SPECTRAL PROPERTIES OF METHYL-2-*cis*-LACHNOPHYLLATE

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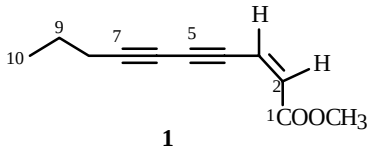
The composition of essential oil (EO) from *Lachnophyllum gossypinum* Bge. contains β-pinene, camphene, and methyl lachnophyllate (**1**), the content of which is 30% [1].

The structure of **1** isolated from the EO had empirical formula $C_{11}H_{12}O_2$ and mp 32.6-32.8°C and was characterized in 1935 [1] based on chemical data. The configuration of the disubstituted double bond was determined as *Z* (*cis*) in 1950 based on the total synthesis of its 2*E*-isomer [2]. This compound is called methyl lachnophyllate and has probably been observed in several other species of the Compositae family [3]. Both geometric isomers occurred together in *Chrysothamnus parryi* (Compositae) [4].



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EO was obtained by steam distillation on a Clevenger apparatus for 3 h from a total of 1850 g of freshly ground raw material. The yield was 10.3 g (0.55%), n_D^{23} 1.5269.

According to our data, the principal components (%) of the EO from *L. gossypinum* collected in July 2005 in the Moiynkumy desert of South Kazakhstan District were **1**, 80.1; β-pinene, 4.8; and caryophyllene, 1.0 (Table 1).

The chemical composition was studied by GC—MS in an Agilent 6890N instrument with an Agilent 5973N mass spectrometric detector. We used a DB-XLB FSC (30 m × 0.25 mm) quartz capillary column with He carrier gas at flow rate 1 mL/min. The GC column was held at 40°C for 10 min with temperature programmed to 240°C at a rate of 2°C/min and then held isothermally for 10 min. The sample was introduced without dividing the flow. The sample volume was 1 μL; vaporizer temperature, 250°C. Mass spectra were recorded in the range *m*/*z* 10-425. The percent composition of the EO was calculated from the peak areas without using correction coefficients. The qualitative analysis was based on comparison of retention times and complete mass spectra with standard oil components and pure compounds, if they were available, and with data in mass spectrometric libraries (Wiley 7th Ed., 390,000 spectra), NIST 02 (175,000 compounds).

Ester **1** was isolated by the following method. EO (7 g) dissolved in ethylacetate (10 mL) was treated with aqueous C_2H_5OH (15 mL, 60%) and cooled to 0°C. The liquid was decanted. The resulting crystals were repeatedly recrystallized from aqueous C_2H_5OH to afford **1**, mp 30-32°C, n_D^{25} 1.5518.

Mass spectrum (EI, 70 eV, *m*/*z*, I_{rel}, %): 176 (100) $[M]^+$, 161 (25), 147 (80), 145 (36), 133 (25), 119 (34).

UV spectrum (EtOH, λ_{max}, nm): 216, 225, 292, 308 (log ε 4.43, 4.45, 4.20, 4.18).

IR spectrum (KBr, ν cm^{-1}): 2959 (C–H), 2226 (C≡C), 1717 (C=O), 1601 (C=C), 1217, 1174, 822.

PMR spectrum (500 MHz, $CDCl_3$, δ, ppm, J/Hz): 0.98 (3H, t, $J_{9,10}$ = 7.5, 3H-10), 1.56 (2H, septet, $J_{9,8}$ = $J_{9,10}$ = 7.5, 2H-9), 2.32 (2H, td, $J_{8,9}$ = 7.5, $^7J_{3,8}$ = 1.0, H-8), 3.75 (3H, s, COOC$\underline{H}_3$), 6.17 (1H, dt, $J_{2,3}$ = 11.5, $^7J_{3,8}$ = 1.0, H-3), 6.19 (1H, d, $J_{2,3}$ = 11.5, H-2).

The *cis*-configuration of the disubstituted double bond was established based on the SSCC of $J_{2,3}$ = 11.5 [5].

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TABLE 1. Principal Components of *Lachnophyllum gossypinum* Essential Oil

Compound*	Content, %
α-Pinene	0.3
β-Pinene	4.8
β-Myrcene	0.5
Limonene	0.7
Camphor	0.8
Caryophyllene	1.0
1,1,5-Trimethyl-1,3-dihydroisobenzofuran-3-one	5.7
5,6,7,8-Tetrahydro-3-oxo-4-ethyl-3H-2-oxonaphthalene	1.0
Methyl-(*Z*)-dec-2-en-4,6-diynoate	80.1
Caryophyllene oxide	0.7

*Components are listed in order of increasing retention time.

**Components of content at least 0.3% are listed.

^{13}C NMR spectrum (125.75 MHz, $CDCl_3$): 13.38 (q, C-10), 21.34 and 21.45 (t, C-8 and C-9), 51.54 (q, O$\underline{C}H_3$), 65.03, 70.69, 86.48, 90.03 (s, C-4, C-5, C-6, C-7), 122.97 and 130.56 (d, C-2, C-3), 164.71 (s, C-1).

Thus, the chemical composition of EO from *L. gossypinum* was investigated for the first time by GC—MS. It was found that the principal component is methyl-dec-2-en-4,6-diynoate, the structure of which was confirmed by spectral methods.

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