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Dolabriferol: A New Polypropionate from the Skin of the Anaspidean Mollusc *Dolabrifera dolabrifera*

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Abstract: A novel polypropionate, dolabriferol (1), has been isolated from the anaspidean mollusc *Dolabrifera dolabrifera*. The structure has been suggested mainly by spectral methods whereas the relative stereochemistry has been determined by X-ray analysis. Copyright © 1996 Published by Elsevier Science Ltd

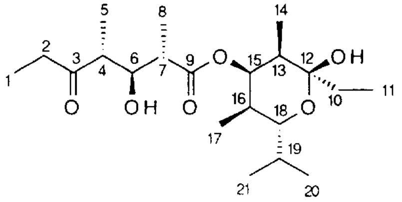
Opisthobranchs are gastropod molluscs scarcely protected by the shell that is either reduced and fragile or internal or completely absent.[2] Very often these molluscs are chemically armed against potential predators.[3-5] Many studies have proved the presence of distasteful molecules in the skin and in the mucous secretion of the animals. The origin of the protective molecules can be either from the diet[6] or by biosynthesis *de novo*.[7]

The subclass Opisthobranchia is split into eight orders: Cephalaspidea, Saccoglossa, Anaspidea, Notaspidea, Gymnosomata, Techosomata, Acochlidiacea and Nudibranchia.[8] Rarely, opisthobranchs exceed ten centimeters in length. In contrast with this general trend, specimens belonging to the order Anaspidea are generally very conspicuous. Because of this, they have been deeply studied in order to explain from a chemical point of view their survival ability.

The order Anaspidea includes five families:[9] Akeridae, Aplysiidae, Dolabriferidae, Dolabellidae, Notarchidae. Almost all chemical studies have been addressed to Aplysiidae specimens, whereas until now no chemical paper has reported studies of Dolabriferidae molluscs. We report the first chemical study of an opisthobranch belonging to Dolabriferidae family, *Dolabrifera dolabrifera* (Rang, 1828).

D. dolabrifera is the most common species of the genus *Dolabrifera*, which differs from the genus *Aplysia* because of the presence of small and asymmetrical parapodia, a flattened body and a calcified internal shell.

Specimens of *D. dolabrifera* were collected off Cuba by SCUBA divers at a depth of 5 meters, in June 1994. The animals were frozen and immediately transferred to ICMIB. A single specimen was carefully dissected in parapodia and hepatopancreas, which were separately extracted with acetone. TLC analysis of both acetone extracts showed that the parapodia contain a main metabolite (R*f* 0.55, light petroleum ether/diethyl ether, 1:1), completely absent in the digestive glands of the mollusc. The remaining frozen animals (11 individuals) were then soaked in acetone by using ultrasound treatment. The diethyl ether soluble fraction (52.1 mg) of the acetone extract was submitted to a preparative chromatographic column (light petroleum ether with increasing amounts of diethyl ether). The main metabolite was isolated in mixture with some fatty acids which were methylated with diazomethane. A further purification (SiO_2 column, light petroleum ether/ diethyl ether, 9:1) afforded pure dolabriferol (**1**, 7.5 mg).



1

The elemental composition ($C_{21}H_{38}O_6$) of **1** was suggested by HREIMS on the molecular peak at *m/z* 386.2684 (M^+; required 386.2668). The ^1H-NMR spectrum showed eight methyls (6 doublets and 2 triplets) immediately suggesting a polypropionate skeleton. The well resolved proton spectrum also displayed signals for the protons of 8 methines and 2 methylenes. The remaining three carbons, detected in the ^{13}C-NMR spectrum at ppm 215.13 (C-3), 173.70 (C-9) and 98.50 (C-12), were assigned to a saturated carbonyl, to an ester carbonyl and to an hemiketal carbon, respectively. ^1H-^1H COSY and ^1H-^1H spin decoupling experiments easily led to the recognition of signals attributable to two isolated ethyl groups (δ 2.46-2.57, H_2-2; δ 1.04, H_3-1 and δ 1.60, H_2-10; δ 0.91, H_3-11), an isolated five carbon system with a central carbinol proton (δ 3.75, H-6) coupled with two methines (δ 2.78, H-4 and δ 2.73, H-7) both bearing a methyl (δ 1.14, H_3-5 and δ 1.32, H_3-8, respectively). The remaining proton sequence was deduced starting from the signal at δ 5.24 (H-15) which, after decoupling, simplified both signals at δ 1.90 (H-13) and at δ 1.78 (H-16) to a sharp quartet and to a double quartet, respectively. H-13 was further linked to H_3-14 (δ 0.99) whereas H-16 exhibited additional coupling with both H_3-17 (δ 0.78) and a proton (H-18) at δ 3.60 further linked to an isopropyl group (δ 1.83, H-19; δ 1.00, H_3-20 and δ 0.83, H_3-21).

The ^1H-^{13}C 2D experiments connected all NMR resonances (Table 1) leading to the structure **1**. In particular, some diagnostic ^1H-^{13}C long-range couplings in the HMBC experiment (J= 10 Hz) placed C-3 between C-2 and C-4, whereas C-9 was directly linked to C-7 and, through the oxygen, to C-15. The relative configuration of C-15, C-16 and C-18 was suggested both by proton coupling constants and by a series of n.O.e. experiments. The large value of the coupling constant between H-16 and H-18 ($J_{H16\text{-}H18}$=10.5 Hz) supported their axial-axial stereochemistry and also the equatorial orientation of H-15 ($J_{H15\text{-}H16}$=2.7 Hz). In addition, steric interactions were observed between the proton at δ 5.24 (H-15) and those resonating at δ 1.90 (H-13), δ 0.99 (H_3-14) , δ 1.78 (H-16) and δ 0.78 (H_3-17), confirming the relative stereochemisty of H-15.

In order to confirm the proposed structure and to establish unambiguously the stereochemistry at the chiral centres, a single crystal X-ray analysis of dolabriferol (**1**) was performed.

Crystal samples of **1** were obtained by slow evaporation of a *n*-hexane solution at room temperature.

Table 1 - ^{1}H- and ^{13}C-NMR data[a,b] for dolabriferol (**1**)

Position	$\delta\ ^{1}H$	m	(Hz)	$\delta\ ^{13}C$	m	C related to H's (HMBC)
1	1.04	t	(7.2)	7.43	q	C-3
2	2.46	dq	(14.5, 7.2)	35.95	t	C-3
	2.57	dq	(14.5, 7.2)			
3	-			215.13	s	
4	2.78	dq	(7.1, 7.1)	49.43	d	C-3, C-6
5	1.14	d	(7.1)	14.35	q	C-3, C-4, C-6
6	3.75	m		75.68	d	
7	2.73	dq	(4.3, 7.1)	43.66	d	C-9
8	1.32	d	(7.1)	15.56	q	C-6, C-7, C-9
9	-			173.70	s	
10	1.60	m		32.45	t	C-12
11	0.91	t	(7.4)	7.23	q	C-12
12	-			98.50	s	
13	1.90	dq	(2.5, 7.2)	39.41	d	C-12
14	0.99	d	(7.2)	20.24	q	C-12, C-13
15	5.24	dd	(2.7, 2.5)	76.85	d	C-9; C-12, C-13, C-18
16	1.78	ddq	(2.7, 7.0, 10.5)	36.45	d	C-18
17	0.78	d	(7.0)	12.91	q	C-15, C-18
18	3.60	dd	(2.0, 10.5)	72.27	d	
19	1.83	dqq	(2.0, 6.8, 6.8)	27.96	d	
20	1.00	d	(6.8)	12.94	q	C-18
21	0.83	d	(6.8)	13.97	q	C-18
OH-6	3.65					
OH-12	3.45					

a) Bruker AMX 500 MHz; $CDCl_3$; δ values are reported in ppm referred to $C\underline{H}Cl_3$ ($\delta\ ^{1}H$ 7.26) and to $\underline{C}DCl_3$ ($\delta\ ^{13}C$ 77.00)
b) Assignments aided by ^{1}H-^{13}C-HETCOR, DEPT, HMQC, ^{1}H-^{1}H-COSY, ^{1}H-^{1}H spin decoupling experiments.

The structure was solved using SIR92 package[10] and refined by full-matrix least squares method to a discrepancy R factor = 0.053, including a restrained refinement (see experimental section) for the ethyl group present at C12 ring-position. In fact, the structure clearly shows a partial conformational disorder pertinent to the equatorial $C10(H_2)$-$C11(H_3)$ group which refines at two positions with occupancy factor 0.5. The alternative positions engaged by the C11 methyl group are respectively in *trans* and *gauche*$^{-}$ orientations with respect to the C13 carbon.

A perspective view of the final crystallographic model of dolabriferol is given in Fig. 1, together with the atomic labelling used in the crystallographic work: the A and B letters refer to the alternative positions of disordered ethyl group. The positional parameters and equivalent isotropic temperature factors for non-hydrogen atoms are reported in Table 2. The present crystallographic study establishes only the relative stereochemisty of **1**, so the shown enantiomer is an arbitrary choice. On this basis the relative configurations at the chiral centres are: C4*R**, C6*S**, C7*S**, C12*S**, C13*R**, C15*R**, C16*R**, C18*R**. Bond lengths, bond angles and some selected torsion angles are reported in Table 3. Intramolecular geometry agrees well with the generally accepted values for similar molecules.[11-13] The pyran ring is close to an ideal chair conformation: O6 and C15 atoms depart 0.616(3) and 0.610(5) Å respectively and on the opposite side from the best plane through the remaining ring atoms. The puckering parameters[14] are $\theta = 4.6(5)^{\circ}$ and Q = 0.537(4) Å. The mean value of the intracycle torsion angles is 54.3(5)°. Besides the carbon substituents in equatorial orientations at C12, C13, C16 and C18 atoms, the pyranoid ring bears an hydroxy group at C12 and a polypropionic chain at C15 carbons, both in axial conformation and isooriented. In the crystal the side-chain is bent around the C7, leading the hydroxyl oxygen O2 properly set to receive an intramolecular hydrogen bond from O4-H hydroxyl group.

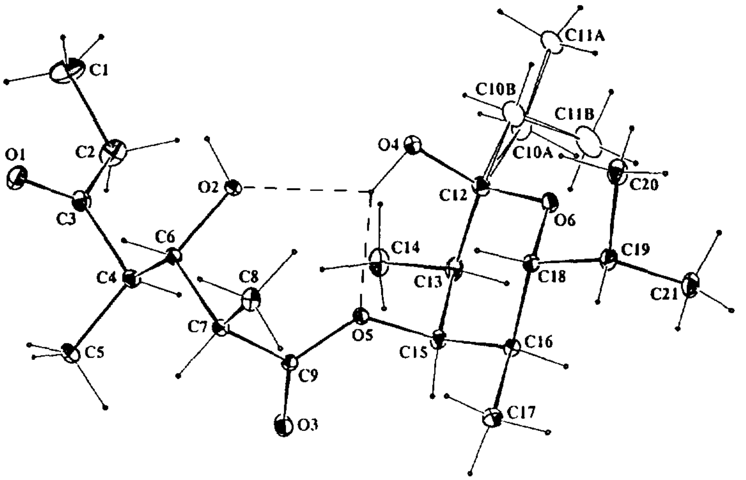


Figure 1. A perspective view of dolabriferol crystal-model. The two alternative positions (open bonds) engaged by the ethyl group are labelled A and B, respectively. Labels of hydrogens were omitted for clarity. Displacement ellipsoids are plotted at the 20% probability level. The dashed lines indicate the intramolecular hydrogen bonds.

Moreover, the intramolecular distance O4·······O5 = 2.854(5) Å is consistent with hydrogen-type interactions, so that O4 acts as donor in two intramolecular H-bonds [O4·······O2 = 2.912(4) Å, H(O4)·······O2 = 2.18 Å, O4-H·······O2 = 130° and O4·······O5 = 2.854(5) Å, H(O4)·······O5 = 2.02 Å, O4-H·······O5 = 141°]. This atom-arrangement can be considered a three-centre hydrogen bond system[15] with the O4-hydrogen, plainly located on a difference Fourier map, that lies near the plane through O2, O4 and O6 atoms. The donor-acceptor and proton-acceptor distances are shorter than the sum of their van der Waals radii, as well as the angles involving H(O4) are within the generally accepted values for this type of interactions.[16]

Table 2 - **Dolabriferol:** Fractional coordinates and equivalent isotropic thermal parameters (Å^2) with e.s.d.'s in parentheses for non-H atoms: $Beq = \frac{1}{3} \Sigma_i \Sigma_j \beta_{ij} a_i^* \mathbf{a}_i a_j^* \mathbf{a}_j$

atom	*x*	*y*	*z*	*B*eq	atom	*x*	*y*	*z*	*B*eq
O1	0.3343(1)	0.0000*	0.6007(3)	9.4(1)	C10A°	0.4806(3)	0.297(3)	0.1757(9)	10.5(4)
O2	0.3457(1)	0.1387(5)	0.3613(2)	5.90(7)	C10B°	0.4774(4)	0.258(3)	0.1785(8)	10.0(4)
O3	0.3226(2)	0.7341(6)	0.3630(3)	11.3(1)	C11A°	0.4861(3)	0.106(2)	0.1152(8)	10.4(3)
O4	0.4021(1)	0.1900(6)	0.2055(2)	6.73(8)	C11B°	0.5124(4)	0.411(4)	0.149(1)	14.5(6)
O5	0.3397(1)	0.5172(5)	0.2490(2)	5.47(6)	C12	0.4272(2)	0.360(1)	0.1755(3)	6.5(1)
O6	0.4080(1)	0.4078(6)	0.0729(2)	6.30(8)	C13	0.4257(2)	0.554(1)	0.2386(3)	6.9(1)
C1	0.4325(3)	-0.120(2)	0.6363(7)	15.0(3)	C14	0.4429(2)	0.525(1)	0.3517(4)	9.2(2)
C2	0.4154(2)	0.089(1)	0.5910(5)	11.0(2)	C15	0.3744(2)	0.6601(8)	0.2145(3)	6.4(1)
C3	0.3617(2)	0.1249(9)	0.5724(3)	6.8(1)	C16	0.3554(2)	0.6887(8)	0.1026(3)	6.4(1)
C4	0.3406(2)	0.3160(8)	0.5127(3)	5.6(1)	C17	0.3037(2)	0.786(1)	0.0800(4)	8.9(2)
C5	0.3084(2)	0.438(1)	0.5717(3)	7.6(1)	C18	0.3586(2)	0.4859(8)	0.0489(3)	5.5(1)
C6	0.3116(1)	0.2399(7)	0.4121(3)	5.03(9)	C19	0.3451(2)	0.494(1)	-0.0655(3)	7.0(1)
C7	0.2828(2)	0.4068(8)	0.3436(3)	5.5(1)	C20	0.3460(2)	0.280(1)	-0.1095(4)	9.8(2)
C8	0.2519(2)	0.306(1)	0.2503(3)	7.6(1)	C21	0.3771(2)	0.642(1)	-0.1086(4)	9.5(2)
C9	0.3162(2)	0.5725(8)	0.3208(3)	6.0(1)					

*fixed to define the origin
°occupancy factor 0.5

Table 3 - **Dolabriferol**: Molecular geometry with e.s.d.'s in parentheses

bond lengths (Å)							
O1-C3	1.220(6)	C1-C2	1.51(1)	C10A-C11A	1.50(2)	C16-C17	1.531(7)
O2-C6	1.437(5)	C2-C3	1.473(7)	C10A-C12	1.53(1)	C16-C18	1.503(7)
O3-C9	1.180(6)	C3-C4	1.518(7)	C10B-C11B	1.50(3)	C18-C19	1.531(6)
O4-C12	1.398(7)	C4-C5	1.535(7)	C10B-C12	1.53(1)	C19-C20	1.499(9)
O5-C9	1.334(6)	C4-C6	1.521(5)	C12-C13	1.519(8)	C19-C21	1.497(9)
O5-C15	1.474(6)	C6-C7	1.530(6)	C13-C14	1.531(7)		
O6-C12	1.426(5)	C7-C8	1.524(6)	C13-C15	1.546(7)		
O6-C18	1.431(5)	C7-C9	1.483(7)	C15-C16	1.522(6)		
bond angles (°)							
C9-O5-C15	121.4(3)	C4-C6-C7	116.1(4)	O4-C12-C10B	99.5(7)	O5-C15-C16	106.5(3)
C12-O6-C18	116.2(3)	C6-C7-C8	110.3(4)	O4-C12-C13	113.3(4)	C13-C15-C16	112.2(4)
C1-C2-C3	115.4(6)	C6-C7-C9	111.1(3)	O6-C12-C10A	102.6(5)	C15-C16-C17	111.4(4)
O1-C3-C2	121.0(5)	C8-C7-C9	113.1(4)	O6-C12-C10B	105.5(5)	C15-C16-C18	110.1(4)
O1-C3-C4	120.2(4)	O3-C9-O5	123.2(5)	O6-C12-C13	109.6(5)	C17-C16-C18	113.6(4)
C2-C3-C4	118.8(5)	O3-C9-C7	124.0(5)	C10A-C12-C13	110.7(7)	O6-C18-C16	109.9(3)
C3-C4-C5	109.3(4)	O5-C9-C7	112.8(4)	C10B-C12-C13	117.9(6)	O6-C18-C19	105.2(4)
C3-C4-C6	107.5(4)	C11A-C10A-C12	114.9(8)	C12-C13-C14	115.9(6)	C16-C18-C19	116.3(4)
C5-C4-C6	112.7(3)	C11B-C10B-C12	110.(1)	C12-C13-C15	111.7(3)	C18-C19-C20	111.0(5)
O2-C6-C4	107.9(3)	O4-C12-O6	110.5(3)	C14-C13-C15	110.2(5)	C18-C19-C21	112.7(4)
O2-C6-C7	109.2(3)	O4-C12-C10A	109.6(7)	O5-C15-C13	106.9(4)	C20-C19-C21	111.1(5)
selected torsion angles (°)							
C1-C2-C3-C4	-170.8(6)	C6-C7-C9-O5	80.2(4)	C13-C12-C10B-C11B	-57.(1)		
C2-C3-C4-C6	109.8(5)	C7-C9-O5-C15	179.6(3)	C16-C18-C19-C20	-175.5(4)		
C3-C4-C6-C7	174.2(3)	C9-O5-C15-C13	117.4(4)	C16 -C18-C19-C21	59.1(6)		
C4-C6-C7-C9	59.1(5)	C13-C12-C10A-C11A	180.0(9)				

The crystal packing is stabilized by an intermolecular hydrogen bond involving O2-H hydroxyl groups and O3 carbonyl oxygens of molecules translated along the *b* direction. Geometrical parameters for this H-bonding are: $O2\cdots O3_{(x,y-1,z)}$ = 2.669(5) Å, $H(O2)\cdots O3_{(x,y-1,z)}$ = 1.69 Å, $O2\text{-}H\cdots O3_{(x,y-1,z)}$ = 177°. In Fig. 2 is shown the molecular arrangement of dolabriferol projected onto *ac* plane.

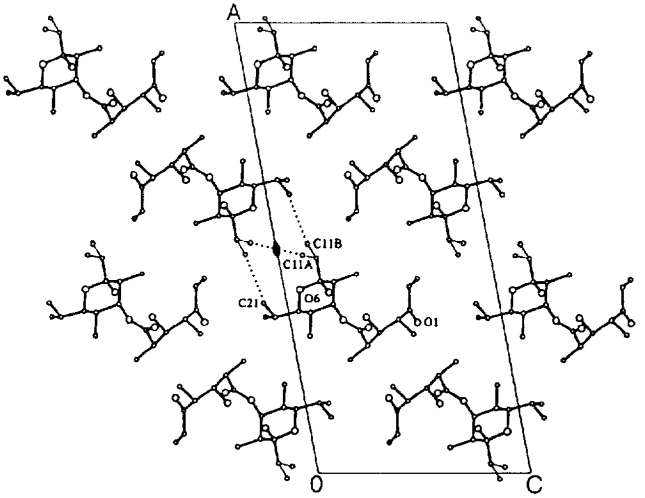


Figure 2. Crystal packing of compound **1** projected onto *ac* plane, the H-atoms have been omitted for clarity. Dotted lines indicate the short contacts involving the disordered ethyl group around the two-fold axis pointed out at **a** = 0.5, **c** = 0.

An attempt to rationalize the conformational disorder of the ethyl group at C12 can be made by considering the short interactions [C11A·······C11A$_{(1-x,y,-z)}$ = 3.40(1) and C11B·······C21$_{(1-x,y,-z)}$ = 3.54(2) Å] between molecules referred by the binary axis at **a** = 0.5, **c** = 0, pointed out in Fig. 2. On the basis of the short contacts and the equal value of multiplicity factors (0.5) of both the alternative positions, it could be postulated that the two-fold related molecules are forced to choose, for the ethyl substituent, alternatively A (energetically more favoured) or B conformations, locally losing the binary symmetry. In this model, the very short contact C11A······C11A is removed and the other not so critical C11B······C21 happen only half times. Anyhow, the choice between the two positions is random in the crystal, so that the symmetry of *C2* crystallographic space group, that was carefully checked, is maintained.

Every attempt to obtain suitable derivatives of **1** with the aim at establishing the absolute stereochemistry at C-6 failed. In fact, treatment with MTPA chloride, in different conditions, afforded a mixture of dehydroderivatives of **1** in the place of the expected Mosher esters. Methanolysis or reduction with $LiAlH_4$ were also conducted in order to obtain the pyranyl diol and determine the absolute configuration at C-15 by applying Mosher method. Unfortunately, both reactions gave complex mixture of products.

Apparently, it is very surprising the finding of an ester obtained by coupling a polypropionate acid with a polypropionate alcohol. However, a tentative rationalization can be suggested (Fig. 3). Most likely, **1** could derive from the polypropionate precursor **2** by opening the tetrahydropyrone ring between C-9 and C-10. Then, the formation of a cyclic hemiketal, by linking C-12 to the hydroxy group at C-18, gives **1**. The numbering of **1** is proposed in agreement with this biogenetic hypothesis.

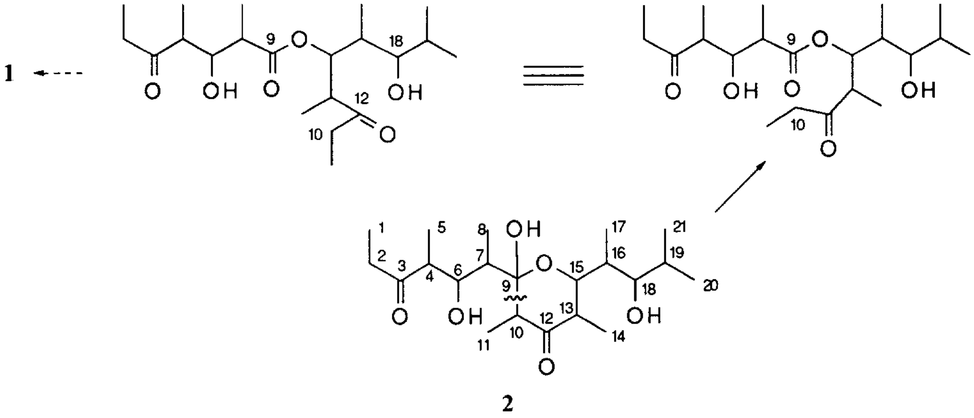


Figure 3. Hypothetical biosynthesis of dolabriferol (1).

EXPERIMENTAL SECTION

General Procedures. The mass spectra were obtained from AEI MS-30 (EIMS) and Kratos MS-50 (HREIMS). IR spectrum was recorded in liquid film on a Bio-Rad FTS instrument. Optical rotation was measured on a Jasco DIP 370 polarimeter. ^1H and ^{13}C-NMR spectra were performed on an AMX 500 Bruker spectrometer. 2D experiments were obtained using standard micro-programs of Bruker software. TLC chromatography was carried out on Merck precoated Si gel (Kieselgel 60 F_{254}) plates. Commercial Merck Si gel 60 (70-230 mesh ASTM) was used for column chromatography eluting with Merck solvents.

Biological material. *D. dolabrifera* was collected by SCUBA divers off Cuba, at a depth of 5 meters, during June 1994. A voucher specimen is deposited at "Departamento de Biologia de Organismos y Sistemas", University of Oviedo, Spain.

Extraction and isolation. A single specimen of *D. dolabrifera* was dissected in parapodia and digestive gland, which were separately extracted with acetone. The ethereal soluble fractions of both acetone extracts were analyzed by TLC chromatography. The remaining 11 individuals (average length 3 cm) were extracted with acetone (100 ml x 3 times), by using ultrasounds. After removal of the organic solvent, the residual water was extracted with Et_2O (50 ml x 3 times). The ethereal phase was concentrated under reduced pressure to give 52.1 mg of crude material, which was chromatographed on a Si-gel column using light petroleum ether with increasing amounts of Et_2O, as eluent. The fractions containing the main metabolite at R_f 0.55 (light petroleum ether/Et_2O, 1:1) were combined, methylated with diazomethane and further chromatographed on a Si-gel column (light petroleum ether/Et_2O, 9:1), giving 7.5 mg of pure dolabriferol (**1**).

Dolabriferol (1). $[\alpha]_D^{25}$= -29.4° ($CHCl_3$, c=0.7); m.p. 112-114°C (*n*-hexane); IR (liquid film)ν_{max} 3444, 2966, 2937, 2879, 1733, 1718, 1126, 1029 cm^{-1}; EIMS at *m/z* 386 (3%), 368 (3%), 325 (3%), 283 (3%), 254 (8%), 199 (6%), 181 (25%), 153 (90%), 124 (100%); HREIMS *m/z* 386.2684 ($C_{21}H_{38}O_6$ requires 386.2668); ^1H- and ^{13}C-NMR data in Table 1.

Crystal data and structure refinement of dolabriferol (1). Single crystals of **1** were obtained by slow concentration of a *n*-hexane solution and a sample of size 0.3x0.2x0.1 mm was selected for the crystallographic study. Accurate cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections at medium θ (23°< θ < 27°), using graphite monochromatized CuKα radiation and Enraf-Nonius CAD-4F diffractometer on line with a MicroVAX Digital computer.

Crystal data: $C_{21}H_{38}O_6$, Mw = 386.53, monoclinic system, space group *C*2, with $Z = 4$, $a = 27.695(9)$, $b = 6.399(1)$, $c = 13.679(5)$ Å, $\beta = 102.07(2)°$, $V = 2371(1)$ Å^3, $D_c = 1.083$ g·cm^{-3}.

2651 independent reflections (θ_{max} = 75°) were collected at room temperature, using ω-2θ scan mode, as suggested by peak-shape analysis. During the data collection the intensities of four standard reflections were monitored every 5 h (4% variation) in order to check the crystal and equipment stability. No significant intensity decay was observed. The intensities were corrected for Lorentz and polarization factors, but not for the absorption effect ($\mu = 5.99$ cm^{-1}).

The structure was solved by direct methods using SIR92 package.[10] The positional and anisotropic temperature parameters for non-hydrogen atoms were refined by full-matrix (on F) least-squares method: the *y* coordinate of O1 oxygen was fixed to define the origin. The structure clearly revealed a conformational disorder concerning the ethyl substituent at C12 ring-position, with two equiprobable sites for the C11 methyl group related by a rotation of about 120°. A restrained refinement[17] was performed for positional and thermal parameters of the two ethyl groups (C10A, C11A and C10B, C11B, respectively): the occupancies were initially refined and later fixed at 0.5. All hydrogens were positioned (distances C-H = 1.02 and O-H = 0.98 Å) on the basis of geometrical considerations, taking into account for hydroxyl and methyl groups the indications of difference Fourier map. H-atoms were included in the last refinement as fixed atoms with isotropic thermal parameters set equal to B_{eq} of the parent atom. At convergence, the final discrepancy index $R = \Sigma ||Fo| - |Fc|| / \Sigma |Fo|$ was 0.053 for the 1669 observed reflection with $I \geq 2.5\ \sigma(I)$; $R_w = 0.058$ with $w^{-1} = [\sigma^2(Fo)+(0.02Fo)^2+0.2]$, $S = 0.912$, $(\Delta/\sigma)_{max} < 0.01$; 24 restrains and 262 refined parameters including the extinction coefficient[18] = $1.0(2)\cdot10^{-6}$. No residual election density is outside the range -0.14÷0.12 e·Å^{-3}.

Atomic scattering factors were taken from Cromer and Waber.[19] All calculations were performed using Enraf-Nonius *SDP* software[20] on a MicroVAX 3100 computer.

Structure factors, anisotropic thermal parameters, hydrogen atom parameters, and complete molecular geometry have been deposited.

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