

# FUROCUMARINS OF THE ROOTS OF HERACLEUM STEVENII AND H. VILLOSA

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We have studied the coumarin composition of the roots of two species of cowparsnip—H. stevenii Manden and H. villosa sp. from the villosa group collected in the region of the village of Shuntuk (Krasnodar territory).

The combined coumarins were isolated from the roots and then separated into individual substances on alumina as described previously [1].

As a result, from the cowparsnip species mentioned, we isolated and identified the following furocoumarins: pimpinellin (mp 118–119° C,  $C_{13}H_{10}O_5$ ), isopimpinellin (mp 148–150° C,  $C_{13}H_{10}O_5$ ), bergapten (mp 188–191° C,  $C_{12}H_8O_4$ ), isobergapten (mp 221–223° C,  $C_{12}H_8O_4$ ), and sphondin (mp 190–191° C,  $C_{12}H_8O_4$ ).

From H. stevenii, in addition to the substances mentioned, we isolated angelicin (mp 139–140° C,  $C_{11}H_6O_3$ ), and from H. villosa xanthotoxin (mp 145–146° C,  $C_{12}H_8O_4$ ).

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# A STUDY OF THE COUMARIN COMPOSITION OF THE ROOTS OF PRANGOS ULOPTERA

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In the present paper, we give the results of a study of the coumarin composition of the roots of Prangos uloptera D.C., collected in the Bichenak mountains of the Nakhichevan Autonomous Soviet Socialist Republic.

The roots (12 kg) were exhaustively extracted with chloroform. The solvent was distilled off in vacuum, and the residue (670 g of resin) was chromatographed on a column of alumina (activity grade III). Elution was carried out with petroleum ether, a mixture of petroleum ether and chloroform, chloroform, and methanol. The rechromatography of the crystalline mixture obtained and repeated recrystallization from suitable solvents led to the isolation of seven substances of a coumarin nature.

From their composition, melting points, IR spectra, and mixed melting points, the compounds were shown to be identical with authentic samples of, respectively: osthole, isoimperatorin, oxypeucedanin, oxypeucedanin hydrate, pranferol, pranchimgin, and marmesin.

When the chloroform extract (9.97 g of resin) was chromatographed on a column of alumina (activity grade II), the following were isolated: osthole, isoimperatorin, oxypeucedanin hydrate, pranchimgin, and a substance with the composition  $C_{20}H_{24}O_6$  (I) and mp 139–140° C (from benzene);  $R_f$  0.19 ( $Al_2O_3$ , activity grade III, ethyl acetate).

Substance (I) exhibits the properties characteristic for a coumarin: it possesses a bright violet fluorescence in UV light and gives a color reaction with diazotized p-nitroaniline. The UV spectrum of (I):  $\lambda_{max}$  224, 252, 330, 334 m $\mu$  (log  $\epsilon$  3.71; 3.14; 3.63, respectively) and  $\lambda_{min}$  262 m $\mu$  (log  $\epsilon$  2.64) agrees with a furocoumarin structure. The deep minimum observed in the 260–270 m $\mu$  region is characteristic for 4',5'-dihydrofurocoumarins.

The IR spectrum of (I) has characteristic absorption bands at 3300 (OH), 1735 (CO of a  $\delta$ -lactone), 1622, 1565, 1505 (aromatic nucleus), 1382, 1362 (gem-dimethyl grouping), and 870  $cm^{-1}$  (furan ring). The UV and IR spectra of (I) are very similar to those of marmesin, which is a 4',5'-dihydrofurocoumarin.

By comparing the data obtained with those known from the literature, we concluded that from the substance (I) we have isolated is a new 4',5'-dihydrofurocoumarin, and we have called it ulopterole.

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## FLAVONIDS OF SORBARIA SORBIFOLIUM

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We have studied the flavonoids of Sorbaria sorbifolium L. (Ural false-spirea) collected in the Khekhtsirskii reserve (Khabarovsk territory).

From the distilled alcoholic extracts purified with chloroform and by repeated chromatographic separation on a column of polyamide sorbent, we isolated an individual substance (I) with the composition  $C_{21}H_{20}O_{11} \cdot 2H_2O$ , mp 230–231° C,  $[\alpha]_D^{20} -44.1^\circ$  (c 0.1; ethanol);  $\lambda_{max}$  267, 354 m $\mu$ , the chemical properties and UV and IR spectra of which corresponded to kaempferol 3-( $\beta$ -D-galactopyranoside)—trifolin [1, 2]. A direct comparison of substance (I) with trifolin confirmed their complete identity (the sample of trifolin was obtained from Prof. Aritomi Masakazu, Japan).

In the mother liquor, after the extraction of substance (I), substances were detected which could not be separated on a column of polyamide sorbent. Substances (II) and (III) were isolated by preparative separation on Fn-16 paper in the ethyl acetate—formic acid—water (10:2:3) system. Substance (II) with  $R_f$  0.68 was eluted with ethanol and was then subjected to acid hydrolysis. Quercetin and the sugar xylose were obtained. The attachment of the sugar at position 3 was shown by the citric acid—zirconium test [3], and, on the basis of this, substance (II) was characterized as quercetin 3-(xyloside). The hydrolysis of substance (III) ( $R_f$  0.59) gave the aglycone kaempferol and the sugar xylose. The position of attachment of the sugar was shown in a similar manner to that for substance (II). Substance (III) is kaempferol 3-(xyloside).

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## FLAVONOLS OF PRUNUS SPINOSA

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By separating on a Kapron column the flavonoids of the leaves of Prunus spinosa L. (family Rosaceae) we isolated glycoside 2, identified as kaempferol 7-(O- $\alpha$ -L-rhamnofuranoside) [1]. This glycoside was obtained by desorption with 45% ethanol. On elution with 40% ethanol, we obtained a mixture of glycoside 2 and glycoside 4, giving the color reactions characteristic for 3-substituted flavonols [2]. Attempts to separate glycoside 4 from glycoside 2 on Kapron were unsuccessful. To isolate glycoside 4, we used the different stabilities of the 3-glycosides and 7-glycosides in an alkaline medium. The action of a 0.5% aqueous solution of caustic potash in the boiling water bath for 2 hr led to the complete cleavage of the kaempferol 7-(rhamnoside), and the 3-glycoside was obtained in the pure state after the neutralization of the solution and purification on Kapron. Mp 210–212° C,  $[\alpha]_D^{20} -154^\circ$  (c 0.1; methanol).