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(1*R**,4*R**,5*S**,12*S**,13*R**)-9-Acetoxycembra-8(*E*),15(17)-dien-16,4-olide monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.111
Data-to-parameter ratio = 10.1

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

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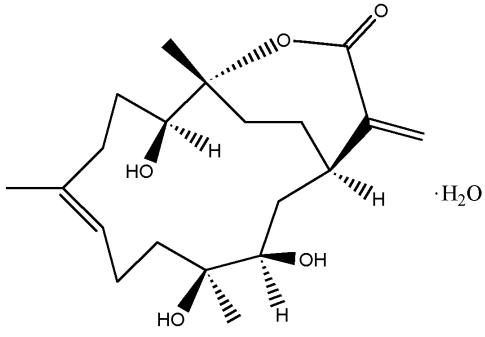
The title compound, $C_{20}H_{32}O_5.H_2O$, crystallizes in the non-centrosymmetric space group *C*2. The molecular skeleton consists of an irregular 14-membered ring and a nearly twist–boat seven-membered γ-lactone ring with a *trans* connection. In the solid-state structure, cembradienolide and water molecules form an $R_2^2(7)$ motif through O—H···O*W* and O*W*—H···O hydrogen bonds, while adjacent molecules form an infinite chain, described by graph-set motif $C_2^2(12)$, along the *c* axis *via* O—H···O hydrogen bonds.

Comment

(1*R**,4*R**,5*S**,12*S**,13*R**)-9-Acetoxycembra-8(*E*),15(17)-dien-16,4-olide monohydrate, (I), was isolated from the soft coral of *Sinularia microclavata Tix-Dur*, which was collected off the Bay of Sanya, Hainan Island, China. The Cembranolide diterpene has been previously isolated from the soft coral *Sinularia capillosa Tix-Dux* (Lin *et al.*, 2002) and *sinularia tenella* (Su *et al.*, 2000). Some cembranolide diterpenes from *Sinularia* genuses exhibit bio-activity (Anjaneylu *et al.*, 1996; Su *et al.*, 2001), while (I) exhibits cytotoxicity against P388 and L1210 cell lines, with ED50 values of 15.0 and 18.5 μg ml^{-1} respectively. Its structure has been elucidated on the basis of spectroscopic methods and the single-crystal X-ray structure of the methanol solvate of (I) (Lin *et al.*, 2002). We report here the crystal structure of monohydrate (I).



(I)

This X-ray study of (I) confirms the structure previously proposed on the basis of spectroscopic data. There is one molecule of (I) and one solvent molecule of water in the asymmetric unit. In the crystal structure, cembradienolide and water molecules form an $R_2^2(7)$ motif (Etter, 1990) through O2—H2···O1*W* and O1*W*—H2*C*···O3 hydrogen bonds (Fig. 1 and Table 2), while adjacent cembradienolide molecules extend in an infinite chain along the *c* axis, described as $C_2^2(12)$, *via* O1—H1···O2 and O3—H3···O4 hydrogen bonds (Fig. 2 and Table 1)

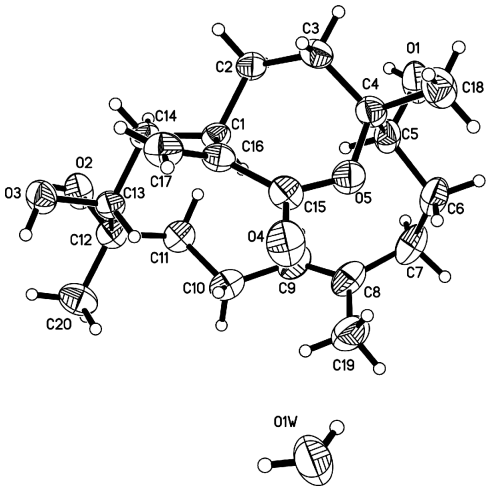


Figure 1
View of the title compound, showing 30% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

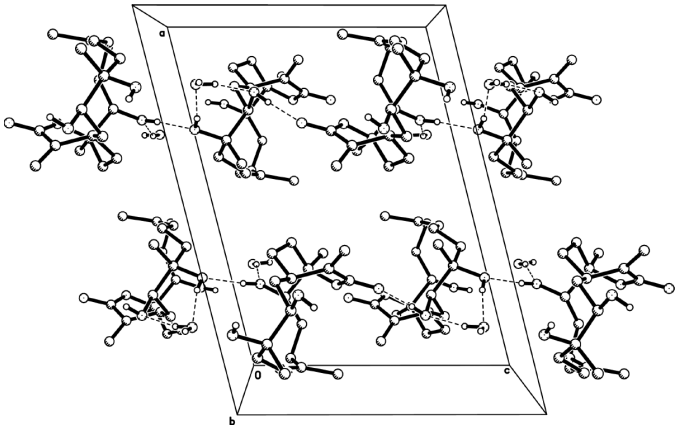


Figure 2
The molecular packing of the title compound, viewed along the *b* axis. All H atoms attached to C atoms have been omitted for clarity.

The molecular skeleton is a bicyclic system with a *trans* connection. The relative molecular configuration was determined as 1R*,4*R**,5*S**,12*S**,13*R**, as shown in Table 1. The 14-membered ring has an irregular conformation, as illustrated in Table 1, which is similar to the previously published results (Lin *et al.*, 2002). The conformation of the seven-membered γ-lactone ring is a near twist-boat with a C15—O5—C4—C3 torsion angle of 2.1 (4)° (Table 2). The conjugation of the exocyclic methylene and the carbonyl group is somewhat perturbed by the O5—C15—C16—C17 torsion angle of −150.8 (3)°, with the result that this compound exhibits bioactivity (Su *et al.*, 2000).

Experimental

Compound (I) was isolated from the soft coral of *Sinularia microclavata Tix-Dur*, which was collected off the Bay of Sanya, Hainan Island, China. The soft coral was extracted with EtOH at room temperature to give a light brown gum. The brown gum was partitioned between ethyl acetate and water. The EtOAc extract was chromatographied on silica-gel column by elution with EtOAc and petroleum. A fraction eluted with EtOAc–petroleum ether (volume ration 4:1) containing cembranolide diterpene (I) was obtained. Crystals of the title compound were obtained by slow evaporation from EtOAc–petroleum ether (4:1).

Crystal data

$C_{20}H_{32}O_5\cdot H_2O$
M_r = 370.47
Monoclinic, *C*2
a = 15.6359 (17) Å
b = 12.1299 (17) Å
c = 11.5236 (14) Å
β = 104.111 (3)°
V = 2119.6 (5) Å^3
Z = 4
D_x = 1.161 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 990 reflections
θ = 2.6–26.9°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colorless
0.50 × 0.50 × 0.48 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.959, T_{max} = 0.961
9058 measured reflections
2451 independent reflections
2072 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.2°
h = −20 → 19
k = −15 → 15
l = −14 → 14

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.07
2451 reflections
243 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.3649P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected torsion angles (°).

C16—C1—C2—C3	37.0 (3)	C10—C11—C12—C13	68.8 (3)
C14—C1—C2—C3	163.2 (2)	C11—C12—C13—C14	54.2 (2)
C1—C2—C3—C4	48.3 (3)	C16—C1—C14—C13	−63.2 (2)
C2—C3—C4—C5	50.2 (3)	C2—C1—C14—C13	172.63 (19)
C3—C4—C5—C6	−173.9 (2)	C4—O5—C15—O4	−157.9 (3)
C4—C5—C6—C7	138.2 (3)	C4—O5—C15—C16	26.2 (4)
C5—C6—C7—C8	−67.5 (4)	O5—C15—C16—C17	−150.8 (3)
C6—C7—C8—C9	106.0 (3)	O5—C15—C16—C1	31.0 (3)
C7—C8—C9—C10	−171.9 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.86	2.673 (3)	173
O2—H2···O1*W*^{ii}	0.82	1.95	2.755 (4)	167
O3—H3···O4^{ii}	0.82	1.96	2.774 (3)	175
O1*W*—H1*B*···O1^{ii}	0.83	1.99	2.769 (3)	156
O1*W*—H2*C*···O3^{iii}	0.86	1.99	2.834 (3)	168

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

H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with C—H distances in the range of 0.93–0.98 Å with U_{iso}(H) = 1.2U_{eq}(C), and O—H distances of 0.82 Å with U_{iso}(H) = 1.5U_{eq}(O). Water H atoms were positioned and fixed during the refinement. In the absence of significant anomalous scattering, the Friedel opposites were merged during the refinement. The crystal packing allows a solvent-accessible void of 48 Å^3.

Data collection: *SMART* (Bruker, 1998); cell refinement:*SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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