C(4′)—C(5′)—C(6′)—O(6′)	−167.9 (5)
C(4′)—C(5′)—O(5′)—C(1′)	62.3 (9)
C(6′)—C(5′)—O(5′)—C(1′)	−176.7 (6)
O(5′)—C(1′)—C(2′)—C(3′)	61.4 (7)
O(5′)—C(5′)—C(6′)—O(6′)	72.3 (7)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
O(2)—H(O2)···O(3′^{i})	1.89 (4)	2.68 (1)	163 (2)
O(3)—H(O3)···O(6′^{ii})	1.87 (7)	2.682 (5)	167 (2)
O(4)—H(O4)···O(5^{iii})	2.06 (5)	2.812 (8)	146 (2)
O(6)—H(O6)···O(2′^{iv})	2.07 (7)	2.831 (5)	155 (2)
O(2′)—H(O2′)···O(*W*)	1.95 (8)	2.699 (7)	153 (2)
O(3′)—H(O3′)···O(3^{v})	1.91 (5)	2.705 (6)	170 (6)
O(4′)—H(O4′)···O(4^{v})	1.91 (8)	2.660 (5)	154 (5)
O(6′)—H(O6′)···O(6)	1.91 (5)	2.728 (4)	154 (6)
O(*W*)—H(1*W*)···O(1)	1.96 (6)	2.76 (1)	169 (6)
O(*W*)—H(2*W*)···O(4′^{ii})	2.09 (9)	3.008 (6)	168 (6)

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

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MSC/AFC Diffractometer Control Software*. Program(s) used to solve structure: *MULTAN*88 (Main *et al.*, 1988). Program(s) used to refine structure: *KPPXRAY ORFLS* (Taga, Masuda, Higashi & Iizuka, 1991). Molecular graphics: *KPPXRAY*. Software used to prepare material for publication: *EDCIF-J* (Osaki & Taga, 1993).

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: KH1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 236–238

(6*S*)-8-*C*-Chloro-6,7,8-trideoxy-1,2:3,4-di-*O*-isopropylidene-α-D-*galacto*-octa-6,7-dienopyranose

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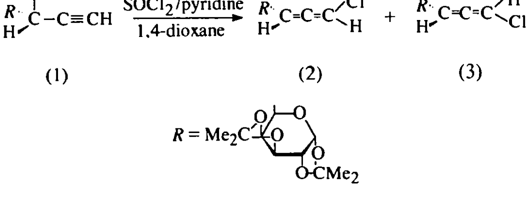
Abstract

The title structure, $C_{14}H_{19}ClO_5$, consists of an allene substituted with a Cl atom at one end joined at the other end to a D-galactopyranose ring bearing two fused isopropylidene groups.

Comment

One aspect of our research (Horton & Liu, 1996) is the investigation of stereospecific conversions of D-galactose-derived propargylic alcohols into chiral haloallenes. This can be accomplished by reacting suitable alcohols with $SOCl_2$/pyridine in boiling 1,4-dioxane. These reactions can afford either two stereoisomers [(2) and (3) as shown in the scheme] or possibly diastereomeric mixtures. These compounds differ in the arrangement of the Cl atom on the allene moiety. In the specific dehydration reaction depicted in the scheme below (Horton & Liu, 1996), only one product was obtained and the single-crystal structure was determined as a means of establishing the arrangement of ligands around the allene group. Our results here establish that the compound synthesized has the arrangement depicted as compound (2).



A *PLATON* (Spek, 1996) representation of the molecule and the atomic labeling scheme is given in Fig. 1 and a stereoview of the unit-cell packing diagram is provided in Fig. 2. The bond lengths and the valence angles given in Table 2 are all within expected ranges for this type of compound. The absolute configuration was assigned to agree with the known chirality of the sugar moiety (*i.e.* α-D-*galacto*). In fact, this rules out the possibility of the molecule being arranged around a center of inversion, as this would result in an inversion at all of the chiral centers in the molecule.

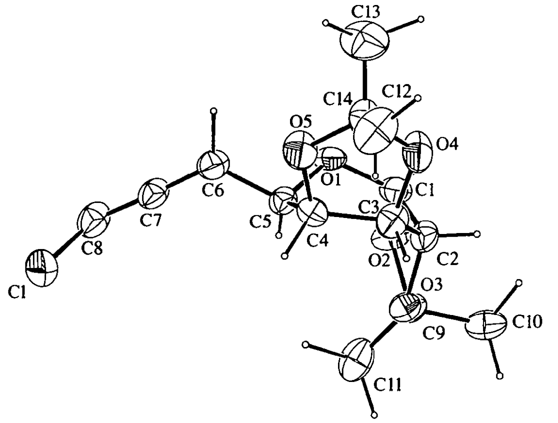


Fig. 1. *PLATON*96 (Spek, 1996) drawing of (2) showing 30% probability displacement ellipsoids. The H atoms are shown as circles of arbitrary size.

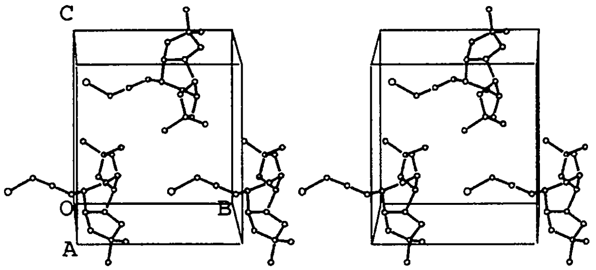


Fig. 2. Stereoview of the unit-cell packing of (2).

The title compound consists of the six-membered galactopyranose ring oriented in a skew-boat conformation. The torsion angles around this ring which provide definitive evidence for the stated conformation (Bucourt, 1974) are listed in Table 2. This clearly affords the best geometry to accommodate the two isopropylidene groups attached to atoms C1, C2 and C3, C4 of the sugar ring. The arrangement of the ligands on the allene group (*i.e.* the sugar moiety and the Cl atom) establishes an overall *S* configuration for this molecule in the allene moiety.

Experimental

Crystals of (2) were obtained by slow crystallization from an ethyl acetate solution.

Crystal data

$C_{14}H_{19}ClO_5$
$M_r = 302.76$
Monoclinic
$P2_1$
$a = 7.720(1)$ Å
$b = 9.764(1)$ Å
$c = 10.643(2)$ Å
$\beta = 106.79(1)^\circ$
$V = 768.1(5)$ Å^3
$Z = 2$
$D_x = 1.31$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 15 reflections
$\theta = 11$–18°
$\mu = 2.37$ mm^{-1}
$T = 296$ K
Irregular
$0.20 \times 0.17 \times 0.15$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.575$, $T_{\max} = 0.701$
1706 measured reflections
1706 independent reflections

1590 reflections with $F > 3\sigma(F)$
$\theta_{\max} = 74^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 10$
$l = -13 \rightarrow 12$
3 standard reflections
frequency: 250 min
intensity decay: 0.8%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.053$
$S = 1.90$
1590 reflections
180 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} = 0.02$

$\Delta\rho_{\max} = 0.202$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
Absolute configuration: assigned to agree with the known chirality of the sugar moiety

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

Cl—C8	1.750 (5)	C1—C2	1.513 (4)
O1—C1	1.411 (5)	C2—C3	1.505 (6)
O1—C5	1.432 (5)	C3—C4	1.528 (7)
O2—C1	1.410 (5)	C4—C5	1.526 (5)
O3—C2	1.423 (5)	C5—C6	1.498 (6)
O4—C3	1.425 (6)	C6—C7	1.289 (7)
O5—C4	1.426 (5)	C7—C8	1.280 (7)
C1—O1—C5	114.2 (3)	O5—C4—C3	104.0 (3)
O1—C1—O2	110.5 (3)	O5—C4—C5	110.5 (3)
O1—C1—C2	114.5 (3)	C3—C4—C5	112.2 (3)
O2—C1—C2	103.8 (3)	O1—C5—C4	110.5 (3)
O3—C2—C1	103.9 (3)	O1—C5—C6	107.0 (3)
O3—C2—C3	109.0 (4)	C4—C5—C6	111.1 (3)
C1—C2—C3	114.0 (3)	C5—C6—C7	124.6 (4)
O4—C3—C2	106.2 (4)	C6—C7—C8	178.0 (5)
O4—C3—C4	103.7 (3)	Cl—C8—C7	122.6 (3)
C2—C3—C4	114.2 (3)		
O1—C1—C2—C3	−21.3 (5)	C3—C4—C5—O1	−38.1 (4)
C1—C2—C3—C4	45.7 (5)	C4—C5—O1—C1	66.5 (4)
C2—C3—C4—C5	−15.2 (5)	C5—O1—C1—C2	−34.8 (5)

From the systematic absences of 0*k*0 and from subsequent least-squares refinement, the space group was determined to be $P2_1$. H atoms were included at calculated positions, with C—H distances set at 0.92–0.98 Å and common isotropic displacement parameters fixed at 1.3 times the $U_{\rm eq}$ values of the atoms to which they are attached.

Data collection: CAD-4*F* software. Cell refinement: CAD-4*F* software. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: NA1258). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9,10-Dihydro-9-oxo-10-acridineacetic Acid

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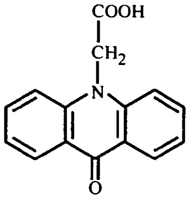
Abstract

In the crystal of the title compound, $C_{15}H_{11}NO_3$, the molecules form chains of strong acid-to-ketone hydrogen bonds, with an O···O distance of 2.585 (3) Å.

Comment

As a part of a study of the coordination properties of the 9,10-dihydro-9-oxo-10-acridineacetate ligand (CMA^-), we have recorded the IR spectra and performed the X-ray structure determinations of a series of metal complexes with the CMA^- ligand (*M*CMA, *M* = Cu, Zn, Ca, Pb; Miernik, Lis, Palus & Reedijk, 1993; Miernik & Lis, 1994, 1996) and also of the free acid, (I), which is presented here.



(I)

An *ORTEP*II (Johnson, 1976) view of the title molecule is presented in Fig. 1. The molecule exhibits a characteristic slightly bent shape; the dihedral angle between the planes determined by the atoms of the two halves of the macrocycle (*i.e.* N1, C7–C13 and N1, C3–C6, C13–C15) is 7.1 (1)°. The geometry of the acridone ring is in a good agreement with that of the CMA^- complexes; differences between average and individual values are statistically insignificant.

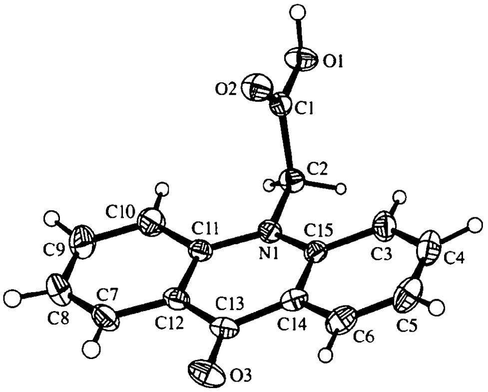


Fig. 1. An *ORTEP*II (Johnson, 1976) view of the CMAH molecule with the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and displacement parameters of the H atoms were artificially diminished by a factor of five for clarity.

A stereoscopic packing diagram is shown in Fig. 2. The CMAH molecules form ketone-to-acid intermolecular hydrogen bonds, which is relatively uncommon for keto carboxylic acids (Vanderhoff, Lalancette & Thompson, 1990; Lalancette, Vanderhoff & Thompson, 1991; Lalancette, Thompson & Vanderhoff, 1991; Lalancette, Coté & Thompson, 1996). The CMAH hydrogen chains are oriented parallel to the [011] and $[01\bar{1}]$ directions. The intermolecular ketone-to-acid distance O3···O1($\frac{1}{2}-x$, $-\frac{1}{2}+y$, $-\frac{1}{2}+z$) is 2.585 (3) Å, with O3···H1($\frac{1}{2}-x$, $-\frac{1}{2}+y$, $-\frac{1}{2}+z$) 1.69 (4) Å, which is characteristic of strong *syn* interactions; the hydrogen-bond angle O3···H1—O1 is 158 (3)°. The participation of both carboxylic O atoms (O1 and O2) in hydrogen bonding, and therefore the existence of typical carboxylic dimers in the solid state of CMAH, was excluded on the grounds of the solid-state (KBr) IR spectra; a very