DEPARTAMENTO DE BIOLOGIA ANIMAL
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SUSTANCIAS NATURALES DE MOLUSCOS OPISTOBRANQUIOS: ESTUDIO DE SU ESTRUCTURA, ORIGEN Y FUNCION EN ECOSISTEMAS BENTONICOS.

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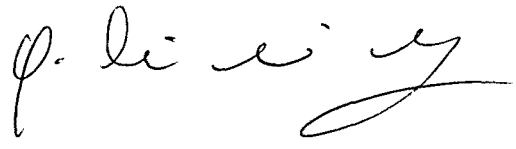
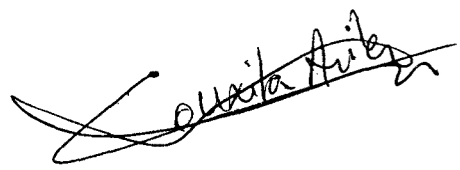


Memoria redactada por Conxita Avila Escartín, para optar al grado de Doctor en Ciencias Biológicas por la Universidad de Barcelona.



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10. ANEXOS

ANEXO 10.I. Datos espectrales de las sustancias de mayor interés.

Longifolina: (3-methyl-2-[(2'E)-3'-methyl-4'-(4"-methyl-2"-furyl)-2'-butenyl] furano; C_{15} H_{18} O_2 EIMS *m/z*: 230 (M^+); ^{1}H-NMR (C_6D_6): 1.60 (3H, s, CH_3-3'); 1.78 (3H, s, CH_3-4"); 1.81 (3H, s, CH_3-3); 3.16 (2H, s, H-4'); 3.22 (2H, d, J =7.0 Hz, H-1'); 5.46 (1H, t, J =7.0 Hz, H-2'); 5.77 (1H, bs, H-3"); 6.00 (1H, d, J = 1.5 Hz, H-4); 6.92 (1H, bs, H-5"); 7.07 (1H, d, J = 1.5 Hz, H-5). ^{13}C-NMR (C_6D_6): 9.7 (CH_3-4" y CH_3-3); 15.8 (CH_3-3'); 25.5(C-1'); 38.6 (C-4'); 109.2 (C-3"); 113.0 (C-4); 113.7 (C-4"); 120.5 (C-3); 122.8 (C-2'); 133.2 (C-3'); 138.0 (C-5"); 140.1 (C-5); 149.8 (C-2); 154.3 (C-2").

El espectro^{1}H-NMR en C_6D_6 se expone en el apartado correspondiente. La numeración coincide con Guella *et al.*, (1985b). Las asignaciones, ya expuestas en el mencionado trabajo, se han confrimado mediante experimentos 2D-NMR: ^{1}H-^{1}H COSY, ^{1}H-^{13}C HETCOR.

Nakafurano-9: $[\alpha]_D$ = - 107.5 (c=0,2 $CHCl_3$); C_{15} H_{18} O_2 EIMS *m/z* (%): 216 (M^+,100), 201 (55), 175 (35). ^{1}H-NMR ($CDCl_3$): 1.08 (3H, s, H-15's); 1.37 (1H, ddd, J = 4.1, 13.3 y 13.3 Hz, H-5a); 1.57 (3H, s, H-13's or H-14's); 1.59 (3H, s, H-14's o H-13's); 1.72 (1H, ddd, J = 2.7, 2.7 y 13.0 Hz, H-12a), 1.79 (1H, ddd, J = 1.8, 4.3 y 13.0 Hz, H-12b); 1.92 (1H, bddd, J = 4.1, 4.1 y 13.3 Hz, H-5b); 1.95 (1H, bd, J = 15.5 Hz H-.9a); 2.25 (1H, ddd, J = 4,1, 13.3 y 16.0 Hz, H-4a); 2.33 (1H, ddd, J = 4.1, 4.1 y 16.0 Hz, H-4b); 2.37 (1H, bdd, J = 5.2 y 15.5, H-9b); 3,17 (1H, m, H-10); 6.06 (1H, d, J =1.7 Hz, H-2); 7.12 (1H, d, J = 1.7 Hz, H-1). ^{13}C-NMR ($CDCl_3$): 13.2 (C-14); 19.8 (C-13); 23.0 (C-4); 30.7 (C-15); 31.8 (C-10); 37.6 (C-6); 38.2 (C-9); 38.6 (C-5); 41.3 (C-12); 113.1 (C-2); 118.3 (C-3); 126.3 (C-7 or C-8); 129.5 (C-8 or C-7); 138.4 (C-1); 156.2 (C-11).

La numeración es acorde con Schulte *et al.*, (1980). Las asignaciones se han comprobado mediante experimentos de desacoplamiento ^{1}H-^{1}H, ^{1}H-^{1}H COSY y ^{1}H-^{13}C HETCOR. La comparación con los datos de NMR previos ha revelado algunas diferencias menores, en particular para las resonancias del ^{13}C-NMR asignadas a los metilos.

iso-Tavacfurano: (3-methyl-2-[(3'E)-3'-methyl-4'-(4"-methyl-2"-furyl)-3'-butenyl] furano $C_{15}H_{18}O_2$ EIMS *m/z*: 230 (M^+); ^{1}H-NMR (C_6D_6): 1.77 (6H, s, CH_3-3 or CH_3-4"); 1.78 (3H, s, CH_3-4" or CH_3-3); 1.88 (3H, s, CH_3-3'); 2.34 (2H, t, J = 7.5 Hz, H-2'); 2.60 (2H, t, J =7.5 Hz, H-1'); 5.95 (1H, bs, H-3"); 5.98 (1H, d, J = 1.5 Hz, H-4); 6.11 (1H, bs, H-4'); 6.91 (1H, bs, H-5"); 7.08 (1H, d, J = 1.5 Hz, H-5).

La numeración y asignaciones son acordes con las de Guella *et al.* (1985b).

Tavacfurano: ópticamente inactivo; UV (Diethyl ether)= 285 nm (ε=7100), 274 nm (ε=10800), 220 nm (ε=7300); $C_{15}H_{18}O_2$; EIMS *m/z*: 230 (M^+); ^{1}H-NMR (C_6D_6): 1.63 (3H, bs); 1.78 (3H, s); 1.83 (3H, s); 2.76 (4H, m); 5.99 (1H, bs); 6.01 (1H, d, J = 1.5 Hz); 6.08 (1H, bs); 6.90 (1H, bs); 7.09 (1H, d, J = 1.5 Hz).

Las asignaciones coinciden con las de Guella *et al.*, (1985).

Dendrolasina: ópticamente inactivo; UV (Diethyl ether) 273 nm (ε=6480); $C_{15}H_{22}O$; EIMS *m/z* (%): 218 (5), 203 (15), 81 (90), 69 (100); ^{1}H-NMR (C_6D_6) δ 1.52 (3H, s), 1.55 (3H, s), 1.67 (3H, s), 2.06 (2H, t, J= 7.3 Hz), 2.14 (2H, t, J= 7.3 Hz), 2.18 (2H, q, J= 7.3 Hz), 5.21 (2H, m), 6.08 (1H, s), 7.07 (1H, s), 7.12 (1H, s); ^{13}C-NMR (C_6D_6) δ: 16.1 (q), 17.8 (q), 25.4 (t), 25.8 (q), 27.1 (t), 28.9 (t), 40.1 (t), 111.3 (d),

124.4 (d), 124.8 (d), 125.2 (s), 131.2 (s), 135.6 (s), 139.2 (d), 142.8 (d).

Agassizina: UV (Diethyl ether) 223 nm (ε=9430), 258 nm (ε=3510); C_{15} H_{18} O; EIMS *m/z* (%): 214 (); ^{1}H-NMR (C_6D_6) δ 0.71 (3H, s); 0.81 (3H, s), 1.63 (2H, m), 2.26 (1H, m), 2.45 (1H, m), 2.59 (1H, m), 3.25 (1H, m), 3.27 (1H, m), 3.46 (1H, bd, J=16 Hz), 5.40 (1H, bd), 5.50 (1H, bd, J= 5 Hz), 5.74 (1H, m), 5.98 (1H, d, J= 1.5 Hz), 7.05 (1H, d, J= 1.5 Hz).

Las asignaciones coinciden con Hochlowski *et al.* (1982).

ent-Furodysinina: $[\alpha]_d$=-43.7° (c= 0.3 $CHCl_3$); UV ($CHCl_3$) 214 nm (ε=8700); C_{15} H_{20} O; EIMS *m/z*: (%): 216 (15), 122 (100), 107 (15); ^{1}H-NMR (C_6D_6) δ 1.12 (3H, s, CH_3-13 or CH_3-14), 1.14 (3H, s, CH_3-14 ó CH_3-13), 1.17 (1H, m, H-10a), 1.44 (1H, ddd, J= 3, 3, y 13 Hz, H-11), 1.54 (1H, m, H-10b), 1.55 (3H, bs, CH_3-15), 1.79 (1H, bdd, J= 6.8 y 17.4 Hz, H-9a), 1.85 (1H, m, H-9b), 2.36 (1H, dd, J= 10.5 y 16.4, H-5a), 2.54 (1H, m, H-6), 2.70 (1H, dd, J= 6.9 y 16.4 Hz, H-5b), 5.46 (1H, bd, J= 6.9 Hz, H-7), 6.13 (1H, bs, H-2), 7.11 (1H, s, H-1). ^{13}C-NMR (C_6D_6) δ 19.6 (C-10), 23.2 (C-15), 26.4 (C-13 ó C-14), 28.0 (C-5), 31.6 (C-6), 31.8 (C-9), 33.0 (C-14 ó C-13), 44.9 (C-11), 108.3 (C-2), 124.9 (C-8), 126.6 (C-7), 133.1 (C-3), 140.9 (C-1), 147.6 (C-4).

Las asignaciones han sido confirmadas mediante experimentos de desacoplamiento ^{1}H-^{1}H, ^{1}H-^{1}H COSY and ^{1}H-^{13}C HETCOR; son acordes con Guella *et al.* (1985).

iso-Nakafurano-9: $[\alpha]_d$= +6° (c= 0.3 $CHCl_3$); U V ($CHCl_3$)= 214 nm (ε=4240); C_{15} H_{20} O; EIMS *m/z*: 216 (M^+, 80), 201 (40), 147 (100), 84 (85); ^{1}H-NMR (C_6D_6) δ 0.72 (3H, s, CH_3-15), 0.92 (3H, d, J=7.2, CH_3-14), 1.37 (1H, m, H-5), 1.44 (1H, dd, J= 5.1 y 14.6 Hz, H-12), 1.47 (1H, bd, J=

14.6 Hz, H-12), 1.83 (1H, ddd, J=3.5, 9.8 y 16.5 Hz, H-5), 1.95 (1H, q, J=7.2 Hz, H-7), 2.30 (2H, m, H-4 y H-9), 2.40 (1H, bd, J= 14.5 Hz, H-9), 2.43 (1H, ddd, J= 3.5, 8.6 y 15.7 Hz, H-4), 3.11 (1H, bm, H-10), 4.59 (1H, bs, J= 2 Hz, H-13a), 4.66 (1H, bs, J= 2 Hz, H-13b), 5.97 (1H, bs, J=1.3, H-2), 7.03 (1H, bs, J=1.3 Hz, H-1).

La numeración y asignaciones son acordes con Tanis y Herrinton (1985). Se han comprobado mediante desacoplamientos ^{1}H-^{1}H COSY y ^{1}H-^{1}H.

iso-Dehidro-dendrolasina: ópticamente inactiva; UV (Diethyl ether) 230 nm (ε=9430), 224 nm (ε=3510); C_{15} H_{20} O; EIMS *m/z*: (%): 216 (5), 201 (20), 93 (100), 81 (90) ^{1}H-NMR (C_6D_6) δ 1.50 (3H, s, CH_3-15), 1.76 (3H, s, CH_3-14), 2.17 (2H, t, J= 7.4 Hz, H-6), 2.31 (2H, t, J= 7.5 Hz, H-5), 2.70 (2H, d, J= 7.0, H-9), 4.89 (1H, bs, H-13a), 4.94 (1H, bs, H-13b), 5.22 (1H, bt, J= 7.0 Hz, H-7), 5.65 (1H, dt, J= 15.5, 7.0 and 7.0 Hz, H-10), 6.07 (1H, bs, H-3), 6.19 (1H, d, J= 15.5 Hz, H-11), 7.05 (1H, s H-4), 7.12 (1H, s, H-1). ^{13}C-NMR (C_6D_6) δ: 16.2 (q, C-15), 18.8 (q, C-14), 25.2 (t, C-5), 28.9 (t, C-6), 43.3 (t, C-9), 111.2 (d, C-3), 115 (t, C-13), 125.2 (s, C-2), 134.6 (s, C-8), 139.2 (d, C-1), 142.2 (s, C-12), 142.8 (d, C-4).

Chelonaplysina-C: [a]25D-55.0° (c 0.24, CHCl3); mp (n-hexane/ethyl ether) 180°-182°; ir ($CHCl_3$) nmax 1755 cm-1; ^{1}H y ^{13}C-NMR ($CDCl_3$) posición carbono (d ^{13}C; d ^{1}H): 1 ó 3 (39.7; 1.18, 1.71), 2 (20.1; 1.52, 1.67), 3 ó 1 (41.4; 1.08, 1.46), 4 (33.3), 5 (58.8; 1.30), 6 (20.5; 1.42, 1.65), 7 (24.9, 1.73), 8 (140.3), 9 (56.0; 2.15), 10 (43.4), 11 (166.7), 12 (32.5; 2.62, 3.14), 13 (38.2; 2.81), 14 (46.9; 3.20), 15 (100.6; 5.88), 16 (101.2; 6.26), 17 (116.3; 5.16, 5.24), 18 (20.6; 0.87), 19 (33.1; 0.86), 20 (13.8; 0.71), $COCH_3$ (169.4), $COCH_3$ (21.1; 2.11); ^{1}H-NMR (C_6D_6) d 6.12

(s, H-16), 5.79 (d J=3.4, H-15), 4.93 (bs, H-17b), 4.74 (d J=2.2, H-17a), 3.08 (m, H-14), 2.68 (dd J=19.2, 6.3, H-12b), 2.03 (m, H-13), 2.00 (d J=19.2, H-12a), 1.96 (bt J=9.5, H-9), 1.51 (3H singlet, CH_3CO), 0.80, 0.78, 0.50 (3H singlets, CH_3-18, CH_3-19 y CH_3-20); ^{13}C-NMR (C_6D_6) d 168.4, 165.5, 141.1, 116.0, 101.3, 100.5, 58.5, 56.1, 47.4, 43.4, 41.5, 39.7, 38.5, 33.2, 32.8, 25.1, 20.7, 20.4, 20.3, 20.0, 13.9; nOed's ($CDCl_3$) irradiado (observado) H-9 (H-15 y H-5), H-13 (H-16, H-17a, H-12b y H-14), H-14 [superpuesto con H-12b] (H-15, H-17a, H-13 y H-12a), H-15 (H-14 y H-9), H-16 (H-12a y H-13), H-17a (H-17b, H-12b y H-13), H-17b (H-17a, H-7 y CH3-20); nOed's (C_6D_6) irradiado (observado H-14 (H-15 y H-13), H-12b (H-12a y H-17a); eims m/z (rel. int.): 376 (2), 361 (4), 316 (15), 301 (10), 283 (7), 192 (20), 137 (75), 123 (100); hreims m/z 376.2269, $C_{22}H_{32}O_5$ son 376.2250.

7-Deacetoxy-olepupuana: δ^1H-NMR ($CDCl_3$), 6.31 (1H, d, J= 2 Hz, H-11); 6.05 (1H, bt, J=2 Hz, H-12); 2.47 (1H, ddd, J= 13.8, 5.2, 1.7, H-7 eq.); 2.28 (1H, bs, H-9); 2.08 (3H, s, CH_3CO); 2.00 (1H, ddddd, J= 13.8, 13.8, 5.7, 1.8, 2 Hz, H-7ax.); 1.71 (1-H, bdd, J= 13.1, 5.7 Hz, H-6eq.); 1.26 (1H, m, H-6ax.); 1.00 (1H, dd, J= 12.5, 2.5, H-5); 0.89 (3H, s); 0.83 (3H, s); 0.81 (3H, s), otras resonancias entre δ 1.70 y δ1.00; δ ^{1}H-NMR (C_6D_6): 6.70 (1H, d, J=2 Hz); 5.91 (1H, bt, J=2 Hz); 2.24 (1H, bs); 2.17 (1H, bdd, J= 4, 13.5 Hz); 1.67 (3H, s, CH_3CO); 0.76 (3H, s); 0.69 (3H, s); 0.68 (3H, s), otras resonancias entre δ 1.80 y δ 0.80; EIMS, m/z (%): 278 ($M^{+\cdot}$,5); 218 (30); 203 (50); 59 (100).

Mediante termolisis (en la placa TLC) la 7-deacetoxy-olepupuana se transforma fácilmente en euryfurano.

ANEXO 10.II. Datos de los ejemplares del apartado 5.17.

ESPECIE y SECCIONES	n	PESO SECO	EXTRACTOS(*)
Gastropteron meckeli	4	321 mg	429 mg (1)
	25	1120 mg	752 mg (1)
			202 mg (2)
borde parápodos	1	5.9 mg	0.9 mg (1)
resto parápodos	1	31 mg	5.2 mg (1)
glándula digestiva	1	27.3 mg	13.6 mg (1)
resto de vísceras	1	14.6 mg	3.0 mg (1)
vísceras	10	140.1 mg	31.4 mg (1)
parte externa	10	236.5 mg	119.4 mg (1)
Philinopsis depicta			
parte externa	1	309 mg	298 mg (1)
digestivo anterior	1	960 mg	223 mg (1)
gland. hermafrodita	1	55 mg	5 mg (1)
glándula digestiva	1	317 mg	82 mg (1)
otras vísceras	1	50 mg	6 mg (1)
puesta	1	290 mg	170 mg (1)
puestas	24	3604 mg	35 mg (2)
secreción mucosa	1	--	423 mg (1)
Haminoea sp			
puestas	100	--	76 mg (2)
Elysia viridis	50	--	200 mg (2)
			65 mg (3)
vísceras	3	1.5 mg	0.2 mg (1)
parte externa	3	8.4 mg	0.6 mg (1)
Elysia timida	3	10.2 mg	17.2 mg (1)
Thuridilla hopei	5	104 mg	40 mg (1)
	27	100 mg	25 mg (2)
	1	2 mg	1 mg (1)
	1	3.5 mg	1 mg (1)
	2	0.5 mg	3.2 mg (1)
puestas	2	7 mg	55 mg (1)
Pleurobranchus membranaceus			
secreción mucosa	1	--	64 mg (1)
Berthella aurantiaca			
vísceras	1	123.2 mg	37.9 mg (1)
parte externa	1	98.0 mg	8.2 mg (1)
puesta	1	1.7 mg	1.3 mg (1)
secreción mucosa	1	--	0.8 mg (2)

Pleurobranchaea meckeli			
glándula digestiva	1	45.5 mg	7.1 mg [1]
resto de vísceras	1	68.1 mg	7.8 mg [1]
parte externa	1	218.2 mg	12.4 mg [1]
glándula digestiva	1	137.7 mg	144.5 mg [1]
resto de vísceras	1	557.4 mg	83.7 mg [1]
parte externa	1	724.7 mg	9.3 mg [1]
Trapania maculata	1	12 mg	1 mg [1]
Limacia clavigera	20	115 mg	14 mg [1]
Polycera quadrilineata	40	--	18 mg [2]
protuberancias	10	5 mg	6 mg [1,#]
resto parte externa	10	31 mg	31 mg [1,#]
vísceras	10	24 mg	61 mg [1,#]
Archidoris tuberculata			
vísceras	3	3.02 g	8 mg [2] 25 mg [3]
parte externa	3	3428 mg	4 mg [2] 7 mg [3]
vísceras	2	136 mg	7 mg [2] 45 mg [3]
parte externa	2	727 mg	6 mg [2] 5 mg [3]
Austrodoris kerguelenensis			
vísceras	1	308 mg	125.8 mg [1]
parte externa	1	628.6 mg	265.7 mg [1] 17 mg [2] 35 mg [3]
Discodoris rosi	22	282 mg	25 mg [1]
Platydoris argo			
borde del manto	1	126 mg	15 mg [1]
borde del pie	1	26 mg	3.5 mg [1]
resto parte externa	1	108 mg	2.5 mg [1]
glándula digestiva	1	20 mg	3.5 mg [1]
puesta	1	53 mg	4 mg [1]
puesta	1	98 mg	19 mg [1]
vísceras	1	45.3 mg	30.8 mg [1]
parte externa	1	477.9 mg	17 mg [1]
vísceras	1	146.7 mg	33.8 mg [1]
parte externa	1	1416 mg	28.8 mg [1]
Dendrodoris languida			
borde del manto	1	35.6 mg	8.0 mg [1]
resto parte externa	1	42.1 mg	5.3 mg [1]
glándula digestiva	1	8.3 mg	2.9 mg [1]
gländ. hermafrodita	1	2.8 mg	1.5 mg [1]
resto de vísceras	1	15.7 mg	4.4 mg [1]
secreción mucosa	1	--	9.9 mg [1]

Dendrodoris subpellucida	1	32.9 mg	2.1 mg (1)
Tritonia nilsodhneri	20	28 mg	31 mg (1)
puestas	13	7.1 mg	12.9 mg (1)
Marionia blainvillea			
vísceras	3	147.4 mg	60 mg (1)
parte externa	3	84.3 mg	30 mg (1)
Tethys fimbria			
parte externa	3	8 g	210 mg (1)

n= número de ejemplares.
(*)= tipo de extracto: 1= acetónico, 2= etéreo, 3= butanólico.
(#)= con elevado contenido en sales debido al Cl_2Mg.