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A fused furanoside-1,4-lactone at 173 K

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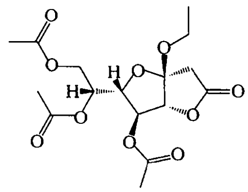
Abstract

The asymmetric unit of the title compound, (ethyl 5,7,8-tri-*O*-acetyl-2-deoxy-α-D-*erythro*-L-*arabino*-3-octulofuranosid)ono-1,4-lactone, $C_{16}H_{22}O_{10}$, contains two symmetry-independent molecules which have very similar molecular dimensions and no significant conformational differences, except for slight twists in some of the substituents. The furanoid sugar ring in both molecules has the envelope conformation, in which the anomeric C atom is out of the plane of the ring. The lactone ring is not planar and also adopts the envelope conformation, but the lactone group itself shows only a very small deviation from planarity.

Comment

Recent growth in the study of the chemistry of *C*-glycosides has been tremendous. These compounds have been widely used as chiral templates for complex synthetic target molecules and many have shown interesting and useful biological activities. One group of *C*-glycoside derivatives that has not been studied widely for synthetic behaviour (Bandzouzi & Chapleur, 1987*a*,*b*; Csuk & Glänzer, 1990, 1991) and enzyme inhibitory activity (Brockhaus & Lehmann, 1977, 1978; Lehmann & Schwesinger, 1982*a*,*b*) consists of those compounds that contain an exocyclic double bond at the anomeric centre. Such compounds are potential inhibitors, in that they interact with the enzyme to form intermediates which are covalently bound to the enzyme. In this paper, we describe the crystal structure of a fused furanoside-1,4-lactone derivative, (I), which was obtained in the course of our search for convenient and efficient approaches to the synthesis of exoalkylenic sugar compounds.



(I)

The structure of compound (I) is composed of two symmetry-independent molecules, one of which (molecule *A*) is shown in Fig. 1. Molecule *B* has essentially the same appearance, and its atom-numbering scheme can be derived from that for molecule *A* by adding 20. Fig. 1 depicts the correct absolute configuration of molecule *A*, which was assigned to agree with the known chirality of D-galactose, from which compound (I) was synthesized. The two molecules, *A* and *B*, have very similar molecular dimensions and no significant conformational differences, except for slight twists in some of the substituents. The bond lengths and angles (Table 1) agree well with those reported for other compounds with a lactone ring (Jeffrey *et al.*, 1967; Kim *et al.*, 1967; Usher & English, 1978; Conde *et al.*, 1980). However, the difference between the lactone ring C—O distances is rather small [0.080 (5) and 0.076 (5) Å in molecules *A* and *B*, respectively] compared with that

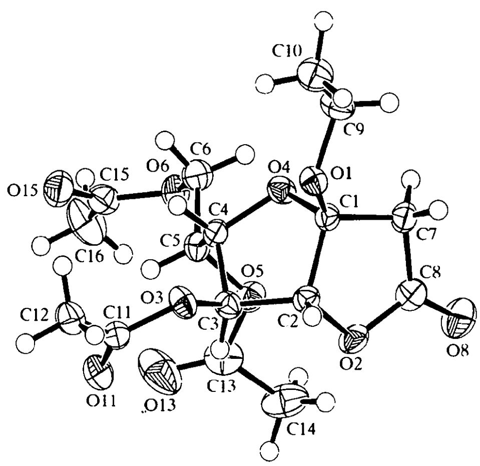


Fig. 1. A view of molecule *A* of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. Molecule *B* has essentially the same appearance.

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in a similar fused-ring system, β-D-glucofuranurono-6,3-lactone [0.135 (7) Å; Kim *et al.*, 1967], indicating a small contribution from the valance bond resonance form.

The furanoid sugar ring has the envelope (*E*) conformation in each molecule. The ring puckering parameters (Cremer & Pople, 1975; Table 2) show that the phase angle, φ_2, is close to 216°, which is one of the ideal values for the *E* form, but that molecule *A* deviates slightly from an ideal *E* conformation towards a half-chair conformation, while molecule *B* has an almost perfect *E* conformation. Each of the anomeric carbon atoms, C1 and C21, deviates from the mean plane defined by the other four furanoid ring atoms by 0.401 (4) and 0.423 (4) Å for molecules *A* and *B*, respectively. These planes have r.m.s. deviations of their constituent atoms of 0.014 and 0.003 Å, respectively, which further demonstrates the less ideal nature of the *E* conformation of molecule *A*.

The lactone ring in each molecule also has the *E* conformation, as φ_2 is close to 252°. The deviation of the anomeric carbon atoms, C1 and C21, from the mean planes defined by the other four lactone ring atoms is 0.362 (4) and 0.412 (5) Å for molecules *A* and *B*, respectively, and the r.m.s. deviations of the constituent atoms from these planes are 0.015 and 0.004 Å, respectively. The lactone ring in molecule *A* also has a less ideal *E* conformation than that in molecule *B*. The atoms of the lactone group in each molecule [C7, C8(=O8), O2 and C2, and C27, C28(=O28), O22 and C22] are planar. The r.m.s. deviations of the constituent atoms from each of these mean planes are 0.022 and 0.007 Å, with the maximum deviation from each plane being 0.0323 (18) and 0.0095 (19) Å for atoms O2 and O22 of molecules *A* and *B*, respectively.

The torsion angles about the C5—C6 bond, involving atom O6 with atoms O5 and C4 (Table 1) in molecule *A*, describe the *gauche-gauche* conformation. This arrangement ensures that the acetyl group bonded to C5 avoids approaching atom O3 and the furanose ring oxygen O4 too closely. Furthermore, the O5—C5—C6—O6 torsion angle shows that atom O6 has a *gauche* relationship with respect to atom O5. In this arrangement, atom O6 is '*trans*' to and farthest away from the ring atom O4. The same conformation is observed for molecule *B*.

Experimental

A solution of 2,3,4,6-tetra-*O*-benzyl-1-*C*-(ethoxylcarbonylmethylene)-α-D-galactopyranose (1.0 g; Li, 1998) in ethanol (10 ml) was hydrogenated (50 psi, 298 K; 1 psi ≃ 6.895 × 10^3 Pa) in the presence of 10% palladium-on-charcoal (100 mg). The solution was filtered, concentrated and then acetylated in the conventional manner. Flash column chromatography (ethyl acetate/hexane, 3:1) gave (I) as the product (0.13 g, 21.2%), m.p. 373–375 K (ethyl acetate/hexane), $[\alpha]_D$ +30.6° (*c* 0.5, $CHCl_3$). Suitable crystals were grown by slow evaporation of a solution in ethyl acetate/hexane.

Crystal data

$C_{16}H_{22}O_{10}$	Mo $K\alpha$ radiation
M_r = 374.34	λ = 0.71073 Å
Monoclinic	Cell parameters from 25 reflections
$P2_1$	θ = 18–20°
a = 9.022 (3) Å	μ = 0.113 mm^{-1}
b = 15.576 (2) Å	T = 173 (1) K
c = 13.489 (3) Å	Prism
β = 103.52 (2)°	0.38 × 0.38 × 0.33 mm
V = 1843.0 (7) Å^3	Colourless
Z = 4	
D_x = 1.349 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-5*R* diffractometer	θ_{max} = 30°
$\omega/2\theta$ scans	$h = 0 \rightarrow 12$
Absorption correction: none	$k = 0 \rightarrow 21$
5850 measured reflections	$l = -18 \rightarrow 18$
5537 independent reflections	3 standard reflections every 150 reflections intensity decay: insignificant
3931 reflections with $I > 2\sigma(I)$	
R_{int} = 0.026	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 0.001
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$wR(F^2)$ = 0.114	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
S = 1.023	Extinction correction: none
5537 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
477 parameters	
H atoms constrained	
$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.2297P]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.393 (3)	O21—C21	1.401 (3)
O2—C2	1.448 (3)	O22—C22	1.444 (3)
O2—C8	1.368 (4)	O22—C28	1.368 (4)
O4—C1	1.431 (3)	O24—C21	1.423 (3)
O4—C4	1.437 (3)	O24—C24	1.443 (3)
C9—O1—C1—C2	173.3 (2)	C29—O21—C21—C22	165.9 (2)
C4—O4—C1—C2	26.2 (3)	C24—O24—C21—C22	29.3 (3)
C8—O2—C2—C1	17.0 (3)	C28—O22—C22—C21	16.9 (3)
O4—C1—C2—O2	89.4 (2)	O24—C21—C22—O22	87.7 (2)
C7—C1—C2—O2	−23.5 (3)	C27—C21—C22—O22	−25.7 (3)
O4—C1—C2—C3	−26.9 (3)	O24—C21—C22—C23	−27.5 (3)
O2—C2—C3—C4	−94.4 (2)	O22—C22—C23—C24	−95.6 (3)
C1—C2—C3—C4	18.0 (3)	C21—C22—C23—C24	16.1 (3)
C1—O4—C4—C3	−14.9 (3)	C21—O24—C24—C23	−19.1 (3)
C2—C3—C4—O4	−2.9 (3)	C22—C23—C24—O24	0.6 (3)
O4—C4—C5—O5	68.6 (3)	O24—C24—C25—O25	62.0 (3)
C3—C4—C5—O5	−51.1 (3)	C23—C24—C25—O25	−56.5 (3)
O4—C4—C5—C6	−51.0 (3)	O24—C24—C25—C26	−57.6 (3)
C3—C4—C5—C6	−170.7 (2)	C23—C24—C25—C26	−176.2 (2)
O5- C5—C6—O6	62.8 (3)	O25—C25—C26—O26	56.2 (3)
C4—C5—C6—O6	−176.6 (2)	C24—C25—C26—O26	175.4 (2)
O4—C1—C7—C8	−89.9 (2)	O24—C21—C27—C28	−86.6 (3)
C2—C1—C7—C8	20.9 (3)	C22—C21—C27—C28	24.5 (3)
C2—O2—C8—C7	−3.7 (3)	C22—O22—C28—C27	−1.0 (3)
C1—C7—C8—O2	−11.4 (3)	C21—C27—C28—O22	−15.4 (3)

Table 2. *Ring puckering parameters in compound (I)*

	Q (Å)	φ_2 (°)
Ideal values[a]		
Five-membered envelope	-	$n \times 36$
Five-membered half-chair	-	$(n \times 36) + 18$
Furanoid ring		
Molecule *A*	0.258 (3)	223.3 (6)
Molecule *B*	0.272 (3)	215.8 (6)
Lactone ring		
Molecule *A*	0.230 (3)	244.8 (7)
Molecule *B*	0.259 (3)	251.1 (7)

[a] Cremer & Pople (1975).

The origin was fixed according to the method of Flack & Schwarzenbach (1988). The program *PLATON* (Spek, 1998) confirmed that there was no overlooked additional symmetry relating the two independent molecules. All H atoms were placed in geometrically calculated positions. The methyl H atoms were refined as rigid groups, which were allowed to rotate but not to tip, with $U_{iso}(H) = 1.5U_{eq}(C)$. All other H atoms were allowed to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*97 direct methods (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1305). Services for accessing these data are described at the back of the journal.

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Amino-11-undecanoic acid cyclic dimer hydrochloride†

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Abstract

The structure of the hydrochloride salt of amino-11-undecanoic acid cyclic dimer ($C_{22}H_{42}N_2O_2 \cdot HCl$) corresponds to two monomeric units of nylon 11. The intensity data were collected at 203 K. Two molecules are linked by two short hydrogen bonds with O···O distances of 2.406 (6) and 2.464 (6) Å between half-protonated amide-O atoms. The methylene groups are all-*trans* except for two terminal groups, which display a (−)synclinal form (*gauche*). The ring is closed through bent amide groups, which are reminiscent of β-turns in proteins. The all-*trans* methylene chains along both sides of the ring are not parallel, but are organized at an angle of about 10.9 (6)°.

Comment

We have determined the structure of a 24-atom ring made up of two units of aminoundecanoic acid, (I). This is part of a research effort aimed at understanding folding in polyamides (nylons) and in related compounds like proteins.

† IUPAC name: 1,13-diazatetracosa-2,14-dioxonium chloride.