2272 reflections
269 parameters
All H-atom parameters refined
Unit weights applied
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
O(1)	0.1253 (1)	0.4182 (1)	0.0991 (2)	0.029 (1)
O(2)	−0.0077 (1)	0.4280 (1)	0.2073 (3)	0.030 (1)
C(1)	0.1724 (2)	0.3252 (1)	0.7301 (4)	0.035 (1)
C(2)	0.1504 (2)	0.2776 (1)	0.6225 (4)	0.034 (1)
C(3)	0.0864 (2)	0.3212 (1)	0.6430 (3)	0.023 (1)
C(4)	−0.0010 (2)	0.3197 (1)	0.7257 (4)	0.024 (1)
C(5)	−0.0233 (2)	0.3187 (1)	0.9179 (4)	0.037 (1)
C(6)	−0.0470 (2)	0.2730 (1)	0.8016 (4)	0.037 (1)
C(7)	−0.0322 (2)	0.3581 (1)	0.5854 (3)	0.022 (1)
C(8)	−0.0909 (2)	0.4043 (1)	0.6132 (4)	0.023 (1)
C(9)	−0.1237 (2)	0.4332 (1)	0.7701 (4)	0.030 (1)
C(10)	−0.0845 (2)	0.4622 (1)	0.6139 (4)	0.031 (1)
C(11)	−0.1183 (2)	0.3617 (1)	0.4990 (4)	0.026 (1)
C(12)	−0.1931 (2)	0.3264 (2)	0.4753 (5)	0.039 (1)
C(13)	−0.1526 (2)	0.3546 (1)	0.3192 (4)	0.031 (1)
C(14)	0.0548 (2)	0.3571 (1)	0.5009 (3)	0.021 (1)
C(15)	0.0963 (2)	0.3758 (1)	0.3613 (4)	0.025 (1)
C(16)	0.0631 (2)	0.4100 (1)	0.2197 (4)	0.024 (1)
C(17)	0.1034 (2)	0.4502 (1)	−0.0529 (4)	0.029 (1)
C(18)	0.1841 (2)	0.4718 (1)	−0.1283 (5)	0.039 (1)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(16)	1.353 (3)	C(7)—C(11)	1.504 (4)
O(1)—C(17)	1.450 (4)	C(7)—C(14)	1.507 (4)
O(2)—C(16)	1.204 (3)	C(8)—C(9)	1.490 (4)
C(1)—C(2)	1.497 (5)	C(8)—C(10)	1.471 (4)
C(1)—C(3)	1.506 (4)	C(8)—C(11)	1.449 (4)
C(2)—C(3)	1.500 (4)	C(9)—C(10)	1.523 (4)
C(3)—C(4)	1.507 (4)	C(11)—C(12)	1.487 (4)
C(3)—C(14)	1.496 (4)	C(11)—C(13)	1.476 (4)
C(4)—C(5)	1.499 (4)	C(12)—C(13)	1.522 (5)
C(4)—C(6)	1.502 (4)	C(14)—C(15)	1.330 (4)
C(4)—C(7)	1.522 (4)	C(15)—C(16)	1.474 (4)
C(5)—C(6)	1.503 (5)	C(17)—C(18)	1.494 (5)
C(7)—C(8)	1.505 (4)		
C(16)—O(1)—C(17)	116.9 (2)	C(7)—C(11)—C(8)	61.3 (2)
C(2)—C(1)—C(3)	59.9 (2)	C(7)—C(11)—C(12)	136.4 (3)
C(1)—C(2)—C(3)	60.4 (2)	C(8)—C(11)—C(12)	139.0 (3)
C(1)—C(3)—C(2)	59.7 (2)	C(7)—C(11)—C(13)	136.2 (2)
C(1)—C(3)—C(4)	129.4 (2)	C(8)—C(11)—C(13)	138.6 (3)
C(2)—C(3)—C(4)	129.3 (2)	C(12)—C(11)—C(13)	61.8 (2)
C(1)—C(3)—C(14)	124.8 (2)	C(11)—C(12)—C(13)	58.7 (2)
C(2)—C(3)—C(14)	126.6 (2)	C(11)—C(13)—C(12)	59.5 (2)
C(4)—C(3)—C(14)	90.8 (2)	C(3)—C(14)—C(7)	90.2 (2)
C(3)—C(4)—C(5)	128.1 (3)	C(3)—C(14)—C(15)	128.9 (2)
C(3)—C(4)—C(6)	127.9 (2)	C(4)—C(7)—C(8)	126.6 (2)
C(5)—C(4)—C(6)	60.1 (2)	C(4)—C(7)—C(11)	129.2 (2)
C(3)—C(4)—C(7)	89.2 (2)	C(8)—C(7)—C(11)	57.6 (2)
C(5)—C(4)—C(7)	128.0 (2)	C(4)—C(7)—C(14)	89.8 (2)
C(6)—C(4)—C(7)	128.2 (2)	C(8)—C(7)—C(14)	128.7 (2)
C(4)—C(5)—C(6)	60.0 (2)	C(11)—C(7)—C(14)	129.0 (2)
C(4)—C(6)—C(5)	59.8 (2)	C(7)—C(8)—C(9)	135.0 (2)
C(7)—C(8)—C(10)	137.3 (2)	C(7)—C(14)—C(15)	140.9 (2)
C(9)—C(8)—C(10)	61.9 (2)	C(14)—C(15)—C(16)	128.0 (2)
C(7)—C(8)—C(11)	61.2 (2)	O(1)—C(16)—O(2)	123.6 (2)
C(9)—C(8)—C(11)	137.9 (2)	O(1)—C(16)—C(15)	109.1 (2)
C(10)—C(8)—C(11)	140.0 (3)	O(2)—C(16)—C(15)	127.3 (3)
C(8)—C(9)—C(10)	58.5 (2)	O(1)—C(17)—C(18)	107.9 (2)
C(8)—C(10)—C(9)	59.6 (2)		

All calculations were performed using the *SHELXS*86 (PC version) programme (Robinson & Sheldrick, 1988) on an IBM PC/AT computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1025). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structural Studies of Intermediates in the Synthesis of Mifepristone (RU 486). III. 3,3-Ethylenedioxy-5(10),9(11)-estradien-17-one

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Abstract

Crystallographic characterization of the title compound, $C_{20}H_{26}O_3$, an intermediate in the synthesis of mifepristone (RU 486), has helped establish the molecular structure and its conformation. An uncommon C(14)-envelope conformation is observed in the five-membered ring due to the presence of a trigonal C atom. The molecules form continuous intermolecular C—H···O hydrogen-bonded chains arranged in a more or less zigzag shape along the *b* axis.

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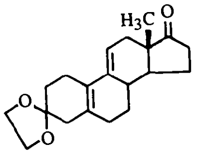
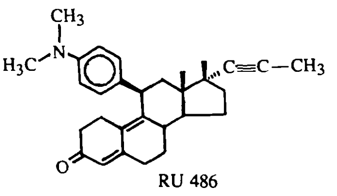
Comment

The title compound, (I), is an intermediate in the synthesis of mifepristone (RU 486), (II), a potent antiprogesterone drug available for clinical use in non-surgical abortion (Ulmann & Dubois, 1989; Heikinheimo, Ylikorkala & Lahteenmaki, 1990).



(I)



(II)

The most convenient method for the introduction of an 11β-aromatic substituent, like in RU 486, is the formation of 5(10),9(11)-estradienes followed by their epoxidation (Gasc & Nedelec, 1971). Crystallographic characterization of the title compound was carried out in order to assign the structure and stereochemistry, and has assisted in locating the diene linkage between C(5), C(10), C(9) and C(11). The conformation of the steroid molecule and atom numbering are shown in Fig. 1. The intramolecular dimensions involving non-H atoms of the steroid molecule are given in Table 2.

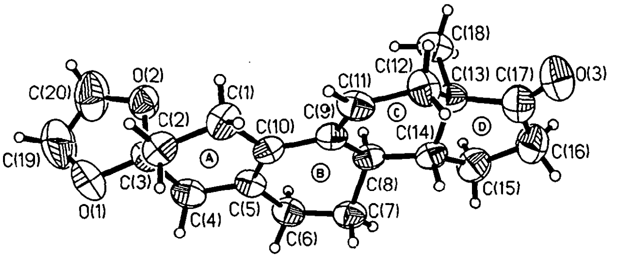


Fig. 1. Perspective view of the title molecule with numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and the H atoms are shown as spheres of arbitrary radii.

The molecular dimensions are in agreement with accepted values reported for other similar steroid structures (Parvez, Fajardo & Shamma, 1988; Duax, Griffin, Strong & Wood, 1989). Conformational features of the molecule may be described in terms of asymmetry parameters (Duax & Norton, 1975) taking into consideration the signs and magnitudes of the intra-annular torsion angles (Table 2). The presence of a double bond between C(5) and C(10) gives rise to a slightly distorted 2β,3α-half-chair conformation in ring *A* with asymmetry parameter ΔC_2[C(2)—C(3)] = 3.11°, and ring *B* approximates to a slightly distorted 7β,8α-half-chair conformation described in terms of ΔC_2[C(7)—C(8)] = 7.35°. Due to the presence of the C(9)═C(11) double bond, ring *C* also shows a slightly distorted 13α,14β half-chair conformation with ΔC_2[C(13)—C(14)] = 1.77°. The structural features of the five-membered ring *D* and the relative strain of the *C*/*D* ring junction are of great importance in the study of steroid chemistry. The conformation of ring *D* here is an uncommon 14β-envelope, with ΔC_s[C(14)] = 4.83°. A similar 14α- envelope conformation for ring *D* has been observed in the crystal structure of 3β-hydroxy-16β-morpholino-5-androsten-17-one and may be attributed to the presence of a trigonal C atom at C(17) (Swenson, Duax, Numazawa & Osawa, 1980). Of all the torsion angles within rings *A*, *B* and *C*, only C(12)—C(13)—C(14)—C(8) is greater than 60° [64.9 (6)°]. The strain inherent in the *C*/*D* ring junction is responsible for the valency angles C(12)—C(13)—C(17) [116.3 (5)°] and C(8)—C(14)—C(15) [120.0 (4)°] being greater than those normally observed for quaternary- (109.5°) and tertiary-substituted C atoms (110.5°) (Geise, Altona & Romers, 1967).

The unit-cell packing, with C—H···O bonds indicated, is shown in Fig. 2. In the crystal, the molecules form continuous intermolecular C—H···O (head-to-tail) hydrogen-bonded chains (Taylor & Kennard, 1982). Each chain is arranged in a more or less zigzag shape along the *b* axis, related by (*x*, *y* + 1, *z*) [C(20)···O(3) 3.244 (8), H(C20)···O(3) 2.552 (8) Å and C(20)—H(C20)···O(3) 130.6 (6)°].

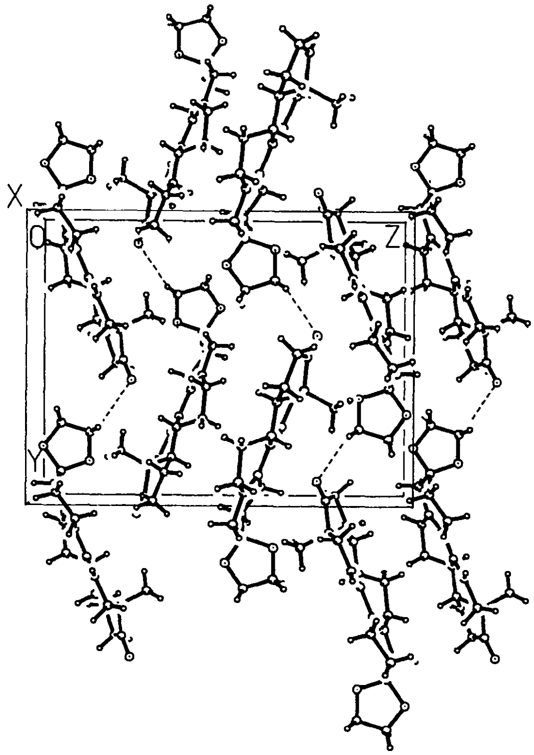


Fig. 2. Molecular packing in the unit cell viewed down the [100] axis. Dashed lines indicate C—H···O interactions.

Experimental

The compound was prepared by a multistep synthesis, according to the method of Teutsch, Costerousse, Philibert & Deraedt (1984), and crystals suitable for X-ray analysis were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{20}H_{26}O_3$
$M_r = 314.4$
Orthorhombic
$P2_12_12_1$
$a = 7.572$ (1) Å
$b = 13.250$ (2) Å
$c = 17.376$ (2) Å
$V = 1743.3$ (4) Å^3
$Z = 4$
$D_x = 1.198$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 2–12°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Needle
0.17 × 0.14 × 0.13 mm
Colourless

Data collection

Siemens *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1365 measured reflections
1346 independent reflections
1046 observed reflections [$I > 3\sigma(I)$]

$\theta_{\max} = 22.4°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 98 reflections
intensity decay: <1%

Refinement

Refinement on F
$R = 0.041$
$wR = 0.049$
$S = 1.088$
1046 reflections
208 parameters
$w = 1/[\sigma^2(F) + 0.0028F^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

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	$U_{\rm eq}$
O(1)	0.1180 (5)	0.1863 (3)	0.5160 (2)	0.080 (2)
O(2)	0.1642 (5)	0.1331 (3)	0.6394 (2)	0.061 (2)
O(3)	0.3032 (5)	−0.5762 (3)	0.7477 (2)	0.085 (2)
C(1)	0.0290 (7)	−0.0682 (4)	0.6063 (4)	0.055 (2)
C(2)	−0.0032 (7)	0.0260 (4)	0.5564 (3)	0.057 (2)
C(3)	0.1506 (7)	0.0974 (4)	0.5622 (3)	0.054 (2)
C(4)	0.3190 (7)	0.0476 (4)	0.5373 (3)	0.057 (2)
C(5)	0.3447 (7)	−0.0596 (4)	0.5652 (3)	0.046 (2)
C(6)	0.5277 (7)	−0.0998 (4)	0.5521 (3)	0.055 (2)
C(7)	0.5365 (7)	−0.2162 (4)	0.5607 (3)	0.053 (2)
C(8)	0.4406 (7)	−0.2497 (4)	0.6336 (3)	0.044 (2)
C(9)	0.2456 (7)	−0.2145 (4)	0.6308 (3)	0.045 (2)
C(10)	0.2134 (7)	−0.1129 (4)	0.5988 (3)	0.045 (2)
C(11)	0.1158 (7)	−0.2746 (5)	0.6576 (3)	0.053 (2)
C(12)	0.1377 (7)	−0.3806 (4)	0.6926 (4)	0.056 (2)
C(13)	0.3322 (7)	−0.3972 (4)	0.7137 (3)	0.048 (2)
C(14)	0.4518 (7)	−0.3610 (4)	0.6472 (3)	0.048 (2)
C(15)	0.6333 (7)	−0.4111 (5)	0.6640 (4)	0.065 (2)
C(16)	0.5792 (7)	−0.5177 (5)	0.6964 (4)	0.081 (3)
C(17)	0.3914 (7)	−0.5067 (5)	0.7228 (4)	0.065 (2)
C(18)	0.3786 (7)	−0.3420 (5)	0.7908 (3)	0.062 (2)
C(19)	0.1472 (7)	0.2732 (5)	0.5645 (4)	0.077 (3)
C(20)	0.1524 (7)	0.2406 (5)	0.6427 (4)	0.074 (2)

Table 2. *Selected geometric parameters* (Å, °)

O(3)—C(17)	1.218 (8)	C(4)—C(5)	1.515 (8)
C(1)—C(10)	1.522 (8)	C(5)—C(10)	1.352 (7)
C(5)—C(6)	1.502 (8)	C(9)—C(11)	1.348 (8)
C(8)—C(9)	1.549 (7)	C(13)—C(17)	1.526 (8)
C(9)—C(10)	1.477 (8)	C(14)—C(15)	1.554 (8)
C(11)—C(12)	1.539 (8)	C(16)—C(17)	1.502 (8)
C(13)—C(18)	1.566 (8)		
C(2)—C(1)—C(10)	114.4 (5)	C(4)—C(5)—C(10)	122.2 (5)
C(3)—C(4)—C(5)	115.4 (4)	C(6)—C(5)—C(10)	124.0 (5)
C(4)—C(5)—C(6)	113.8 (4)	C(7)—C(8)—C(9)	109.9 (4)
C(5)—C(6)—C(7)	112.2 (4)	C(8)—C(9)—C(11)	120.4 (5)
C(9)—C(8)—C(14)	110.8 (4)	C(10)—C(9)—C(11)	123.2 (5)
C(8)—C(9)—C(10)	116.3 (4)	C(1)—C(10)—C(9)	118.3 (5)
C(5)—C(10)—C(9)	121.1 (5)	C(9)—C(11)—C(12)	126.6 (5)
C(1)—C(10)—C(5)	120.6 (5)	C(12)—C(13)—C(18)	110.6 (5)
C(11)—C(12)—C(13)	109.2 (4)	C(12)—C(13)—C(14)	109.8 (4)
C(17)—C(13)—C(18)	106.8 (5)	C(14)—C(13)—C(18)	111.3 (4)
C(14)—C(13)—C(17)	101.6 (6)	C(8)—C(14)—C(13)	113.1 (4)
C(13)—C(17)—C(16)	109.8 (5)	O(3)—C(17)—C(16)	123.7 (6)
O(3)—C(17)—C(13)	126.5 (5)		

C(10)—C(1)—C(2)—C(3)	−44.8 (6)
C(1)—C(2)—C(3)—C(4)	58.5 (6)
C(2)—C(3)—C(4)—C(5)	−42.0 (6)
C(9)—C(11)—C(12)—C(13)	15.8 (8)
C(8)—C(9)—C(11)—C(12)	−0.3 (9)
C(14)—C(8)—C(9)—C(11)	15.8 (7)
C(3)—C(4)—C(5)—C(10)	10.9 (8)
C(4)—C(5)—C(10)—C(1)	3.1 (8)
C(2)—C(1)—C(10)—C(5)	14.3 (7)
C(8)—C(9)—C(10)—C(5)	8.0 (7)
C(6)—C(5)—C(10)—C(9)	4.5 (8)
C(10)—C(5)—C(6)—C(7)	15.4 (7)
C(5)—C(6)—C(7)—C(8)	−46.9 (6)
C(6)—C(7)—C(8)—C(9)	58.4 (6)
C(7)—C(8)—C(9)—C(10)	−39.6 (6)
C(9)—C(8)—C(14)—C(13)	−47.5 (6)
C(12)—C(13)—C(14)—C(8)	64.9 (6)
C(11)—C(12)—C(13)—C(14)	−45.0 (6)
C(14)—C(13)—C(17)—C(16)	26.9 (6)
C(17)—C(13)—C(14)—C(15)	−39.4 (5)
C(13)—C(14)—C(15)—C(16)	37.3 (5)
C(14)—C(15)—C(16)—C(17)	−20.4 (6)
C(15)—C(16)—C(17)—C(13)	−4.1 (7)

The structure was refined by full-matrix least squares. H atoms were located from difference Fourier maps; they were positioned geometrically and included as riding atoms with fixed isotropic displacement parameters in the structure-factor calculations.

Data collection: Siemens diffractometer software (P3 4.21/V). Cell refinement: Siemens diffractometer software (P3 4.21/V). Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: VJ1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Dideoxy-D-*erythro*-hex-2-enono-1,5-lactone and 1,4-Lactone Obtained from Tri-*O*-acetyl-D-glucal

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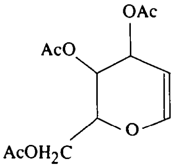
Abstract

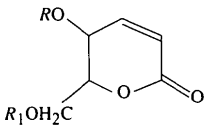
In the 1,5-lactone compound, $C_6H_8O_4$, the lactone ring adopts a conformation between sofa and half-chair. The five-membered ring in the 1,4-lactone, $C_6H_8O_4$, is planar and the side chain is in a staggered conformation. The crystal structure of the 1,5-lactone is stabilized by both O—H···O and C—H···O hydrogen bonds and that of the 1,4-lactone by O—H···O hydrogen bonds.

Comment

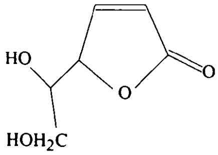
The title compounds have been prepared in high yield from tri-*O*-acetyl-D-glucal (1). Treatment of (1) with pyridinium chlorochromate in dichloroethane gave the acetate form of the δ-lactone, (2*a*) (R = R_1 = Ac) (Rollin & Sinay, 1981). This diacetate lactone, when treated with titanium tetraisopropoxide in 2-propanol for 72 h, gave 2,3-dideoxy-D-*erythro*-hex-2-enono-1,5-lactone (2*b*) (R = R_1 = H) and some of the γ-lactone (3). Prolonging the reaction for a further 24 h gave mainly the γ-lactone, 2,3-dideoxy-D-*erythro*-hex-2-enono-1,4-lactone, (3). Both the lactones are substructures for many natural products and have been synthesized with a view to elaborating them into some of these natural products (El-Zayat *et al.*, 1985). The crystal structure determination of these compounds was carried out to confirm the structures proposed from spectroscopic data.



(1)



(2*a*) $R = R_1$ = Ac
(2*b*) $R = R_1$ = H



(3)

Fig. 1 shows an *ORTEP* (Johnson, 1965) plot and the numbering scheme of the δ-lactone. The bond lengths and angles observed in this structure are comparable with those of other related naturally occurring lactones, such as asperlin, asperlinol (Fukuyama, Katsube, Noda, Hamasaki & Hatsuda, 1978) and goniotriol (Alkofahi, Ma, McKenzie, Byrn & McLaughlin, 1989). In general, the observed bond lengths are shorter than the normal values, *e.g.* the C1—C2 single-bond distance of 1.469 (3) Å is much less than the usual value and may be attributed to the effect of the neighbouring C=O and C=C bonds. The δ-lactone ring adopts a conformation in between sofa and half-chair, the corresponding asymmetry parameters are ΔC_s(C2) = 0.099 (1) and ΔC_2(C1—C2) = 0.045 (1) (Nardelli, 1983*a*), with more tendency towards half-chair conformation. This intermediate conformation of the lactone ring is observed in asperlin and in the chloro-derivative of asperlinol, but the sofa conformation is favoured in asperlinol and goniotriol. The two hydroxyl groups

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