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A furodysinin lactone derivative from the marine sponge *Dysidea fragilis*

K. Ravikumar,[a]* S. Selvanayagam,[b] T. Venkateshwar Goud,[c] P. Krishnaiah[c] and Y. Venkateswarlu[c]

[a]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]Natural Products Laboratory, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: ravikumar_iict@yahoo.co.in

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.104
Data-to-parameter ratio = 11.0

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

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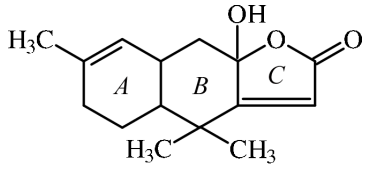
The title compound, 4a,5,6,8a,9,9a-hexahydro-9a-hydroxy-4,4,7-trimethylnaphtho[2,3-*b*]furan-2(4*H*)-one, $C_{15}H_{20}O_3$, is a sesquiterpenoid isolated from the marine sponge *Dysidea fragilis*. The furodysinin lactone skeleton consists of two six-membered rings, which adopt chair and half-chair conformations, and a furan ring. The molecules are arranged in a helical pattern through O—H···O hydrogen bonds.

Comment

In the course of a search for bioactive drugs from marine sponges, *Dysidea fragilis* (montagu) (family Dysideidae), which is widespread on the southeast coast of India, was selected as it has been reported that furodysinin lactone acts as a potent agonist to human leukotriene B4 receptor (Mong *et al.*, 1990). The isolation of furodysinin lactone was reported by Gorde & Cardellina (1984), and its relative stereochemistry was established, based on the sign of optical activity (Horton *et al.*, 1990). Spectroscopic analysis suggests that the title compound, (I), is a closely related derivative of furodysinin lactone. In order to establish the structure of (I), an X-ray analysis was undertaken and the results are presented here.



(I)

The structure of (I) with the atom-numbering scheme is shown in Fig. 1. The molecular framework of the furodysinin lactone consists of three fused rings, two six- and one five-

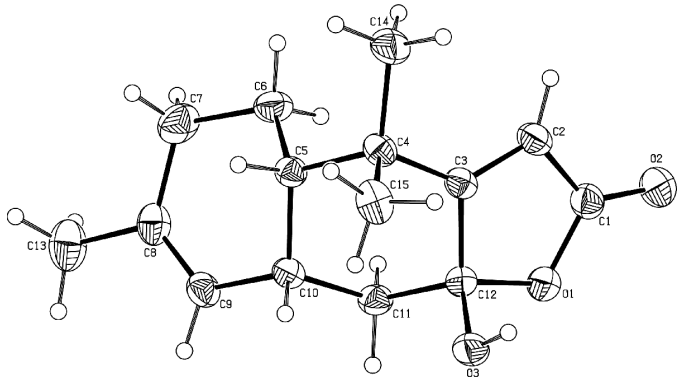


Figure 1
View of (I), showing 30% probability displacement ellipsoids.

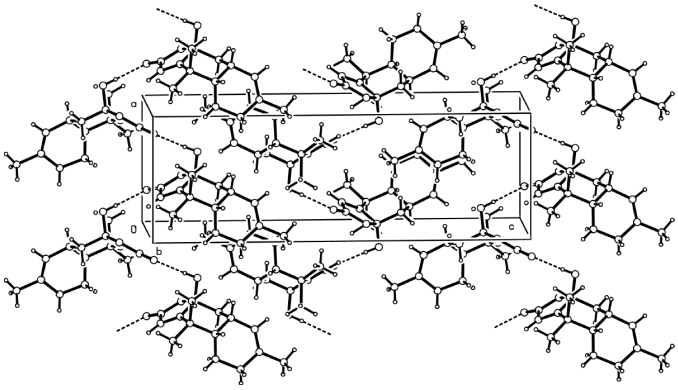


Figure 2
Packing diagram viewed approximately along the *b* axis, showing the helical arrangement of the molecules. Dashed lines denote O—H···O hydrogen bonds.

membered. Bond lengths and angles in (I) are normal (Table 1).

The conformation of ring *A* is a half-chair, with asymmetry parameter ΔC_2(C5—C6) = 0.038 (1) (Nardelli, 1983). Ring *B* adopts a chair conformation, with puckering parameters q_2 = 0.031 (2) Å, q_3 = 0.536 (2) Å = Q_T, θ = 3.3 (2)° (Cremer & Pople, 1975). Atoms C3 and C10 are displaced from the base plane C12/C11/C5/C4 of ring *B* by 0.577 (2) and −0.647 (1) Å, respectively. Methyl atoms C15 and C14 attached to C4 occupy axial and equatorial positions [C10—C5—C4—C15 = −69.9 (2)° and C10—C5—C4—C14 = 170.5 (2)°]. The hydroxyl group attached to C12 in an axial position [C10—C11—C12—O3 = 75.7 (2)°] is displaced by 1.279 (1) Å from the base plane C12/C11/C5/C4. The dihedral angle between the lactone ring and the base plane of ring *B* is 54.1 (1)°.

The crystal packing is stabilized by a network of intermolecular O—H···O hydrogen bonds involving the hydroxyl group and the carbonyl atom O2 (Table 2). These hydrogen bonds link the molecules in a helical fashion along the *a* axis (Fig. 2).

Experimental

An ethanol extract of *Dysidea fragilis* was chromatographed on a Sephadex LH-20 column (1:1 methanol–dichloromethane) followed by silica gel chromatography, eluting with hexane/ethyl acetate. Crystals of the title compound were obtained by slow evaporation of a hexane/acetone (1:1) solution.

Crystal data

$C_{15}H_{20}O_3$
M_r = 248.31
Orthorhombic, $P2_12_12_1$
a = 7.1736 (7) Å
b = 8.7391 (9) Å
c = 21.301 (2) Å
V = 1335.4 (2) Å^3
Z = 4
D_x = 1.235 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4309 reflections
θ = 2.5–27.7°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.980, T_{max} = 0.987
8149 measured reflections
1837 independent reflections
1705 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.0°
$h = -8 \rightarrow 9$
$k = -10 \rightarrow 11$
$l = -27 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.13
1837 reflections
167 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.1311P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.349 (2)	C3—C12	1.508 (2)
O1—C12	1.475 (2)	C4—C15	1.544 (3)
O2—C1	1.205 (2)	C5—C10	1.543 (2)
O3—C12	1.379 (2)	C8—C9	1.316 (3)
C1—C2	1.463 (3)	C11—C12	1.510 (2)
C2—C3	1.325 (2)		
O2—C1—O1	121.3 (2)	C8—C9—C10	124.8 (2)
O2—C1—C2	129.5 (2)	C9—C10—C5	110.4 (2)
C2—C3—C4	131.1 (2)	O3—C12—O1	108.2 (2)
C2—C3—C12	109.3 (2)	O3—C12—C11	109.4 (1)
C9—C8—C7	121.9 (2)	O3—C12—C3	115.2 (2)
C12—C3—C4—C15	66.8 (2)	C8—C9—C10—C5	19.8 (3)
C3—C4—C5—C10	49.5 (2)	C6—C5—C10—C9	−49.2 (2)
C14—C4—C5—C10	170.5 (2)	C4—C5—C10—C11	−54.7 (2)
C15—C4—C5—C10	−69.9 (2)	C5—C10—C11—C12	55.1 (2)
C10—C5—C6—C7	61.4 (2)	C10—C11—C12—O3	75.7 (2)
C5—C6—C7—C8	−41.5 (3)	C10—C11—C12—C3	−52.0 (2)
C6—C7—C8—C9	10.6 (3)	C4—C3—C12—C11	54.8 (2)
C7—C8—C9—C10	0.2 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	2.00	2.812 (2)	174

Symmetry code: (i) $\frac{1}{2}+x, \frac{3}{2}-y, -z$.

The H atoms were positioned geometrically and treated as riding on their parent C and O atoms [C—H = 0.93–0.98 Å, O—H = 0.82 Å; U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms]. Due to the lack of significant anomalous scattering, the absolute configuration was not determined by X-ray diffraction and Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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