C(7)—C(3)—O(2)	105·1 (7)	C(3)—C(7)—O(1)	104·5 (6)
O(2)—C(4)—O(3)	109·2 (6)	C(1)—O(1)—C(7)	112·4 (6)
O(2)—C(4)—C(6)	108·8 (6)	C(3)—O(2)—C(4)	109·6 (6)
C(6)—C(4)—O(3)	103·5 (6)	C(4)—O(3)—C(5)	108·3 (6)
		C(5)—O(4)—C(6)	106·5 (6)

The most pronounced change is shown by the bonds between O(3) and the attached C atoms, C(4) and C(5). Bond O(3)—C(4) has increased from 1·382 (7) to 1·441 (9) Å while O(3)—C(5) has dropped from 1·486 (8) to 1·388 (10) Å and the ring angles at O(3) and O(4) have decreased by 2·4 (5) and 3·9 (5)° respectively. Comparison with similar structures where an isopropylidene ring is fused to a furanose ring, using data extracted from the Cambridge Crystallographic Data File (Kennard, Watson, Allen, Motherwell, Town & Rogers, 1975), shows that, of 21 fragments published, 15 have the bond distance O(3)—C(5) > O(3)—C(4) and the remaining 6 have the reverse. In addition, the bond distance C(4)—O(2) resembles O(3)—C(5) in that it too is shorter than O(3)—C(4) in the same 6 structures and longer in the other 15. It is difficult to establish if this effect is due to ring substituents but, for the present case, the *gem* diol group, in which the O atoms have a strong attraction for their connected H atoms, may affect the electron distribution along the chain of atoms C(2)—C(3)—O(2)—C(4)—O(3)—C(5).

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Structure of 9-Annulene Derivatives. 1. 5,13-Diphenyl-8*H*-cyclonona[*b*]naphthalene, $C_{29}H_{22}$

BY MAZHAR-UL-HAQUE,* JAMIL AHMED AND WILLIAM HORNE

Department of Chemistry, University of Petroleum and Minerals, Dhahran, Saudi Arabia

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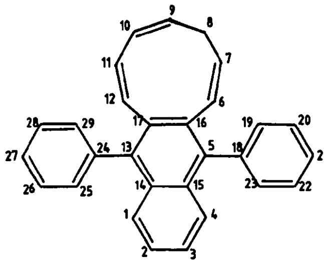
Abstract. $M_r = 370{\cdot}5$, monoclinic, space group $C2/c$, $a = 13{\cdot}468\ (17)$, $b = 22{\cdot}137\ (5)$, $c = 13{\cdot}859\ (2)$ Å, $\beta = 92{\cdot}22\ (4)°$, $U = 4128{\cdot}8$ Å^3, $Z = 8$, $D_x = 1{\cdot}19$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}073$ mm^{-1}, $F(000) = 1568$, $T = 295$ K. Final $R = 0{\cdot}047$ for 2120 observed reflections. The nine-membered ring adopts a distorted tub conformation. There is evidence of steric overcrowding. The molecules are packed by van der Waals interactions.

Introduction. Cyclononatetraene, the parent 9-annulene, is an unstable, non-planar, anti-aromatic molecule which readily undergoes valence isomerization to *cis*-8,9-dihydroindene at temperatures above 200 K (Anastassiou, Orfanos & Gebrian, 1969; Baldwin, 1972). Several subsequent investigations have demonstrated that benzene or naphthalene annelation of cyclononatetraene inhibits the isomerization reaction to the extent that solid derivatives stable at room temperature have been prepared (Anastassiou, Libsch & Griffith, 1973; Anastassiou, Kasmai & Sabahi, 1979).

Until now, no solid-state structural studies have been published on these benzene- and naphthalene-annelated cyclononatetraenes; indeed, the only 9-annulene derivative to have been previously investigated, 4-(diethylamino)-1,10-ethano-5-methylcyclopentacyclononene (Lindner, Kitschke, Hafner & Ude, 1980, involves a fused tricyclic ring system with stereochemical constraints markedly different from those of the parent annulene.

The present paper reports results obtained by an X-ray study on a representative naphthalene-annelated cyclononatetraene (I).



(I)

Experimental. Suitable crystals prepared by isothermal concentration of an ether solution of (I). Crystal dimensions 0·30 × 0·35 × 0·90 mm. Enraf–Nonius CAD-4 diffractometer, take-off angle 5°, graphite-crystal-monochromatized Mo $K\alpha$ radiation. Cell dimensions obtained by least-squares refinement of setting angles of 25 accurately centred large-angle reflections. 3543 independent reflections (to $\theta < 25°$) measured using ω–2θ scans, background scans being taken on each side of peaks. Range of hkl: −15–16, 0–22, 0–16. Intensities of three standard reflections showed no significant variation during data collection. Automatic multisolution routine of *SHELX76* (Sheldrick, 1976) used to generate a series of E maps, one of which correctly located all non-H atomic positions. 2120 reflections considered observed, having $I \geq 3\ \sigma(I)$, only these used in subsequent steps. $R_{\text{int}} = 0{\cdot}004$. Three cycles of full-matrix least-squares refinement with isotropic temperature factors gave $R = 0{\cdot}127$, incorporation of anisotropic temperature factors reduced R to 0·093. All H atoms located at this stage from a difference map and further refinement of non-H atoms anisotropically and H atoms isotropically reduced R to 0·047. Weighting scheme based on counting statistics was used, $w = 3{\cdot}0/[\sigma^2|F_o| + 0{\cdot}0003F_o^2]$, minimizing $\sum w\Delta F^2$; $R_w = 0{\cdot}050$, $S = 3{\cdot}9$, $\Delta/\sigma = 0{\cdot}12$. Final difference map showed no peaks or depressions larger than $0{\cdot}27$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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* To whom correspondence should be addressed.

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