Crystal structure of 2,3-di-*O*-acetyl-5,6-dideoxy-α-*D*-lyxo-5-hexenofuranose, $C_{10}H_{14}O_6$

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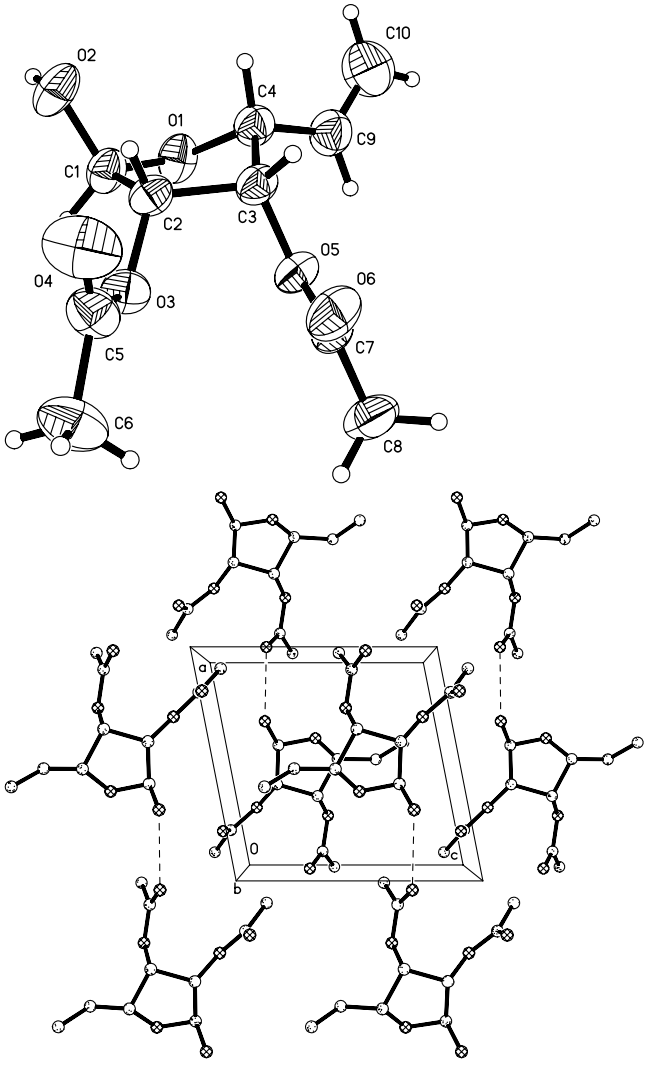

Abstract

$C_{10}H_{14}O_6$, monoclinic, $P2_1$ (no. 4), a = 8.195(2) Å, b = 8.672(2) Å, c = 8.479(1) Å, β = 101.02(1)°, V = 591.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.131, T = 293 K.

Source of material

The title compound has been obtained by vitamin B_{12}-catalysed fragmentation of methyl 2,3-di-*O*-acetyl-6-deoxy-6-iodo-α-*D*-mannopyranoside in methanol [1,2]. Isolation by chromatography (petroleum ether/ethyl acetate) gave a (67 : 33) mixture of α- and β-anomers as colourless oil. Crystallization from ethyl acetate/hexane gave the title compound as a pure α-anomer in the form of colourless crystals [1] (m.p. 352 - 353 K); $[\alpha]_D^{20}$ = +70.6 (c = 1.00, mixture of anomers in equilibrium).

Discussion

The furanose ring system of the molecule has an envelope conformation. C4 deviates by 0.58 Å from the plane given by C1/C2/C3/O1 (rmsd = 0.026 Å; figure, top; 40 % probability ellipsoids). The hydrogens of the methyl groups C6 and C8 of the acetyl moieties shows a rotational disorder with population parameters of 0.5 each. The double bond is clearly identified by the distance of 1.286(4) Å between C9 and C10. The crystal packing shows along [010] an antiparallel orientation of the molecules in the (010) plane. This effect is forced by the influence of the intermolecular hydrogen bond between the hydroxy group O2–H2A and the carbonyl oxygen O6 of the acetyl moiety (figure, bottom). The H2A···O6 distance is 1.94(3) Å and the angle O2–H2A···O6 is 167(3)°.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.3 × 0.5 × 0.7 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.08 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff scan
$2\theta_{max}$:	64.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8521, 4266
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3354
$N(param)_{refined}$:	178
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	2*a*		0.397(3)	–0.006(3)	0.790(3)	0.049(6)
H(2A)	2*a*		0.212(4)	0.172(4)	0.751(3)	0.072(8)
H(2)	2*a*		0.591(3)	0.260(3)	0.813(3)	0.055(6)
H(3)	2*a*		0.707(3)	0.251(2)	0.599(2)	0.039(5)
H(4)	2*a*		0.434(2)	0.255(3)	0.487(3)	0.042(5)
H(6A)	2*a*	0.50	0.8822	–0.0898	1.0615	0.121
H(6B)	2*a*	0.50	0.8857	0.0118	1.2151	0.121
H(6C)	2*a*	0.50	1.0216	0.0347	1.1097	0.121
H(6D)	2*a*	0.50	0.9774	0.0609	1.1960	0.121
H(6E)	2*a*	0.50	0.9739	–0.0407	1.0425	0.121
H(6F)	2*a*	0.50	0.8381	–0.0636	1.1478	0.121
H(8A)	2*a*	0.50	0.8985	–0.1860	0.5446	0.103
H(8B)	2*a*	0.50	1.0377	–0.1631	0.6976	0.103
H(8C)	2*a*	0.50	1.0613	–0.0958	0.5318	0.103
H(8D)	2*a*	0.50	1.0998	–0.1106	0.6381	0.103
H(8E)	2*a*	0.50	0.9606	–0.1335	0.4851	0.103

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(8F)	2*a*	0.50	0.9371	–0.2008	0.6509	0.103
H(9)	2*a*		0.545(4)	0.000(5)	0.320(4)	0.11(1)

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(10A)	2*a*		0.351(5)	0.262(5)	0.206(5)	0.12(1)
H(10B)	2*a*		0.414(5)	0.107(5)	0.090(5)	0.11(1)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*a*	0.3833(1)	0.0493(2)	0.5703(1)	0.0348(5)	0.0612(8)	0.0432(6)	–0.0107(5)	0.0053(4)	–0.0103(6)
C(1)	2*a*	0.4158(2)	0.0891(2)	0.7353(2)	0.0306(6)	0.056(1)	0.0414(8)	–0.0043(6)	0.0063(5)	–0.0049(7)
O(2)	2*a*	0.3096(2)	0.2062(2)	0.7681(2)	0.0321(6)	0.087(1)	0.0605(8)	0.0043(6)	0.0090(5)	–0.0215(7)
C(2)	2*a*	0.5927(2)	0.1560(2)	0.7716(2)	0.0291(5)	0.0441(7)	0.0394(7)	–0.0003(6)	0.0021(5)	–0.0028(7)
O(3)	2*a*	0.6993(1)	0.0589(1)	0.8826(2)	0.0401(6)	0.0469(7)	0.0446(6)	–0.0010(5)	–0.0026(5)	0.0003(5)
C(3)	2*a*	0.6449(2)	0.1628(2)	0.6072(2)	0.0301(6)	0.0384(7)	0.0463(8)	–0.0015(6)	0.0074(5)	–0.0012(6)
C(4)	2*a*	0.4775(2)	0.1540(2)	0.4913(2)	0.0364(6)	0.0465(8)	0.0400(7)	0.0047(7)	0.0037(5)	–0.0013(7)
O(4)	2*a*	0.7990(3)	0.2627(2)	1.0328(2)	0.100(1)	0.065(1)	0.072(1)	–0.009(1)	–0.029(1)	–0.0133(8)
O(5)	2*a*	0.7375(1)	0.0264(1)	0.5812(2)	0.0325(5)	0.0446(6)	0.0517(6)	0.0034(5)	0.0080(4)	–0.0046(5)
C(5)	2*a*	0.7992(2)	0.1279(3)	1.0084(2)	0.0447(8)	0.065(1)	0.0438(8)	–0.0093(8)	–0.0046(7)	0.0048(8)
O(6)	2*a*	0.9730(2)	0.1400(2)	0.7027(2)	0.0317(5)	0.0668(9)	0.081(1)	–0.0040(6)	0.0099(6)	–0.0061(8)
C(6)	2*a*	0.9068(3)	0.0107(4)	1.1075(3)	0.073(2)	0.082(2)	0.072(2)	0.001(1)	–0.024(1)	0.018(1)
C(7)	2*a*	0.9021(2)	0.0306(2)	0.6353(2)	0.0338(7)	0.060(1)	0.0460(8)	0.0050(7)	0.0110(6)	0.0039(8)
C(8)	2*a*	0.9821(3)	–0.1167(3)	0.5991(3)	0.054(1)	0.081(2)	0.069(1)	0.026(1)	0.007(1)	–0.007(1)
C(9)	2*a*	0.4803(3)	0.0944(3)	0.3263(3)	0.055(1)	0.069(1)	0.0437(9)	0.0046(9)	0.0064(7)	–0.0045(9)
C(10)	2*a*	0.4048(5)	0.1606(5)	0.1971(3)	0.105(2)	0.102(2)	0.046(1)	0.013(2)	0.003(1)	0.005(1)

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