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18 March 1968

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UDC 547.972

FLAVONOIDS OF THE LEAVES OF POLYGONUM CORIARIUM. I

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Khimiya Prirodnykh Soedinenii, Vol. 4, No. 5, p. 321, 1968

From a methanolic extract of the leaves of Polygonum coriarium Grig. by absorption chromatography on polyamide with the use of preparative paper chromatography [with the systems 15% acetic acid and ethyl acetate–formic acid–water (10 : 2 : 3)] we have isolated three flavonoids.

It has been shown by the results of alkaline cleavage, reduction, acid hydrolysis, elementary analysis, spectroscopic investigations in the UV region using ionizing and complex-forming reagents, and IR spectroscopy [1–4] that the first of the substances isolated is quercetin, with mp 312° C, mp of the pentaacetate 194° C, R_f in 15% acetic acid 0.07; the second is avicularin [quercetin 3-(α-L-arabofuranoside)] with mp 216°–217° C, $[\alpha]_D^{22}$ −172.5° (c 0.68; methanol), R_f 0.46; and the third is quercitrin[quercetin 3-(α-L-rhamnofuranoside)] with mp 183°–185° C, $[\alpha]_D^{22}$ −160.6° (c 0.74; methanol), R_f 0.61.

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20 March 1968

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UDC 577.15/17:582.89

THE ESSENTIAL OIL OF ARTEMISIA LERCHEANA

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Khimiya Prirodnykh Soedinenii, Vol. 4, No. 5, pp. 321–322, 1968

On treating the epigeal part of Artemisia lercheana Web. et Stechm. collected in the Makhach-Kala district with steam we obtained 0.18% of essential oil (on the weight of the air-dry plant collected in the flowering period).

The physicochemical constants of the oil are as follows: d_4^{20} 0.9192, n_D^{20} 1.4680; $[\alpha]_D$ −29.28°; acid no. 0.14; ester no. 1762.

A preliminary fractionation of the essential oil was carried out by vacuum distillation. To isolate the individual components we used repeated chromatography on alumina, (neutral, activity grade II).

The individual components of the essential oil were identified by their IR spectra, by suitable derivatives, and by the results of gas-chromatographic analysis. The analysis was carried out on a UKh-1 chromatograph using as the stationary liquid phases PEG-400 (temperature of separation 120° C) and bis-(β-cyanoethyl) ether (temperature of separation 70° C), deposited on INZ-600 inert carrier (grain size 0.3–0.4 mm) in an amount of 20% of the weight of the carrier. The carrier gas was helium and the rate of flow 30–50 ml/min. Limonene was used as the standard substance for calculating the retention volumes.

It was found that the main component of the essential oil (~60%) is camphor mp 177°–178° C (subl.). A mixture with an authentic sample of camphor gave no depression of the melting point. The IR spectrum of the substance with mp 177°–178° C was identical with that of camphor [1].

The monoterpene fraction of the essential oil contained myrcene (saponification product of its adduct with maleic anhydride, mp 120° C), camphene (hydration product—isoborneol—with mp 211°–212° C), and p-cymene. The IR spectra of the compounds isolated from the essential oil coincided with the IR spectra of myrcene, camphene, and p-cymene given in the literature [1].

The results obtained were confirmed by gas-liquid chromatography. In addition, together with the hydrocarbons mentioned above, this fraction of the oil was found (but only by GLC) to contain α-pinene, β-pinene, limonene, γ-terpinene, β-phellandrene (?) and two unidentified hydrocarbons.

We also established the presence in the essential oil of 1, 8-cineole, with bp 62° C (14 mm); d_4^{20} 0.9230, n_D^{20} 1.4600; adduct with resorcinol, mp 80° C. In addition, the oil contains very small amounts of sesquiterpene alcohols which we have not studied.

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26 March 1968

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UDC 547.597

DERIVATIVES OF 3α-GLYCYRRHETIC ACID

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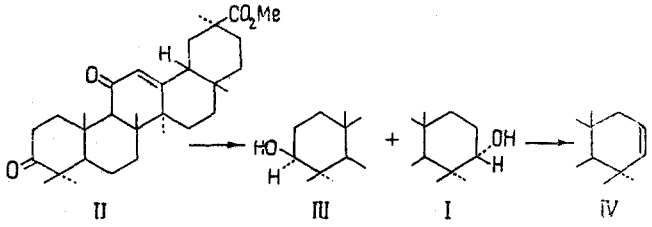
Khimiya Prirodnykh Soedinenii, Vol. 4, No. 5, pp. 322–323, 1968

We have obtained methyl 3α-glycyrrhetate (I) by the reduction of methyl 18 β H-3-oxoglycyrrhetate (II) with aluminum isopropoxide. The ratio of the yields of (I) and its 3β-epimer (III) was 6 : 4. Methyl epiglycyrrhetate has mp 217°–218° C; UV spectrum: $\lambda_{max}^{ethanol}$ 250 mμ (log ε 4.06); IR spectrum: ν 1728, 1665, and 1620 cm^{-1}. The substance gives an acetate with mp 220°–220.5° C; IR spectrum: ν 1257 cm^{-1} (OAc).



II III I IV

A proof of the axial orientation of the hydroxy group in (I) is the production of a Δ^2-compound (IV) with mp 202°–204° C on dehydration with phosphorus pentachloride in toluene. The structure of (IV) is confirmed by its IR spectrum, which contains a strong band with a frequency of 731 cm^{-1}, characteristic for a cis-disubstituted double bond.

25 November 1967

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