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(+)-Deacetylhexahydromycoepoxydiene

Hai-Yan Chen,[a] Yong-Cheng Lin,[a]* L. L. P. Vrijmoed[b] and E. B. Gareth Jones[c]

[a]School of Chemistry and Chemical Engineering, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China, [b]Department of Biology and Chemistry, City University of Hong Kong, People's Republic of China, and [c]National Center for Genetic Engineering and Biotechnology, Bangkok, Thailand

Correspondence e-mail: ceslyc@zsu.edu.cn

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The title compound, $C_{14}H_{22}O_3$, was isolated from the ammonium formate/10% Pd—C-catalysed hydrogen-transfer reaction of mycoepoxydiene. In the reaction product, the stereochemistry of the chiral carbons C-1, C-6, C-7 and C-8 remains unchanged, and the conformation of the δ-lactone ring is a chair.

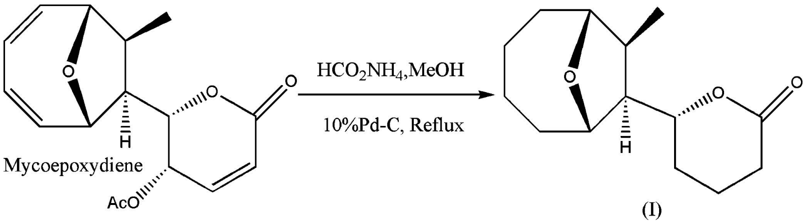
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.097
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In recent years, published reviews clearly indicate the tremendous potential of marine microorganisms as a source of new pharmaceuticals (Pietra, 1997; Fusetani, 2000; Faulkner, 2002, and references therein). Our research has focused on the secondary metabolites of marine fungi on the South China Sea coast (Lin *et al.*, 2001).



(I)

Mycoepoxydiene was isolated from the solid-state fermentation of a rare fungus designated as OSF66617 (Cai *et al.*, 1999) and later from the liquid-state fermentation of a marine entophytic fungus (No. 1893) (Chen, 2003). The first asymmetric total synthesis of mycoepoxydiene has been accomplished (Takao *et al.*, 2004). Mycoepoxydiene has been found to show cytotoxicity toward human tumor cells *in vitro* (Takao *et al.*, 2004). We have prepared hexahydromycoepoxydiene by a catalytic hydrogenation reaction (Chen *et al.*, 2005). Now we report the crystal structure of the title compound, (I), which is the hexahydrodeoxy product of mycoepoxydiene by a catalytic hydrogen-transfer reaction.

The absolute configuration of mycoepoxydiene was confirmed by the asymmetric total synthesis (Takao *et al.*, 2004). In the synthesis of (I), the chiral C atoms were not involved except C12. Thus, (I) retained the *R* configuration at C1, C7 and C10, and the *S* configuration at C6 and C8 (Fig. 1).

Table 1 lists selected parameters for the title compound. The title compound contains no classical hydrogen bonds.

Experimental

A solution of mycoepoxydiene (0.1 mmol) and ammonium formate (0.60 mmol) in absolute methanol (10 ml) with 10% Pd—C (0.29 mg) was heated under reflux for 12 h (reaction monitorized by thin-layer

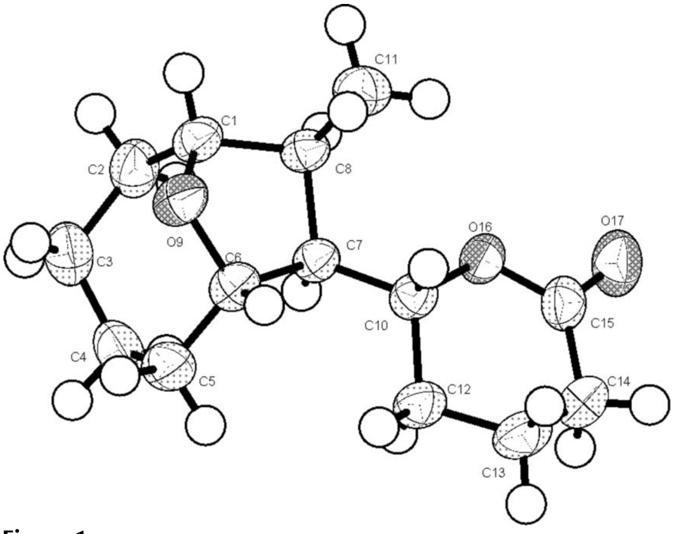


Figure 1
View of the title compound, (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level (Spek, 2003).

chromatography). The catalyst was filtered off, washed with fresh methanol, and recrystallized from acetonitrile (yield 90%); $[\alpha]^{20}_{D}$ +207° (c 0.027, MeOH).

Crystal data

$C_{14}H_{22}O_3$
$M_r = 238.32$
Orthorhombic, $P2_12_12_1$
$a = 6.8200\ (12)$ Å
$b = 6.9277\ (12)$ Å
$c = 27.431\ (5)$ Å
$V = 1296.0\ (4)$ Å^3
$Z = 4$
$D_x = 1.221$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 999 reflections
$\theta = 3.0$–$26.8°$
$\mu = 0.08$ mm^{-1}
$T = 273\ (2)$ K
Block, colourless
0.46 × 0.40 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{\min} = 0.960$, $T_{\max} = 0.982$
6732 measured reflections

2831 independent reflections
2304 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$
$\theta_{\max} = 27.0°$
$h = -4 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -35 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.097$
$S = 1.03$
2831 reflections
156 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.1042P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O9—C1	1.4382 (19)	C10—C12	1.519 (2)
O9—C6	1.4420 (18)	C13—C12	1.511 (2)
O17—C15	1.200 (2)		
C1—O9—C6	107.05 (10)	O17—C15—C14	122.75 (15)
O17—C15—O16	117.76 (15)	O16—C15—C14	119.45 (15)
C1—C8—C7—C10	−154.06 (12)	C8—C7—C10—C12	−177.82 (13)
C6—C7—C10—O16	−175.15 (13)	C6—C7—C10—C12	62.48 (17)

The H atoms were initially refined with soft restraints to regularize their geometry, after which they were refined with riding constraints only: C—H = 0.96–0.98 Å and $U_{\rm iso}(\rm H) = 1.2U_{\rm eq}(\rm C)$. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *enCIFer* (CCDC, 2004).

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