used to solve structure: *SIR*93 (Altomare *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1172). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Synthesis and Relative Configuration of (±)-(1α,2β,3β,4α)-1,2,3,4-Cyclopentanetetrol 1-Acetate

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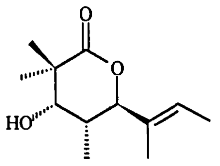
Abstract

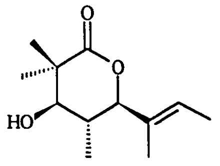
The title compound (alternative IUPAC name: 2,3,4-trihydroxycyclopentyl acetate, $C_7H_{12}O_5$) is a precursor in the reaction pathway of the synthesis of helicascolides. The cyclopentane ring adopts a twist conformation with the two hydroxy groups in β positions. The molecules are linked by a network of hydrogen bonds.

Comment

Helicascolides A, (1), and B, (2), are two isomeric δ-lactones isolated from the Hawaiian mangrove ascomycete *Heliscascus kanaloanus* (Poch & Gloer, 1989), a marine fungus. Metabolites with these kinds of structures have, to the best of our knowledge, never been obtained from terrestrial fungi. Marine microorganisms are therefore a promising source of new potentially bioactive substances (Kobayashi & Ishibashi, 1993).

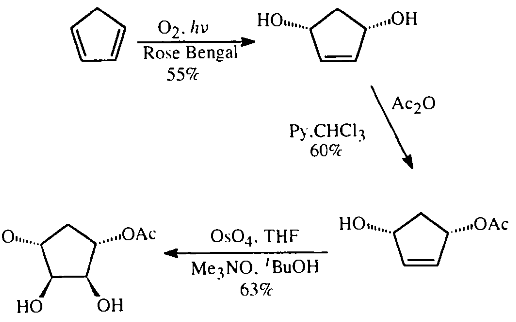


(1)



(2)

The title compound, (3), is a precursor in the reaction pathway to the synthesis of heliscacolides A and B. As shown in the reaction scheme below, (3) was obtained stereoselectively from cyclopentadiene. The enantioselective synthesis of (3), obtained as a clear unstable oil, has been described by Deardorff, Shambayati, Myles & Heerding (1988). In order to determine unambiguously its molecular conformation, which will be of aid in the prediction of the steric course of subsequent reactions, a crystal structure determination was undertaken.



(3)

As shown in Fig. 1, only the compound with two hydroxy groups in β positions was obtained. This result may be ascribed to steric effects due to the voluminous intermediate osmate ester. The cyclopentane ring is in a ^3_4T conformation as shown by the Cremer & Pople (1975) puckering parameters, q_2 = 0.421 (3) Å, Φ_2 = 91.7 (4)°. The molecules are linked by a network of hydrogen bonds: $O1\cdots O3^{i}$ = 2.691 (2), $HO1\cdots O3^{i}$ = 1.83 (4) Å, $O1—HO1\cdots O3^{i}$ = 173 (3)°; $O2\cdots O1^{ii}$ = 2.694 (2), $HO2\cdots O1^{ii}$ = 1.89 (4) Å, $O2—HO2\cdots O1^{ii}$ = 178 (3)°; $O3\cdots O2^{iii}$ = 2.708 (3), $HO3\cdots O2^{iii}$ = 1.88 (4) Å, $O3—HO3\cdots O2^{i}$ = 171 (3)° [symmetry codes: (i) $x - 1, y, z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, z$; (iii) $\frac{1}{2} + x, y, \frac{1}{2} - z$].

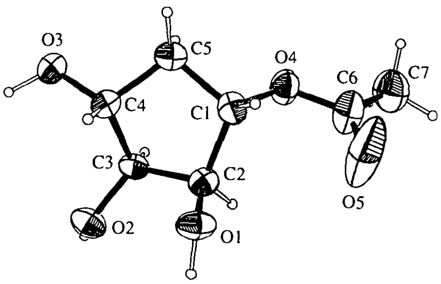


Fig. 1. Projection of the structure of $C_7H_{12}O_5$ showing the atom labelling. 50% probability displacement ellipsoids are shown for the non-H atoms; H atoms are at an arbitrary scale.

Experimental

Crystal data

$C_7H_{12}O_5$
$M_r = 176.17$
Orthorhombic
$Pbca$
$a = 6.374\ (1)$ Å
$b = 9.917\ (2)$ Å
$c = 26.743\ (3)$ Å
$V = 1690.6\ (8)$ Å^3
$Z = 8$
$D_x = 1.384$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–19°
$\mu = 0.119$ mm^{-1}
$T = 293$ K
Irregular
0.70 × 0.50 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1483 measured reflections
1483 independent reflections
1155 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 24.97°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 31$
2 standard reflections
frequency: 30 min
intensity decay: 0.9%

Refinement

Refinement on F^2
$R(F) = 0.0472$
$wR(F^2) = 0.1185$
$S = 1.068$
1464 reflections
147 parameters
Only coordinates of H atoms refined, one common U_{iso}
$w = 1/[\sigma^2(F_o^2) + (0.0590P)^2 + 1.4987P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.396$ e Å^{-3}
$\Delta\rho_{min} = -0.363$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.025 (3)
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)

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

	x	y	z	U_{eq}
O1	0.1679 (3)	0.6796 (2)	0.31650 (7)	0.0402 (5)
O2	0.3596 (3)	0.9179 (2)	0.29021 (7)	0.0393 (5)
O3	0.8140 (3)	0.8238 (2)	0.30423 (7)	0.0424 (5)
O4	0.4326 (3)	0.6540 (2)	0.43263 (6)	0.0424 (5)
O5	0.1284 (5)	0.5480 (4)	0.43685 (10)	0.1123 (13)
C1	0.4303 (4)	0.6386 (2)	0.37875 (9)	0.0342 (6)
C2	0.2899 (4)	0.7446 (2)	0.35376 (9)	0.0318 (6)
C3	0.4456 (4)	0.8431 (2)	0.33027 (8)	0.0290 (5)
C4	0.6250 (3)	0.7526 (2)	0.31452 (9)	0.0326 (6)
C5	0.6524 (4)	0.6620 (3)	0.35969 (10)	0.0421 (7)
C6	0.2705 (5)	0.6027 (3)	0.45750 (11)	0.0537 (8)
C7	0.2841 (7)	0.6289 (5)	0.51191 (13)	0.0703 (10)
HO1	0.059 (5)	0.729 (3)	0.3103 (12)	
HO2	0.348 (5)	0.996 (4)	0.2978 (12)	
HO3	0.814 (5)	0.853 (3)	0.2748 (13)	

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

O1—C2	1.419 (3)	C1—C5	1.523 (4)
O2—C3	1.414 (3)	C1—C2	1.534 (3)
O3—C4	1.423 (3)	C2—C3	1.528 (3)
O4—C6	1.330 (3)	C3—C4	1.513 (3)
O4—C1	1.449 (3)	C4—C5	1.516 (3)
O5—C6	1.191 (4)	C6—C7	1.481 (5)
C6—O4—C1	116.7 (2)	C4—C3—C2	103.1 (2)
O4—C1—C5	107.9 (2)	O3—C4—C3	113.6 (2)
O4—C1—C2	111.5 (2)	O3—C4—C5	110.5 (2)
C5—C1—C2	107.0 (2)	C3—C4—C5	102.5 (2)
O1—C2—C3	111.0 (2)	C4—C5—C1	104.5 (2)
O1—C2—C1	108.3 (2)	O5—C6—O4	122.2 (3)
C3—C2—C1	103.8 (2)	O5—C6—C7	125.4 (3)
O2—C3—C4	113.2 (2)	O4—C6—C7	112.3 (3)
O2—C3—C2	113.3 (2)		

The H atoms were placed in the positions found in difference Fourier maps and refined with an overall isotropic displacement parameter that converged to $U_{iso} = 0.061\ (3)$ Å^2.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1141). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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