variation of the water concentration during the reaction is expressed by the bar in figures. Further workup of the reaction products was carried out according to the method of Kornblum et al.[8]

Determination of Water Concentration. The water content of THF was determined by a coulometric Karl–Fischer apparatus (Kyoto Electronic Co. Ltd., Type MK-AII). Its sensitivity was better than 20 μL of water for 1-mL solutions (i.e., ca. 1 mM). When the same THF solution was measured in triplicate, the relative error was less than 8%.

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Registry No. 1, 3019-88-3; 2, 100-39-0; 2-benzyloxynaphthalene, 613-62-7; 1-benzylnaphth-2-ol, 36441-31-3.

New Clerodane Diterpenoid from *Teucrium eriocephalum*

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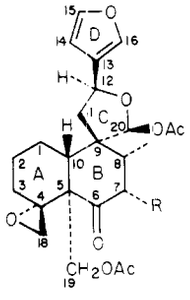
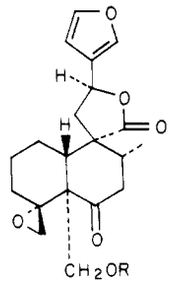
Continuing our studies on diterpenic compounds from the *Teucrium* species (family Labiatae),[2,3] we have now investigated *T. eriocephalum* Wk.,[4] a species which grows only in limited areas of northeast Spain. From aerial parts of this plant a new diterpenoid, eriocephalin (1), has been isolated.

Eriocephalin (1) is the 7α-hydroxy derivative of gnaphalidin (2), a diterpene recently isolated[3] from *Teucrium gnaphalodes* L'Her. together with related products.

The present paper deals with the structure determination of eriocephalin and with a comparative study of ^{13}C NMR spectra of 1, 2, gnaphalin (4), and 19-acetylgnaphalin (5).



1, R = OH
2, R = H
3, R = OAc



4, R = H
5, R = Ac

Eriocephalin had a $C_{24}H_{30}O_9$ molecular formula, and its IR spectrum showed characteristic absorptions for a furan ring (3145, 3130, 1505, 880 cm^{-1}), a ketone (1715 cm^{-1}), one or more ester groups (1732 cm^{-1}, br), and a hydroxyl group [3500 (KBr), 3610 (CCl_4) cm^{-1}]. The presence of this last function was confirmed because Ac_2O–pyridine treatment of compound 1 yielded a monoacetyl derivative (3, $C_{26}H_{32}O_{10}$) of the natural product.

Table I. ^{13}C Chemical Shifts of Compounds 1, 2, 4, and 5[c]

	compd			
carbon no.	1	2	4	5
1	22.6 t	23.3 t	23.5 t	23.5 t
2	25.7 t	24.9 t	24.8 t	24.8 t
3	30.4 t	33.0 t	32.9 t	32.8 t
4	60.8 s	61.1 s	61.9 s	61.2 s
5	53.6 s[a]	54.3 s	55.5 s	54.2 s
6	206.7 s	205.6 s	207.6 s	206.5 s
7	74.6 d[b]	45.0 t[a]	44.1 t[a]	43.5 t[a]
8	43.0 d	41.7 d	40.8 d	41.6 d
9	53.2 s[a]	51.6 s	51.6 s	51.6 s
10	51.2 d	55.1 d	54.5 d	55.6 d
11	49.6 t	45.7 t[a]	43.7 t[a]	43.7 t[a]
12	73.2 d[b]	71.1 d	71.9 d	71.9 d
13	127.6 s	125.7 s	124.9 s	124.8 s
14	108.5 d	108.4 d	107.8 d	107.8 d
15	139.3 d	139.4 d	139.3 d	139.4 d
16	143.4 d	143.4 d	144.2 d	144.2 d
17	10.6 q	18.0 q	17.1 q	17.1 q
18	51.9 t	49.2 t	50.0 t	48.5 t
19	62.5 t	61.8 t	62.4 t	62.1 t
20	98.8 d	97.8 d	176.5 s	176.8 s
OCOMe	170.1 s	170.4 s		170.5 s
	169.3 s	169.3 s		
OCOMe	21.4 q	21.3 q		20.8 q
	20.9 q	20.7 q		

[a,b] These assignments may be reversed, but those given here are considered to be the most likely. [c] In ppm relative to Me_4Si.

The ^1H NMR spectrum of eriocephalin (1) showed signals for a secondary methyl group at δ 0.89 (d, J = 7 Hz), a β-substituted furan ring (two α-furan protons at δ 7.36 and one β-furan proton at δ 6.37), and two acetates (δ 2.08 and 1.97), one of which was placed on a methylene group attached to a fully substituted carbon atom (a two-proton singlet at δ 4.69) and the other one on an hemiacetalic carbon atom without vicinal protons (one-proton singlet at δ 6.33). The closure of this hemiacetal group was revealed by a one-proton triplet at δ 5.20 (J = 8 Hz) which must be allylic to the furan ring and vicinal to a methylene group. All these signals are also encountered in the ^1H NMR spectrum of gnaphalidin (2). In addition, the ^1H NMR spectrum of 1 showed a doublet signal (1 H, J = 6 Hz) at δ 4.77 assigned to the geminal proton of a secondary hydroxyl group, because it was shifted at lower field (δ 5.60) in the ^1H NMR spectrum of the acetyl derivative. On the other hand, the ^1H NMR spectrum of compound 3 showed a typical pattern (which is not clear in the spectrum of 1) for an α,α-disubstituted oxirane ring [δ 2.92 (1 H, dd, J_1 = 4 Hz, J_2 = 2 Hz) and 2.40 (1 H, d, J = 4 Hz)] identical with those found in gnaphalidin (2) and related diterpenoids.[3]

All the above data were in agreement with structure 1 for eriocephalin, in which the secondary hydroxyl group was placed on C-7 by double-resonance experiments. When the signal of the secondary methyl group, or also the doublet at δ 4.77, was irradiated, a clear modification was observed at δ 2.44 (H-8), and the signals at δ 0.89 (secondary methyl group) and 4.77 (geminal proton of the alcohol) collapsed to singlets on irradiation at H-8. The same effects were observed between the signals at δ 1.03 (3 H-17), 2.50 (H-8), and 5.60 (H-7) in the ^1H NMR

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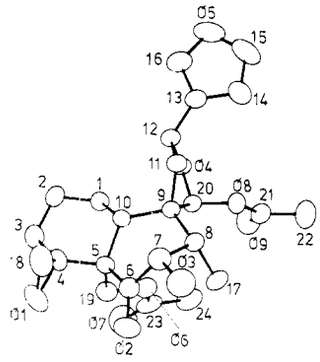

Figure 1. ORTEP[6] drawing of eriocephalin (1). For the sake of clarity no H atoms are represented. Thermal ellipsoids are scaled to 50% probability.

spectrum of compound 3. Thus, the hydroxyl group must be placed on C-7, and considering that the secondary methyl group is equatorial as in gnaphalidin,[3] this hydroxyl group may be axial on the basis of the coupling constant (J = 6 Hz) between H-7 and the axial H-8.

Comparison between the ^{13}C NMR spectra of gnaphalidin (2) and eriocephalin (1) also indicated the presence of an additional OH group on C-7 of 1 (see Table I). In particular the diamagnetic shift ($\Delta\delta$ = −7.4 ppm) experienced by C-17 of 1 with respect to gnaphalidin (2) clearly supports this conclusion.

Eriocephalin (1) showed a positive Cotton effect ($\Delta\epsilon_{294}$ = +2.53) whereas gnaphalidin (2) possessed a negative value ($\Delta\epsilon_{305}$ = −0.72), but as the absolute configuration of clerodin-like diterpenoids has been recently reversed,[5] a single-crystal X-ray determination was undertaken in order to establish the structure and absolute configuration of eriocephalin (1). Figure 1 shows the absolute configuration of the final X-ray model. The best least-squares ring planes in the molecule have than calculated. Ring A presents an approximate chair conformation having C-3 and C-10 at 0.570 and −0.808 Å, respectively, out of the plane defined by C-1, C-2, C-4, and C-5; ring B shows a distorted boat conformation, with C-7 and C-10 at 0.794 and 0.397 Å, respectively, out of the plane defined by C-5, C-6, C-8 and C-9; ring C has an envelope conformation, C-9 being at the flap and 0.634 Å out of the plane; ring D is nearly planar and makes an angle of 88.5° with the plane of C ring. Both acetyl groups show the usual geometry, the carbonyl oxygen atoms being cis with respect to C-19 and C-20.

The H atom at O-3 is 2.17 Å away from the O-2 atom (the sum of the van der Waals radii being 2.72 Å), forming an intramolecular hydrogen bond O-3–H-3···O-2 with O-3···O-2 = 2.684 Å. All bond distances and angles are of the usual magnitudes. There are no intermolecular distances among nonhydrogen atoms less than 3.0 Å. (For most details on the X-ray structure determination see the Experimental Section.)

Therefore, in accordance with the terminology suggested by Rogers et al.,[5] eriocephalin (1) has the absolute configuration of neoclerodane.

Experimental Section

The melting point of 1 was determined in a Kofler apparatus and is uncorrected. The optical rotation was measured with a Perkin-Elmer 141 polarimeter with a 1-dm cell. Elemental analysis was carried out in Madrid[1c] with the help of an automatic analyzer. IR spectra were determined on a Perkin-Elmer 257 spectrometer. ^1H and ^{13}C NMR spectra were measured at 100 and 25.2 MHz, respectively, in $CDCl_3$ solution with Me_4Si as internal standard. Assignments of ^{13}C chemical shifts were made with the aid of off-resonance and noise-decoupled ^{13}C NMR spectra. Mass spectra were obtained on a JEOL MS-01SG-2 instrument.

Isolation of Eriocephalin (1). Dried and finely powdered *T. eriocephalum* plants (1.5 kg), collected near Adra (Almería, Spain), were extracted with acetone (10 L) at room temperature for 1 week. The solvent was evaporated, the residue extracted with ethyl acetate, and the extract washed with water and dried. Evaporation of the solvent yielded a gum (30 g) which was subjected to dry-column chromatography over silica gel (400 g, Merck No. 7734, deactivated with 15% water). Elution with petroleum ether and petroleum ether–EtOAc (9:1 and 4:1) gave plant waxes which were rejected; elution with petroleum ether–EtOAc (7:3) gave eriocephalin (1, 50 mg after crystallization from petroleum ether–EtOAc): mp 197–200 °C; $[\alpha]^{20}_D$ +76.1° (*c* 0.26, $CHCl_3$); IR (KBr) 3500, 3145, 3130, 3045, 3010, 2990, 2950, 2930, 2880, 1732, 1715, 1505, 1493, 1455, 1430, 1365, 1235, 1165, 1125, 1097, 1080, 1065, 1020, 1010, 1000, 978, 965, 935, 910, 880, 860, 840, 808, 775, 660, 640 cm^{-1}; CD (EtOH, *c* 0.54) $\Delta\epsilon_{294}$ = +2.53; for ^1H and ^{13}C NMR see text and Table I, respectively; MS (70 eV, direct inlet) *m/e* (rel intensity) 462 (M^+, 1.4), 444 (M^+ − 18, 4), 402 (M^+ − 60, 19), 384 (M^+ − 60 − 18, 4), 374 (18), 308 (23), 283 (13), 248 (21), 231 (18), 220 (24), 203 (24), 189 (23), 163 (67), 135 (30), 105 (33), 94 (100, base peak), 91 (43), 81 (73), 79 (37), 69 (24), 67 (24), 60 (40), 55 (30). Anal. Calcd for $C_{24}H_{30}O_9$: C, 62.32; H, 6.54. Found: C, 62.12; H, 6.58.

Compound 3. Ac_2O–pyridine treatment of eriocephalin (1, 6 mg) yielded 3 (6 mg) as a syrup: ^1H NMR δ 7.32 (2 H, m, $W_{1/2}$ = 5 Hz, H-15 and H-16), 6.34 (1 H, s, H-20; 1 H, m, $W_{1/2}$ = 4 Hz, H-14), 5.60 (1 H, d, J = 5 Hz, H-7), 5.20 (1 H, t, J = 8 Hz, H-12), 4.64 (2 H, s, 2 H-19), 2.92 (1 H, dd, J_1 = 4 Hz, J_2 = 2 Hz, H-18), 2.40 (1 H, d, J = 4 Hz, H-18), 2.50 (1 H, m, $W_{1/2}$ = 10 Hz, H-8), 2.13, 2.05, and 1.95 (3 H each, s, 3 OAc), 1.03 (3 H, d J = 7 Hz, 3 H-17); MS (70 eV, direct inlet) M^+ at *m/e* 504, mol wt of $C_{26}H_{32}O_{10}$ 504.

X-ray Structure Determination of 1. $C_{24}H_{30}O_9$ crystallizes in the space group $P2_12_12_1$, Z = 4, with a = 16.1108 (4), b = 12.7158 (3), and c = 11.0454 (3) Å. The molecular weight is 462.50 and the calculated density is 1.357 g cm^{-3}. Intensities of 2189 independent Friedel pairs were measured up to θ = 65° on a computer-controlled four-circle diffractometer. Graphite-monochromated Cu Kα radiation (1.5418 Å) and the $\omega/2\theta$ scan technique were used. No crystal decomposition was observed during the data collection process. After the usual correction for Lorentz and polarization effects, 2106 Friedel pairs were considered as observed according to the criterion $I > 2\sigma(I)$ and were used in the calculations.[7] The structure was solved by a multisolution tangent formula approach using the program MULTAN[8] and the 150 normalized structure factors greater than 1.6. The structure was first refined anisotropically by full-matrix least-squares analysis with unit weights by using *hkl* reflections. The hydrogen atoms were located on a difference map. A convenient weighting scheme[9] was selected to prevent bias in $w\Delta^2F$ vs. $|F_o|$ and sin θ/λ. Several cycles of weighted anisotropic refinement (fixed isotropic parameters for H atoms) including both *hkl* and $\overline{hkl}$ reflections gave the following unweighted and weighted discrepancy indices: R = 0.050 and R_w = 0.060. The absolute configuration was confirmed by comparing the 49 more relevant Bijvoet pairs, giving the following discrepancy indices:[10] average Bijvoet difference $R_1 = \sum|[F_o(+h) - F_o(-h)] - [F_c(+h) - F_c(-h)]|/N$ = 0.154 (0.401 for the reversal enantiomorph), average Bijvoet ratio $R_2 = \sum|R_o - R_c|/N$ = 0.019 (0.048), and $R_3 = \sum|\Delta I_o - \Delta I_c|/\sum|\Delta I_o|$ = 0.613

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(1.471 for reversal enantiomorph) with N = number of Bijvoet pairs, $R_o = \Delta I_o/\langle F_o^2\rangle$, $R_c = \Delta I_c/\langle F_c^2\rangle$, $\Delta I_o = F_o^2(+h) - F_o^2(-h)$, and $\Delta I_c = F_c^2(+h) - F_c^2(-h)$.

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Registry No. 1, 71774-90-8; 2, 70544-92-2; 3, 71774-91-9; 4, 70544-90-0; 5, 64756-04-3.

Supplementary Material Available: A list of bond distances, bond angles, deviations of atoms from the ring planes, and torsion angles (3 pages). Ordering information is given on any current masthead page.

Syntheses of 7,12-Dimethylbenz[*a*]anthracene-3,4- and -1,4-diones[1]

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In view of the recent communication[3] in which the synthesis of 7,12-dimethylbenz[*a*]anthracene-3,4-dione (**1**) by oxidation of 3-hydroxy-7,12-dimethylbenz[*a*]anthracene (**2**) with benzeneselenic anhydride was described, we thought it of interest to report some of our results in the same area.

We have oxidized **2**[4] with Fremy's salt[5] to obtain pure **1**, mp 154–155 °C, in 72% yield calculated on the recovery of 22% of **2**. We made no attempt to reduce **1** to 3,4-dihydro-3,4-dihydroxy-7,12-dimethylbenz[*a*]anthracene[3] but did reduce **1** to 3,4-dihydroxy-7,12-dimethylbenz[*a*]anthracene (**3**) in almost quantitative yield by sodium dithionite.

In a similar way 1-hydroxy-7,12-dimethylbenz[*a*]anthracene[4] (**4**) was oxidized to 7,12-dimethylbenz[*a*]anthracene-1,4-dione (**5**) in 94% yield based on recovery of 47% of **4**, and **5** was reduced to 1,4-dihydroxy-7,12-dimethylbenz[*a*]anthracene (**6**). The yields of **1** and **5** were not increased by using larger amounts of Fremy's salt, and unchanged **2** and **4** were always recoverable in about the amounts mentioned above. Interestingly, 2-hydroxy- and 4-hydroxy-7,12-dimethylbenz[*a*]anthracenes[4] were recovered unchanged on treatment with Fremy's salt, probably because oxidation would require attack at the hindered 1-position. A similar failure of enzymic hydroxylation of 7,12-dimethylbenz[*a*]anthracene to yield appreciable amounts of 1,2-dihydro 1,2-diols has been noted, 5,6-dihydro diols, 3,4-dihydro diols, 8,9-dihydro diols, and 10,11-dihydro diols being the products.[6]

The 3-hydroxy-7,12-dimethylbenz[*a*]anthracene (**2**) we used was prepared by a new route, shown in Scheme I, which we regard as preferable to the other syntheses of **2**.[3,4]

The starting materials for our present synthesis of **2** were two readily available chemicals, namely, phthalic anhydride and 2-bromo-6-methoxynaphthalene.[7] Condensation via the Grignard reagent afforded *o*-(6-methoxy-2-naphthoyl)benzoic acid[8] (**7**) in 65% yield. The remaining steps to 3-hydroxy-7,12-dimethylbenz[*a*]anthracene (**2**) went well as indicated in Scheme I.

Experimental Section[9]

***o*-(6-Methoxy-2-naphthoyl)benzoic Acid (7).** In the best of several experiments a hot solution of 11.85 g of 2-bromo-6-methoxynaphthalene in 150 mL of benzene was added to a suspension of 1.21 g of sublimed magnesium in 50 mL of 1:1 ether–benzene containing 0.5 mL of ethylene dibromide[10] during 30 min. After reflux was maintained for 15 h the Grignard reagent was added to a stirred suspension of 8.88 g of phthalic anhydride in 100 mL of 1:1 ether–benzene. After 4 h at reflux the mixture was cooled, treated with dilute HCl, and worked up as usual to yield 11.3 g (72%) of **7**, mp 164–166 °C. Recrystallization from benzene yielded **7**, mp 169–170 °C, with little loss.[8] In a similar run starting with 47.4 g of bromo compound a somewhat lower yield was obtained.

3-Methyl-3-(6-methoxy-2-naphthyl)phthalide (8).[12] To a solution of 6.0 g of **7** in 100 mL of 1:1 ether–THF was added 32 mL of 1.6 M methyllithium (Ventron). After refluxing for 15 h the mixture was worked up as usual to yield 5.5 g (91%) of **8**, mp 159–161 °C.

***o*-[1-(6-Methoxy-2-naphthyl)ethyl]benzoic Acid (9).**[12] A mixture of 4.56 g of **8**, 30 g of zinc dust activated with 0.5 g of $CuSO_4$, 25 mL of pyridine, and 240 mL of 10% KOH was held at reflux for 15 h and worked up to yield 4.4 g (97%) of **9**, mp 207–208 °C.

3-Methoxy-7,12-dimethylbenz[*a*]anthracene (11). Ten milliliters of anhydrous HF was stirred in 0.55 g of **9**. After 15 min the solution was poured on ice and worked up as usual to yield a yellow viscous oil which was dissolved in 10 mL of ether and treated with 3 mL of 1.5 M methyllithium. After 15 h of refluxing, the crude product was chromatographed over neutral alumina using benzene to yield 450 mg (87%) of **11**,[4] mp 131–132 °C.

***o*-[1-(6-Methoxy-2-naphthyl)ethyl]acetophenones (10).**[12] To a solution of 1.4 g of **9** in 30 mL of THF was added 10 mL of 1.5 M CH_3Li. After 5 h at reflux the mixture was treated with dilute HCl and after removal of the THF on a rotary evaporator there was obtained an almost quantitative yield of **10**, mp 106–107.5 °C.

Attempts to cyclize **10** to **11** with PPA (room temperature for 7 h gave 65% of **11**), with HF for 15 min (37% of **11**), or with $CF_3CO_2H + ZnCl_2$ at reflux for 4 h (42% of **11**) did not afford **11** in as high a yield as was obtained by the benz[*a*]anthrone route above described.

7,12-Dimethylbenz[*a*]anthracene-3,4-dione (1).[12] A solution of 3.0 g of Fremy's salt and 1.36 g of **2**[4] in 50 mL of $^1/_6$ M KH_2PO_4, 400 mL of water, and 400 mL of methanol was stirred for 15 h

(1) This work was supported by Grant No. CA07394, awarded by the National Cancer Institute, DHEW.
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(9) All melting points are uncorrected. The term "worked up as usual" means that an ether–benzene solution of the reaction products was washed with acid and/or base to separate acidic and neutral products. The solvents were then removed on a rotary evaporator or by distillation. In general the IR, NMR, and mass spectra were taken and agreed with those expected from the structures.
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(11) This quinone, **1**, was reported[3] to melt at 181–183 °C. However, Dr. Harvey has informed us that a deteriorated sample was involved and that the true melting point of **1** is 155–156 °C as we found.
(12) These new compounds gave analyses within ±0.3% of the theoretical C and H.

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