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ESSENTIAL OIL FROM LYCOPUS EUROPAEUS

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In a phytochemical study of Lycopus europaeus L. (European bugleweed, family Labiatae) [1, 2], we have established the presence of β-sitosterol (0.09%), flavone (2.28%) and coumarin substances (0.12%), a nitrogenous base (choline, 0.18%), amino acids, and an essential oil which has not previously been studied. The content of essential oil obtained by distillation with steam from the air-dry epigeal part (in the flowering-fruit-bearing phase) amounts to 0.2% on the absolutely dry weight.

The oil is reddish with a characteristic odor and has the following constants: n_D^{20} 1.5217, d_{20}^{20} 0.9520, $[\alpha]_D^{20}$ +15°.

We studied the component fractions of the essential oil by gas-liquid chromatography on a Khrom-2 chromatograph with a flame ionization detector; the carrier gas was nitrogen and the rate of flow 20 ml/min. The analysis was carried out with a column 1.6 m long and 0.4 mm in diameter filled with Celite (80–100 mesh) upon which 15% of the weight of the carrier of poly(ethylene sebacate) was deposited. The analysis was carried out at 78° C (low-boiling fraction) and 184° C (high-boiling fraction), and the components of the essential oil were identified by their retention times and by the addition of known substances to the samples. In the low-boiling components we identified α-pinene camphene, limonene, γ-terpinene, p-cymene, and terpinolene. The investigation at 184° C showed the presence in the oil, in addition, of linaloyl acetate, linalool, camphor, bornyl acetate, geranyl acetate, nerol, and geraniol.

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ISOLATION OF 7-ACETOXYROYLEANONE FROM SALVIA NEMOROSA

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The family Labiatae includes more than 500 species of sage, Salvia, of which more than 70 grown in the Soviet Union [1]. We have made a chemical study of three samples of the roots of Salvia nemorosa L. (violet sage). Two of them were collected in September 1961 and September 1968 in the Poltava region and the third in July 1967 in the environments of Belogorsk (Crimea). Qualitative reactions (distillation with sodium hydroxide; magnesium acetate) and thin-layer chromatography on silica gel showed that each of the samples investigated contained three substances of quinone nature but no tanshinones.

Column chromatography yielded a yellow crystalline substance with the composition $C_{22}H_{30}O_5$, mp 195–198° C (yield 0.14%). The IR spectrum of the substance (mull in paraffin oil) exhibited absorption bands at (cm^{-1}) 3370 (OH), 1718, 1650, 1629, 1604. In a chloroform solution of the substance, the band at 1718 cm^{-1} is shifted to 1738 cm^{-1}, which is characteristic for ester carbonyl groups. The acid residue is acetyl, as follows from the presence in the NMR spectrum of the substance of a three-proton singlet at 1.97 ppm. The bands at 1650 and 1629 cm^{-1} also undergo a shift in the high-frequency direction in chloroform (to 1675 and 1645 cm^{-1}, respectively). The first band can be ascribed to a quinoid carbonyl and the second to a quinoid carbonyl bound by an intramolecular hydrogen bond to a neighboring hydroxy group. UV spectrum: λ_{max}^{EtOH} 272 mμ (log ε 4.15) and 406 mμ (log ε 2.93). By comparing the constants of the substance studied with literature data, and also taking into account the fact that all the quinones known for sage are phenanthrene derivatives, we have established that the quinone isolated from the roots of Salvia nemorosa L. is 7-acetoxyroyleanone, which has been obtained previously [2] from the roots of Inula royleana together with royleanone and 6,7-dehydroroyleanone.

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LACTONES OF THE FRUIT OF XANTHOGALUM PURPURASCENS

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From the fruit of Xanthogalum purpurascens Lallem. collected in the region of Dombaiskaya polyana (Northern Caucasus) we have established the presence of 7.4% of lactones consisting of a mixture of four components with Rf 0.92, 0.75, 0.55 (traces), and 0.00 (traces). The mobile system was n-hexane–benzene–methanol (5 : 4 : 1) and the stationary phase a 10% solution of formamide in methanol.

By chromatography on acid alumina we isolated two lactones: $C_{19}H_{20}O_5$, mp 82–83° C, $[\alpha]^{20}_D$ −129.3° (CH_3Cl), Rf 0.92 (A); and $C_{16}H_{14}O_5$, mp 147–148.5° C, Rf 0.75 (B), and also a substance $C_{29}H_{50}O$, mp 139–140° C (C).

Lactone A had a UV spectrum characteristic for a dihydropyrano- or dihydrofurocoumarin with a small peak in the 258 mμ region (λ_{max} 220, 258, 329, mμ; log ε 4.8, 3.76, 4.24). The UV spectrum of the lactone had absorption bands of of a lactone carbonyl (1728 cm^{-1}), of the −C=C− bond of an unsaturated α-pyrone (1630 cm^{-1}), of a −C=C− bond of an aromatic nucleus (1570, 1495 cm^{-1}), and of the stretching vibrations of an α-pyran ring (1393, 1350 cm^{-1}). The broad carbonyl band and also the change in Rf as a result of treatment with alkali showed the presence of an acyl residue in its molecule. Hydrolytic cleavage with alkali gave a hydroxylactone $C_{14}H_{14}O_4$, mp 183–183.5° C (from ether), $[\alpha]^{20}_D$ −6.8°, and angelic acid.

On the basis of mixed melting points and the physicochemical constants and IR spectra of the substance and the product of its hydrolysis, lactone A was identified as agasyllin (3'-angeloyloxy-3',4'-dihydropyrano-5',6':6,7-coumarin), which has been isolated previously in the crystalline state from Agasyllis latifolia (Bieb.) Boiss [1] and also in the form of an oil from Seseli libanotis (L.) Koch [2]. From its constants, IR spectrum, and a mixed melting point, lactone B was identified as isooxypeucedanin.

By its chemical properties (Liebermann-Burchard reaction for sterols), IR spectrum, and a mixed melting point, substance C was identified as β-sitosterol.

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