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5,8-Dihydroxy-10-methyl-5,8,9,10-tetrahydro-2*H*-oxecin-2-one

Hoong-Kun Fun,[a]* Weraphol Bhilabutra,[b] Pittaya Tuntiwachwuttikul[c] and Suchada Chantrapromma[d]‡

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of Biology, Faculty of Science, Chaing Mai University, Muang, Chaing Mai 50002, Thailand, [c]Department of Chemistry, Faculty of Science, Silpakorn University, Salaya, Nakorn Pathom 73000, Thailand, and [d]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

‡ Additional correspondence author, email: suchada.c@psu.ac.th

Correspondence e-mail: hkfun@usm.my

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Key indicators

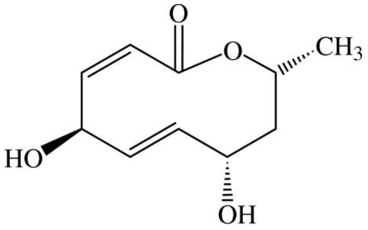
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.112
Data-to-parameter ratio = 10.2

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

The title compound, $C_{10}H_{14}O_4$, known as modiolide A, is a ten-membered macrolide which has been isolated for the first time from the endophylic fungus *Periconia siamensis* (CMUGE015), obtained from the leaves of *Thysanoleana lafifolia*. The two disubstituted olefinic groups are in *Z* and *E* configurations. Intermolecular O—H···O hydrogen bonds link the molecules into double-layer sheets parallel to the *ab* plane. The crystal packing is further stabilized by weak C—H···O interactions.

Comment

In the course of screening for biologically active substances and new metabolites from fungi, the title compound, (I), has been isolated for the first time from the endophylic fungus, *Periconia siamensis* (CMUGE015) obtained from the leaves of *Thysanoleana lafifolia* collected in the Suthep-Pui National Park, Chiang Mai, in the northern part of Thailand. The title compound has been isolated previously in a colourless oil form from the cultured broth of the fungus *Paraphaeosphaeria* sp. (strain N-119), derived from a marine horse mussel *Modiolus auriculatus* (Tsuda *et al.*, 2003), but no X-ray crystal structure determination was reported. We report here the crystal structure of (I).



(I)

In (I) (Fig. 1), the two disubstituted olefinic groups C2═C3 [1.328 (2) Å] and C5═C6 [1.332 (2) Å] exist in *Z* and *E* configurations, respectively. The ten-membered ring has a total puckering parameter (Cremer & Pople, 1975) θ = 1.019 (2) Å. The geometry of the ester carbonyl and methyl groups is characterized by a dihedral angle of 81.70 (17)° between the O1/O2/C1/C2 and C7/C8/C9/C10 mean planes. The absolute configurations at C4, C7 and C9 were assigned on the basis of the reported molecular structure (Tsuda *et al.*, 2003), whose chirality was determined by the exciton chirality method (Harada & Nakanishi, 1983) using the *p*-methoxycinnamoyl chloride treatment. The two hydroxyl groups attached at C4 and C7, and the methyl substituent at C9, have *R*, *S* and *R* configurations, respectively. All bond lengths and angles (Table 1) in (I) show normal values (Allen *et al.*, 1987).

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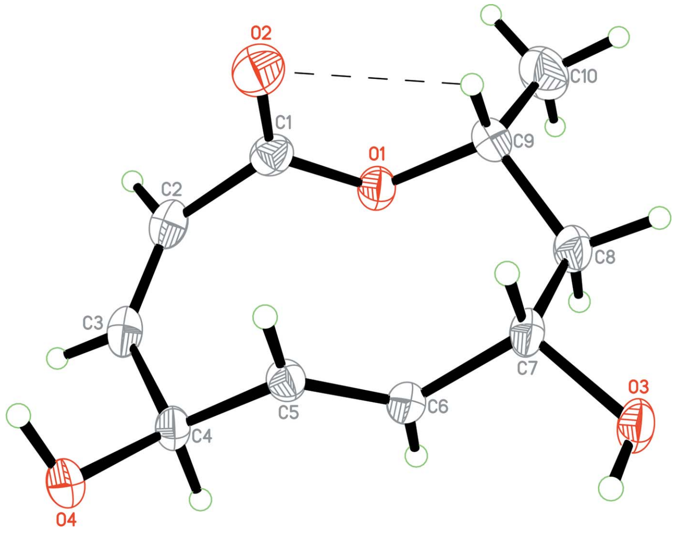

Figure 1
View of (I), showing the atomic numbering and 60% probability displacement ellipsoids. The dashed line indicates an intramolecular C—H···O interaction.

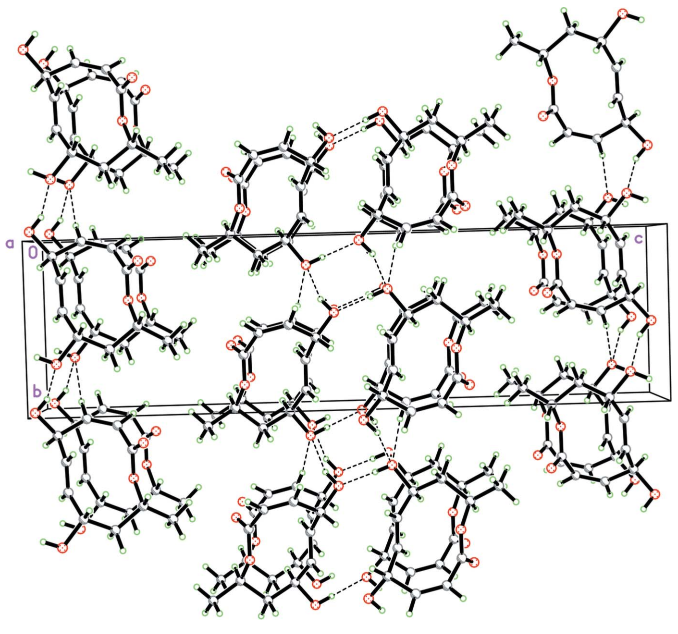

Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

Carbonyl atom O2 is involved in a weak intramolecular C—H···O interaction (Table 1 and Fig. 1). The hydroxyl groups participate in intermolecular O—H···O hydrogen bonds (Table 1), which link the molecules into double-layer sheets parallel to the *ab* plane. The crystal packing (Fig. 2) is further stabilized by weak C—H···O interactions (Table 1).

Experimental

Periconia siamensis (CMUGE015) was grown on 100 plates of F1 medium and incubated for 14 d at 301 K. The culture medium was then cut into small pieces and extracted with EtOAc (93 × 800 ml). The residue (2.8 g) obtained after evaporation of the solvent was subjected to column chromatography over silica gel to afford (I) (178 mg). Colourless plate-shaped single crystals of (I) were obtained by recrystallization from EtOAc over several days (m.p. 462–464 K).

Crystal data

$C_{10}H_{14}O_4$
$M_r = 198.21$
Orthorhombic, $P2_12_12_1$
$a = 4.9431$ (1) Å
$b = 7.5220$ (1) Å
$c = 27.2779$ (5) Å
$V = 1014.25$ (3) Å^3
$Z = 4$
$D_x = 1.298$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 100.0$ (1) K
Plate, colourless
0.52 × 0.38 × 0.13 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.950$, $T_{\max} = 0.987$
28044 measured reflections
1756 independent reflections
1694 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.112$
$S = 1.25$
1756 reflections
173 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.1643P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1*O3*···O4$^{\rm i}$	0.83 (3)	1.91 (3)	2.7344 (18)	177 (3)
O4—H1*O4*···O3$^{\rm ii}$	0.86 (3)	1.94 (3)	2.7563 (18)	158 (3)
C3—H3···O3$^{\rm iii}$	0.96 (2)	2.44 (2)	3.393 (2)	173 (18)
C9—H9···O2	0.97 (3)	2.26 (3)	2.743 (2)	110 (2)

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

Methyl H atoms were placed in calculated positions, with C—H distances of 0.96 Å and refined as riding, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. The remaining H atoms were located in a difference map and refined isotropically. The 1194 Friedel pairs were merged before the final refinement as there is no significant anomalous dispersion for the determination of the absolute configuration.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12a) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Harada, N. & Nakanishi, K. (1983). *Circular Dichroic Spectrometry – Exciton Coupling in Organic Stereochemistry*. Milley Valley, CA: University Sciences Books.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tsuda, M., Mugishima, T., Komatsu, K., Sone, T., Tanaka, M., Mikami, Y. & Kobayashi, J. (2003). *J. Nat. Prod.* **66**, 412–415.