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Methyl α-D-Lyxopyranoside

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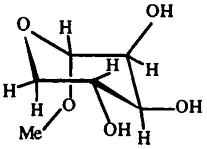
Abstract

The structure of the title compound, $C_6H_{12}O_5$, solved by direct methods from X-ray data collected at low temperature, shows the classical chair conformation of a deoxy sugar.

Comment

Methyl α-D-lyxopyranoside, (I), represents one of the few members of the family of simple pyranosides whose structure at atomic resolution has not yet been described; the determination of its structure was undertaken as part of a wider study of the affinity of carbohydrates to lectins (Evdokimov, Gilboa & Frolow, 1996).



(I)

Although the structures of β-L-lyxopyranose (neutron study: Nordenson, Takagi & Jeffrey, 1978), methyl α-D-lyxofuranoside (Groth & Hammer, 1968) and tri-O-acetyl-α-D-lyxopyranose (Herpin, Famery, Auge & Davido, 1976) have been reported, the absence of a determination for (I) may be attributed to its reluctance to crystallize.

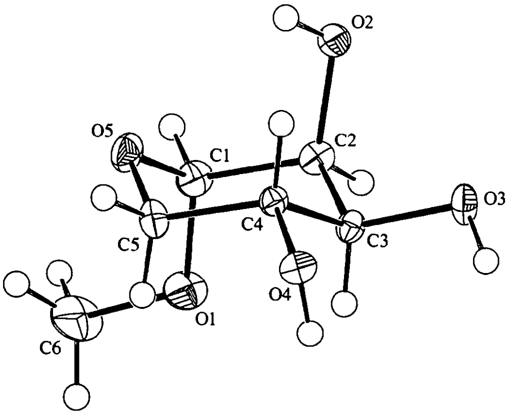


Fig. 1. Displacement ellipsoid plot (*SHELXTL-Plus*; Sheldrick, 1991) of (I). Ellipsoids represent 50% probability and H atoms are represented by spheres of arbitrary radii.

The asymmetric unit contains one molecule of (I) in a classic chair conformation (Fig. 1). Bond lengths and angles are normal and generally consistent with those of other pyranosides (Kirby, 1983; Fuchs, Schleifer & Tartakovsky, 1984).

The crystal packing of methyl α-D-lyxopyranoside involves a dense pattern of hydrogen bonds, linking molecules into a three-dimensional network (Fig. 2 and Table 2).

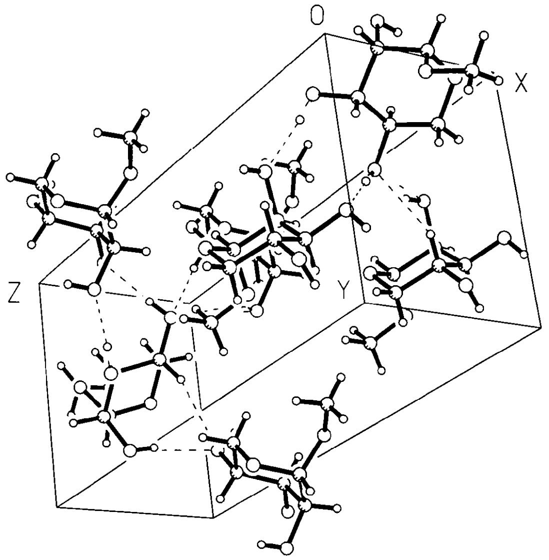


Fig. 2. A view of the crystal packing in which hydrogen bonds, represented by dashed lines, link molecules into a three-dimensional network.

Experimental

Methyl α-D-lyxopyranoside was synthesized from D-lyxose (Sigma) by Fischer anomerization (2.5% HCl/MeOH; Fischer, 1890). The product was purified by dissolution in diethyl ether/ethyl acetate and extracted into water. The water was removed by co-evaporation with benzene. Crystals were grown from ethyl acetate solution.

Crystal data

$C_6H_{12}O_5$
$M_r = 164.16$
Orthorhombic
$P2_12_12_1$
$a = 5.806\,(1)$ Å
$b = 7.714\,(2)$ Å
$c = 17.081\,(3)$ Å
$V = 765.0\,(3)$ Å^3
$Z = 4$
$D_x = 1.425$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 6$–$15°$
$\mu = 0.125$ mm^{-1}
$T = 103\,(2)$ K
Prism
$0.3 \times 0.2 \times 0.2$ mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: none
3646 measured reflections
1768 independent reflections
1658 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0657$

$\theta_{\rm max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 22$
3 standard reflections monitored every 200 reflections frequency: 57 min intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0577$
$wR(F^2) = 0.1502$
$S = 1.073$
1766 reflections
149 parameters
H atoms refined freely with individual isotropic displacement parameters

$w = 1/[\sigma^2(F_o^2) + (0.103P)^2 + 0.0579P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.73$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.37$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	$U_{\rm eq}$
O4	0.1598 (3)	0.6195 (2)	0.21944 (9)	0.0166 (7)
O3	0.5273 (3)	0.3655 (2)	0.23980 (9)	0.0178 (8)
O2	0.2836 (3)	0.1363 (2)	0.34049 (9)	0.0168 (8)
O5	0.0064 (3)	0.4114 (2)	0.40628 (8)	0.0205 (8)
O1	0.3464 (3)	0.5074 (2)	0.46638 (9)	0.0254 (11)
C4	0.1539 (3)	0.4858 (2)	0.27680 (11)	0.0129 (9)
C3	0.3939 (3)	0.4331 (3)	0.30305 (11)	0.0135 (9)
C5	0.0163 (4)	0.5445 (3)	0.34791 (12)	0.0186 (10)
C1	0.2291 (4)	0.3661 (3)	0.43439 (12)	0.0199 (11)
C2	0.3775 (4)	0.2969 (2)	0.36760 (11)	0.0153 (9)
C6	0.2401 (7)	0.5725 (4)	0.5364 (2)	0.037 (2)

Table 2. *Selected torsion angles (°) and hydrogen-bonding parameters (Å, °)*

C1—O5—C5—C4	61.16	C1—C2—C3—C4	−55.25
O5—C1—C2—C3	57.66	C3—C4—C5—O5	−57.08

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H22···O5	0.83 (3)	2.50 (3)	2.890 (2)	110 (3)
O2—H22···O4$^{\rm i}$	0.83 (3)	1.99 (3)	2.773 (3)	157 (3)
O3—H23···O2$^{\rm ii}$	0.86 (3)	1.88 (2)	2.729 (2)	173 (3)
O4—H24···O3$^{\rm ii}$	0.84 (2)	1.88 (3)	2.718 (2)	178 (3)

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *Xtal*3.2 (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Strained Cyclononadienyne

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Abstract

The title compound, methyl 8-chlorobicyclo[7.1.0]-deca-1,8-dien-4-yne-2-carboxylate, $C_{12}H_{11}ClO_2$, crystallizes with two chemically equivalent and essentially geometrically and conformationally identical molecules in the asymmetric unit. A cyclopropane ring is fused to a nine-membered ring such that it is flanked by two double bonds; the presence of a triple bond in the larger ring generates considerable strain, evidenced in the marked non-linearity of the alkyne unit [165.2 (2)–168.4 (2)° at the C atoms] and the wide angles within the nine-membered ring at the ring fusion atoms [153.09 (10)–155.5 (2)°].

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