C(3)	0.2545 (6)	0.2515 (5)	0.6498 (4)	0.088 (13)
C(4)	0.5797 (4)	0.3745 (3)	0.7078 (2)	0.064 (9)
C(5)	0.7280 (5)	0.4197 (5)	0.6454 (3)	0.086 (12)
C(6)	0.0897 (5)	1.0896 (3)	0.8895 (2)	0.051 (8)
N(2)	0.0953 (4)	1.1883 (3)	0.9438 (2)	0.061 (8)
C(7)	0.2441 (5)	1.1823 (4)	0.9961 (2)	0.071 (11)
C(8)	0.3840 (6)	1.2514 (5)	0.9442 (4)	0.102 (15)
C(9)	−0.0534 (6)	1.3101 (4)	0.9532 (3)	0.076 (11)
C(10)	−0.0473 (7)	1.4341 (5)	0.8803 (3)	0.084 (12)
N(3)	−0.1833 (3)	0.9373 (2)	0.6656 (2)	0.048 (6)
C(11)	−0.3175 (4)	0.8707 (4)	0.6766 (2)	0.066 (9)
C(12)	−0.4403 (4)	0.8919 (4)	0.6137 (2)	0.063 (9)
C(13)	−0.4338 (3)	0.9866 (3)	0.5344 (2)	0.037 (6)
C(14)	−0.2938 (4)	1.0558 (3)	0.5233 (2)	0.048 (7)
C(15)	−0.1752 (4)	1.0277 (3)	0.5892 (2)	0.051 (8)

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

Te—S(1)	2.624 (1)	S(1)—C(1)	1.755 (3)
Te—S(2)	2.826 (1)	S(2)—C(1)	1.686 (3)
Te—S(3)	2.720 (1)	S(3)—C(6)	1.701 (4)
Te—S(4)	2.573 (1)	S(4)—C(6)	1.723 (3)
Te—N(3)	2.700 (2)	C(1)—N(1)	1.329 (3)
C(6)—N(2)	1.333 (5)		
S(1)—Te—S(2)	65.37 (2)	Te—S(4)—C(6)	89.9 (1)
S(3)—Te—S(4)	67.04 (2)	S(2)—Te—N(3)	74.0 (1)
Te—S(1)—C(1)	90.9 (1)	S(3)—Te—N(3)	73.6 (1)
Te—S(2)—C(1)	85.7 (1)	S(1)—C(1)—S(2)	118.0 (1)
Te—S(3)—C(6)	85.6 (1)	S(3)—C(6)—S(4)	117.4 (2)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with a packing diagram, have been deposited with the IUCr (Reference: DE1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Sesterterpene Lactone from *Petrosaspongia nigra* sp. nov.

Richard C. Cambie, Allick R. Lal and Clifton E. F. Rickard

Chemistry Department, University of Auckland, Private Bag 92019, Auckland, New Zealand

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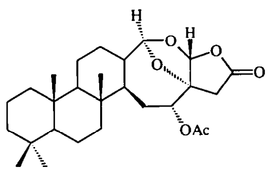
Abstract

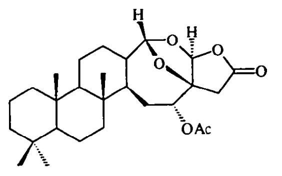
A sponge from the new genus *Petrosaspongia* has yielded two new isomeric sesterterpene lactones. We report here the structure of one of these lactones, [4a*S*-(4aα,4bβ,6aα,7β,8aα,11aβ,12β,13aβ,13bα,15aβ)]-12-acetoxyoctadecahydro-1,1,4a,13b-tetramethyl-7,11a-epoxy-2*H*-furo[2,3-*b*]phenanthro[1,2-*f*]oxocin-10(11*H*)-one, $C_{27}H_{40}O_6$.

Comment

As part of a search for biologically active compounds from South Pacific sponges, extracts from *Petrosaspongia nigra* sp. nov. yielded two sesterterpene lactones, (1) and (2). Preliminary details have been published elsewhere (Lal, Cambie, Rickard & Bergquist, 1994) in which the sponge was incorrectly assigned to the genus *Dactylospongia*. Further investigations have confirmed that the sponge belongs to a new genus (Bergquist, 1995). The NMR spectra of these compounds were almost identical with those recorded (Kernan *et al.*, 1989) for cyclization products of luffolide, a novel anti-inflammatory sesterterpene from the sponge *Luffariella* sp., but which had been assigned different structures. In order to confirm our structure assignments and relative stereochemistries, a single-crystal X-ray crystallographic analysis was carried out on our lactone, (2).



(1)



(2)

An *ORTEP*II (Johnson, 1976) diagram showing the numbering scheme is given in Fig. 1. The molecule is shown to be hexacyclic with an acetal function and a γ-lactone. The *A*, *B* and *C* rings show the usual *trans*-fused arrangement and ring *D* is also *trans* fused. An ideal *trans*-fused *A*, *B*, *C* ring system has torsion angles C3—C4—C5—C6, C2—C1—C10—C9 and C6—C7—C8—

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C14 of 180° and C1—C10—C9—C11 of 60°. The values found for (2) are −178.0 (5), 165.5 (5), −162.2 (4) and 59.6 (6)°, respectively, the angles deviating most being those associated with the methyl groups. A search of the Cambridge Crystallographic Database (Allen *et al.*, 1987) reveals 68 structures with three fused rings bearing methyl substituents at C8 and C10. Of these, only one (Manes *et al.*, 1988) does not have both ring junctions *trans*; for this structure the torsion angles are 179.9, 168.5, 84.8 and 64.2, respectively, consistent with *trans* fusion of rings *A* and *B* and *cis* fusion of rings *B* and *C*.

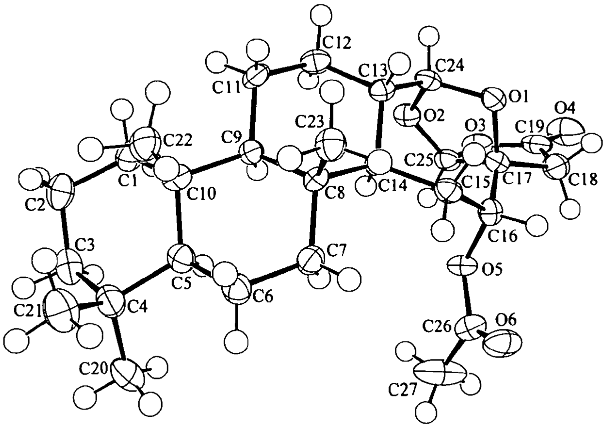

Fig. 1. The molecular structure (*ORTEPII*; Johnson, 1976) showing 50% probability ellipsoids. The configuration shown is arbitrary.

The eight-membered ring has a somewhat different geometry from that found in (8*S*,9*R*,11*R*,13*S*)-8,9:8,11:9,13-triepoxy-17-nor-8,9-secolabdane (Grant, Hanton, Tsai & Yap, 1991), which has a similar acetal system, due to the different mode of fusion of the pendant five-membered rings. In that structure, the acetal ring is fused to equivalent C atoms but the second five-membered ring is joined adjacent to the junction with ring *C* instead of opposite. The result is that the eight-membered rings show different puckering in the two structures. Least-squares-planes equations and deviations from the planes of the two five-membered rings are given in Table 3. The lactone ring, C17—C18—C19—O3—C25, has C18 0.327 Å out of the plane and approximates an envelope conformation. The ring containing the acetal functions, C17—O1—C24—O2—C25, has C24 0.316 Å from the plane and the other atoms also show significant deviations from the plane. The conformation of this ring is best described as intermediate between twist and envelope. The mode of fusion of the rings leads to a very rigid system. Bond distances and angles are all within the normally expected range (*International Tables for X-ray Crystallography*, 1962). There are no significant intermolecular interactions, the closest approach between non-H atoms being in excess of 3.2 Å.

Lactones (1) and (2) showed no biological activity in an antiviral assay and only minimal activity in antimicrobial assays. Lactone (2), however, gave an IC_{50} of 619 ng ml^{-1} in a P388 antitumor assay.

Experimental

The title compound is an extract from the sponge *Petrosaspongia nigra* sp. nov. (Lal, Cambie, Rickard & Bergquist, 1994).

Crystal data

$C_{27}H_{40}O_6$
$M_r = 460.59$
Orthorhombic
$P2_12_12_1$
$a = 8.758\,(2)$ Å
$b = 10.010\,(4)$ Å
$c = 28.466\,(6)$ Å
$V = 2495.5\,(13)$ Å^3
$Z = 4$
$D_x = 1.226$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9.2$–$10.9°$
$\mu = 0.085$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.42 × 0.15 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2520 measured reflections
2520 independent reflections
1780 observed reflections [$I > 2\sigma(I)$]

$\theta_{\max} = 24.96°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 33$
3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0564$
$wR(F^2) = 0.1183$
$S = 1.103$
2491 reflections
303 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.7355P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.505$
$\Delta\rho_{\max} = 0.209$ e Å^{-3}
$\Delta\rho_{\min} = -0.270$ e Å^{-3}
Extinction correction: none
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)*

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

	x	y	z	U_{eq}
O1	−0.2615 (4)	0.5931 (3)	0.02663 (11)	0.0304 (8)
O2	−0.3031 (4)	0.5715 (3)	0.10503 (12)	0.0308 (9)
O3	−0.2962 (4)	0.3470 (3)	0.08689 (14)	0.0394 (10)
O4	−0.3501 (5)	0.1988 (4)	0.0309 (2)	0.0605 (13)
O5	0.1000 (4)	0.5047 (3)	0.07711 (12)	0.0300 (9)
O6	0.3193 (4)	0.5588 (4)	0.03993 (14)	0.0448 (11)
C1	−0.0957 (6)	1.0581 (6)	0.2306 (2)	0.0340 (13)
C2	−0.0108 (7)	1.1297 (6)	0.2702 (2)	0.0417 (15)
C3	0.1447 (7)	1.0686 (6)	0.2793 (2)	0.044 (2)
C4	0.2490 (7)	1.0643 (6)	0.2360 (2)	0.0379 (13)
C5	0.1587 (6)	1.0001 (5)	0.1949 (2)	0.0302 (12)
C6	0.2498 (6)	0.9839 (6)	0.1495 (2)	0.0348 (13)

C7	0.1750 (6)	0.8793 (5)	0.1179 (2)	0.0308 (12)
C8	0.0069 (6)	0.9089 (5)	0.1053 (2)	0.0252 (11)
C9	−0.0812 (5)	0.9498 (5)	0.1506 (2)	0.0248 (11)
C10	−0.0053 (6)	1.0544 (5)	0.1840 (2)	0.0301 (12)
C11	−0.2490 (6)	0.9812 (5)	0.1388 (2)	0.0309 (12)
C12	−0.3273 (6)	0.8578 (5)	0.1187 (2)	0.0333 (13)
C13	−0.2407 (5)	0.7940 (5)	0.0776 (2)	0.0261 (11)
C14	−0.0684 (5)	0.7775 (5)	0.0876 (2)	0.0246 (11)
C15	0.0109 (6)	0.7183 (5)	0.0438 (2)	0.0288 (12)
C16	0.0120 (6)	0.5676 (5)	0.0398 (2)	0.0262 (12)
C17	−0.1457 (6)	0.5024 (5)	0.0430 (2)	0.0290 (12)
C18	−0.1622 (6)	0.3727 (5)	0.0166 (2)	0.0380 (14)
C19	−0.2802 (6)	0.2964 (6)	0.0425 (2)	0.0395 (14)
C20	0.3853 (7)	0.9742 (7)	0.2489 (2)	0.053 (2)
C21	0.3130 (7)	1.2041 (6)	0.2256 (2)	0.048 (2)
C22	−0.0071 (7)	1.1979 (5)	0.1633 (2)	0.0396 (14)
C23	0.0040 (6)	1.0152 (5)	0.0661 (2)	0.0339 (13)
C24	−0.3200 (6)	0.6620 (5)	0.0664 (2)	0.0275 (12)
C25	−0.2071 (6)	0.4677 (5)	0.0920 (2)	0.0317 (13)
C26	0.2547 (7)	0.5088 (6)	0.0724 (2)	0.0354 (13)
C27	0.3284 (7)	0.4391 (8)	0.1137 (2)	0.071 (2)

Table 2. *Selected geometric parameters (Å)*

O1—C24	1.422 (6)	C6—C7	1.527 (7)
O1—C17	1.438 (6)	C7—C8	1.544 (7)
O2—C25	1.387 (6)	C8—C23	1.541 (6)
O2—C24	1.431 (5)	C8—C14	1.556 (7)
O3—C19	1.369 (7)	C8—C9	1.558 (6)
O3—C25	1.445 (6)	C9—C11	1.541 (7)
O4—C19	1.199 (6)	C9—C10	1.563 (7)
O5—C26	1.362 (6)	C10—C22	1.552 (7)
O5—C16	1.456 (6)	C11—C12	1.524 (7)
O6—C26	1.194 (6)	C12—C13	1.533 (7)
C1—C2	1.529 (7)	C13—C24	1.526 (7)
C1—C10	1.545 (7)	C13—C14	1.545 (6)
C2—C3	1.515 (8)	C14—C15	1.546 (7)
C3—C4	1.534 (8)	C15—C16	1.512 (7)
C4—C21	1.536 (8)	C16—C17	1.530 (7)
C4—C20	1.540 (8)	C17—C18	1.507 (7)
C4—C5	1.551 (7)	C17—C25	1.537 (7)
C5—C6	1.528 (7)	C18—C19	1.481 (8)
C5—C10	1.567 (7)	C26—C27	1.512 (8)

Table 3. *Least-squares planes and deviations from planarity (Å)*

Plane	O1, O2, C17, C25	
Equation	$6.05\,(2)X + 6.76\,(3)Y + 7.21\,(6)Z = 2.70\,(2)$	
Deviations	O1	−0.081 (2)
	O2	0.086 (2)
	C17	0.122 (3)
	C25	−0.127 (3)
	C24	0.316 (7)
Plane	O3, C17, C19, C25	
Equation	$-7.29\,(1)X - 5.00\,(2)Y + 6.83\,(10)Z = 3.26\,(1)$	
Deviations	O3	−0.043 (3)
	C17	0.023 (2)
	C19	0.027
	C25	−0.039
	C18	0.327

During refinement a number of reflections with large negative values of F_o^2 were omitted. These have been flagged with negative standard deviations in the table of observed and calculated structure factors (deposited). The absolute configuration could not be determined [Flack (1983) x parameter = 3.9 (2.7)]. Each H atom was included in a calculated position and allowed to ride on the atom to which it is attached; each H-atom displacement parameter was taken as 20% greater than that of the parent atom.

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

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(2-biphenyl) Telluride

Hongli Chen,[a] Thomas A. Hamor,[a] Karanbir Singh[b] and William R. McWhinnie[b]

[a]*School of Chemistry, University of Birmingham, Birmingham B15 2TT, England, and* [b]*Department of Chemical Engineering & Applied Chemistry, Aston University, Aston Triangle, Birmingham B4 7ET, England*

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Abstract

The title compound, $C_{24}H_{18}Te$, consists of discrete molecules with the Te atom two-coordinate, Te—C 2.105 (5) and 2.125 (5) Å, and angle C—Te—C 96.2 (2)°. The biphenyl residues have their phenyl rings steeply inclined to each other, with dihedral angles of 66.5 (2) and 74.3 (3)°. The inter-ring bond lengths are 1.497 (7) and 1.480 (8) Å.

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