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# COUMARINS OF THE ROOTS OF HERACLEUM SOMMIERI AND THE FRUIT OF H. ASPERUM

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In the roots of Heracleum sommieri Manden., collected in the Mestia region of the Georgian SSR, by chromatography on paper in the petroleum ether–formamide system [1] we have found eight compounds of a coumarin nature from which, by means of column chromatography on acidic alumina, we have isolated pimpinellin $C_{13}H_{10}O_5$ with mp 117–119° C, isopimpinellin $C_{13}H_{10}O_5$ with mp 148–149° C, bergapten $C_{12}H_8O_4$ with mp 188–189° C, and sphondin $C_{12}H_8O_4$ with mp 189–192° C.

In the fruit of H. asperum M. B. prepared in the Kazbek region of the Georgian SSR using the system given above we have found six coumarin substances. From them, on a column of alumina, we have obtained bergapten and a furocoumarin fluorescing brownish yellow in UV light with mp 148–151° C, $[\alpha]_D^{21}$ +24.5° (chloroform), which is readily acetylated (mp 105–108° C) and oxidized and forms a number of derivatives under the action of acids. It has not yet been possible to identify this substance. A third substance was eluted from the column together with the bergapten. It was possible to isolate this compound only by means of column partition chromatography on silica gel (stationary phase formamide, mobile phase a 1 : 1 mixture of petroleum ether and benzene). This substance, $C_{17}H_{16}O_5$, with mp 100–102° C, proved to be phellopterin [2].

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# FLAVONOIDS OF SERRATULA INERMIS

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We have studied the flowers of S. inermis Gilib. [1, 2] and S. tinctoria (dyers sawwort) ssp. eu-tinctoria Br.-Bl. By chromatography on Kapron [3] of a methanolic extract, we have isolated two substances of a flavonoid nature.

Substance (I), $C_{15}H_{10}O_5$, forms bright yellow acicular crystals with mp 348–350° C. Its acetyl derivative $C_{21}H_{16}O_8$ with mp 186–187° C has three acetyl groups.

Substance (II), $C_{15}H_{10}O_6$, forms yellow acicular crystals with mp 330–331° C (from 50% ethanol). Its acetyl derivative $C_{23}H_{18}O_{10}$ with mp 226–227° C has four acetyl groups.

In the cyanidin reaction, substance (I) forms an orange pigment which is extracted completely by octanol [4], which shows its aglycone nature. With a solution of ferric chloride it gives a green coloration, which may be an indication of the presence of a free 5-hydroxy group in it. The products of the alkaline fusion of the flavonoid are phloroglucinol and p-hydroxybenzoic acid.

In the cyanidin reaction, substance (II) forms an orange-red pigment which can be extracted from aqueous solutions with octanol. With ferric chloride it gives a green color and it reduces an ammoniacal solution of silver nitrate [5]. The products of alkaline fusion are phloroglucinol and protocatechuic acid.

From the results of a spectroscopic investigation in the UV region with ionizing and complex-forming reagents [6], a chromatographic comparison in several systems of solvents, and the absence of depressions of the melting points of substances (I) and (II) in admixture with apigenin and luteolin, respectively, flavonoid (I) can be characterized as 5,7,4'-trihydroxyflavone or apigenin and flavonoid (II) as 5,7,3',4'-tetrahydroxyflavone or luteolin.

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# SCROPHULEIN—A NEW FLAVONOID FROM SCROPHULARIA GROSSHEIMII

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From the herb S. grossheimii Schischk. we have isolated apigenin, luteolin, diosmetin, and a substance of a flavone nature which we have called scrophulein.

Scrophulein has the composition $C_{17}H_{14}O_6$, mol. wt. 314 (mass spectrometrically), mp 266–268° C (from 50% ethanol), $\lambda_{max}$ 345, 280 mμ, $R_f$ 0.05 (15% $CH_3COOH$), 0.51 (60% $CH_3COOH$), 0.31 [benzene–ethyl acetate–acetic acid–formamide (24.5 : 73.5 : 2 : 1)]. In the cyanidin reaction, the substance forms an orange pigment which can be extracted by octanol. On acid hydrolysis no cleavage was observed.

From the results of IR spectroscopy, scrophulein contains a hydroxy group (3400 $cm^{-1}$), a methoxy group (2960 and 2850 $cm^{-1}$), the carbonyl group of a γ-pyrone (1670 $cm^{-1}$), an aromatic system (1620, 1580, 1520, and 1510 $cm^{-1}$) and also 1,4- (820, 840 $cm^{-1}$) and 1,2,3,4,5-substitution (865 $cm^{-1}$) in benzene rings [1,2].

In the UV spectra, the maxima of bands I and II are of almost the same intensity, which is characteristic for flavones [3]. The functional groups were determined spectrophotometrically in the UV region, using diagnostic reagents. The following results were obtained: $\lambda_{max}$ with sodium acetate 350 and 290 mμ ($\Delta\lambda_1$ 5 mμ), $\lambda_{max}$ with sodium ethoxide 400 and 245 mμ ($\Delta\lambda_1$ + 55 mμ), $\lambda_{max}$ with zirconyl nitrate 365 and 300 mμ ($\Delta\lambda_1$ + 20 mμ).

Thus, the results of UV spectroscopy show that scrophulein contains free phenolic hydroxy groups in positions 5 and 4'.

On demethylation with hydriodic acid, scrophulein gave scutellarein. A spectroscopic investigation showed that with zirconyl nitrate scutellarein gives a bathochromic shift of +34 mμ, while apigenin gives one of 55 mμ. Consequently, there is probably a methoxy group in position 6 of scrophulein and a second methyl group substitutes the 7-hydroxy group.

By analyzing literature information on the various methoxy derivatives of scutellarein—hispidulin (6-methoxy-), pectolinarigenin (6,4'-dimethoxy-), and micanin (6,7,4'-trimethoxy-) [4]—it may be concluded that scrophulein is a new natural methoxy derivative of scutellarein—5,4'-dihydroxy-6,7-dimethoxyflavone.