EXPERIMENTAL

^{1}H NMR spectra were recorded at 80 MHz in $CDCl_3$ with TMS as int. ref. The values are given in δ units. Mass spectra were recorded at 70 eV, with direct inlet. The collection of plant material and its extraction method has already been described [1]. Fraction 3 obtained after CC [1] yielded mainly stearic acid by crystallization. The filtrate after further CC afforded stearic acid and a mixture, which after exhaustive TLC (petrol–EtOAc, 19:1) gave stearic acid (20 mg) and 1 (8 mg, R_f 0.60).

Tanavulgarol (1). Colourless oil; $[\alpha]_D^{26}$ +80° ($CHCl_3$, *c* 0.3); UV $\lambda_{max}^{CHCl_3}$ nm: 243 (α, β-unsaturaed β,β disubstituted ketone); IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3400, 1670, 1370; MS *m/z* (rel. int.): 236 $[M]^+$, ($C_{15}H_{24}O_2$) (1), 218 $[M-H_2O]^+$ (9), 203 $[218-Me]^+$ (8), 135 $[218-C_3H_7CO]^+$ (20), 120 $[135-Me]^+$ (25), 83 $[C_3H_7CO]^+$ (35), 69 (60), 55 (70), 43 (100). (Found: C, 76.29; H, 10.18. $C_{15}H_{24}O_2$ requires C, 76.27; H, 10.17%). ^{1}H NMR ($CDCl_3$): δ4.10 (*ddd*, *J* = 11, 7, 3 Hz, H-2), 2.13 (*m*, H-3), 2.70 (*d*, *J* = 18 Hz, H-8), 2.60 (*d*, *J* = 18 Hz, H-8'), 5.70 (*br s*, H-10), 2.00 and 1.70 (*br s*, H-12 and H-13), 0.90 (*s*, H-14), 0.88 (*d*, *J* = 7 Hz, H-15).

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SALVISYRIACOLIDE, A SESTERTERPENE FROM *SALVIA SYRIACA*

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Key Word Index—*Salvia syriaca*; Labiatae; sesterterpene; salvisyriacolide.

Abstract—The extract of the aerial parts of *Salvia syriaca* afforded a polar sesterterpene lactone with four hydroxy groups. Acetylation gave a triacetate. The structure was elucidated by high field NMR spectroscopy.

Some time ago two sesterterpenes were reported from *Salvia hypoleuca* [1]. We have studied a further species from Iran, *S. syriaca* L. The polar fractions were separated by TLC and HPLC. Finally a colourless oil was obtained. CIMS indicated the presence of a sesterterpene with the molecular formula $C_{25}H_{40}O_6$. This was supported by CIMS of the corresponding triacetate obtained by mild acetylation (*m/z* 563 corresponding to $C_{31}H_{46}O_9 + 1$) and by the ^{13}C NMR spectrum of **1** (Table 1) which showed 25 carbon signals. The ^{1}H NMR spectrum (Table 1), as well as the IR spectrum (1765 cm^{-1}), indicated the presence of a butenolide. This was established by spin decoupling. Saturation of a narrowly split signal at δ5.86 changed the methyl doublet at δ2.07 to a singlet and sharpened the broadened signal at δ4.90. The proton corresponding to the latter signal was further coupled with allylic protons which showed threefold doublets at δ2.80 and 2.29. These signals and the coupling partners nicely agreed with the corresponding signals of salvileucolide methyl ester (**2**) isolated from *S. hypoleuca* [1]. Several further signals also were similar. However, a changed substitution pattern was indicated by the absence of the methoxy signal which was replaced by a broadened two proton singlet at δ3.69 which was shifted down field in the corresponding triacetate (δ4.09 *d* and 3.59 *d*). Accordingly, a hydroxymethyl group at C-4 was very likely. Furthermore a double doublet at δ3.58 and a threefold doublet at 3.91 required two secondary hydroxy groups. Both signals were shifted downfield in the spectrum of the triacetate.

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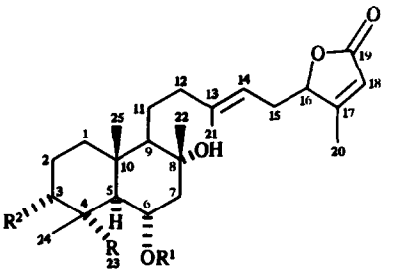
Table 1. ^{1}H NMR and ^{13}C NMR spectral data of compounds 1 and 1a

H	1	1a	^{13}C NMR (1)	
1α	1.06 *ddd*	1.07 *ddd*	1	38.2 *t*
1β	1.64 *m*	1.64 *m*	2	26.7 *t*
2α	1.67 *m*	1.69 *m*	3	72.4 *d*
2β		1.81 *m*	4	43.2 *s*
3α	3.58 *dd*	4.81 *dd*	5	59.6 *d*
5α	1.39 *d*	1.91 *d*	6	67.5 *d*
6β	3.91 *ddd*	5.10 *ddd*	7	52.0 *t*
7α	1.60 *m*	1.61 *dd*	8	73.3 *s*
7β	2.13 *dd*	2.10 *dd*	9	53.5 *d*
9α	1.13 *dd*	1.11 *dd*	10	38.5 *s*
11	1.33 *ddt*	1.34 *ddt*	11	23.1 *t*
11′	1.50 *ddt*	1.52 *ddt*	12	42.0 *t*
12	2.07 *m*	2.05 *m*	13	140.5 *s*
12′			14	117.3 *d*
14	5.03 *br dd*	5.02 *br dd*	15	30.1 *t*
15	2.80 *ddd*	2.74 *ddd*	16	84.4 *d*
15′	2.29 *ddd*	2.31 *ddd*	17	168.5 *s*
16	4.90 *br dd*	4.91 *br dd*	18	116.5 *d*
18	5.86 *dq*	5.87 *dq*	19	173.5 *s*
20	2.07 *d*	2.09 *d*	20	16.8 *q*
21	1.64 *s*	1.66 *br s*	21	16.3 *q*
22	1.17 *s*	1.24 *s*	22	25.4 *q*
23	3.69 *br s*	3.59 *d*	23	67.2 *t*
23′		4.09 *d*	24	12.5 *q*
24	0.80 *s*	0.88 *s*	25	14.0 *q*
25	0.83 *s*	0.98 *s*		
OAc		2.01 *s*		
		2.04 *s*		
		2.12 *s*		

J [Hz]: 1α,1β = 13; 1α,2α = 4.5; 1α,2β = 11; 2α,2β = 13; 2β,3α = 12; 2α,3α = 5; 5α,6β = 6β,7α = 11; 6β,7β = 4.5; 9α,11′ = 4; 11,11′ = 14; 11,12 = 11′,12′ = 7; 14,15 = 7.5; 14,15′ = 7; 15,15′ = 15; 15,16 = 4.5; 15′,16 = 6.5; 16,18 = 1.5; 18,20 = 1; (compound 1a: 23,23′ = 12).

The observed coupling constants required equatorial orientation of these groups. Spin decoupling indicated that one hydroxy group was at C-6 while the other one was at C-1 or C-3. A third hydroxy group was most probably at C-8. All data therefore agreed with the structure 1 and for the corresponding triacetate had structure 1a. NOE difference spectroscopy with 1a allowed the assignment of the methyl signals, the stereochemistry and also indicated that one hydroxy group was at C-3. Clear effects were observed between H-24, H-6 (8%), H-23 (5%) and H-23′ (5%), between H-25 and H-6 (10%), between H-9 and H-5 (8%), between H-22, H-6 (8%) and H-25 (8%) as well as between H-3 and H-5 (4%). Compound 1 has been named salvisyriacolide.



	R	R¹	R²
1	CH_2OH	H	OH
1a	CH_2OAc	Ac	OAc
2	CO_2Me	H	H

The isolation of sesterterpenes from two *Salvia* species may be of chemotaxonomic interest. Further investigations may show whether this rare type of natural product in higher plants [2] is more widespraed in the large genus *Salvia*.

EXPERIMENTAL

The air-dried aerial parts (200 g, collected 30 km N of Taleghan, Iran, voucher deposited in the Herbarium of the Dept of Botany, Shahid Beheshty University, Tehran) were extracted at room temp with MeOH–Et_2O–petrol (1:1:1). After evaporation the residue was treated with MeOH to remove longchain saturated compounds. The soluble part was separated by CC (silica gel). The polar fractions obtained with Et_2O–MeOH (9:1), were separated by TLC (Et_2O) and further by HPLC (RP 8, MeOH–H_2O, 7:3, *ca* 100 bar) affording 25 mg 1 (R_2 5.2 min.).

Salvisyriacolide (1). Colourless oil; IR $\nu_{max}^{CHCl_3}$, cm^{-1}: 3600 (OH), 1765 (γ-lactone), 1655 (C=C); CIMS (*i*-butane) *m/z* (rel. int.): 437 $[M+1]^+$ (12) ($C_{25}H_{40}O_6+1$), 419 $[437-H_2O]^+$ (16), 401 $[419-H_2O]^+$ (24), 383 $[401-H_2O]^+$ (38), 307 (100); $[\alpha]_D^{28}$ −18° ($CHCl_3$; *c* 0.2).

Compound 1 (10 mg) was acetylated with Ac_2O (2 hr, 70°) affording the triacetate (1a); colourless oil; CIMS (*i*-butane) *m/z* (rel. int.): 563 $[M+1]^+$ (0.5) ($C_{31}H_{46}O_9+1$), 545 $[563-H_2O]^+$ (3), 503 $[545-\text{ketene}]^+$ (3), 485 $[503-H_2O]^+$ (7), 425 $[485-\text{MeCOOH}]^+$ (25), 365 $[425-\text{MeCOOH}]^+$ (100); ^{1}H NMR spectral data: Table 1.

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