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RUTIN AND HESPERIDIN FROM THE LEAVES OF Citrus limonia

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We have investigated the flavonoid compounds of the leaves of Citrus limon (L.) Burm. fil.(C. limonia Osbeck – Meyer's lemon) collected on the Dukhumi Experimental Station of Subtropical Crops of the N. I. Vavilov All-Union Scientific-Research Institute of Plant-growing [1].

The leaves (3.1 kg) were frozen with liquid nitrogen, homogenized, and fixed with boiling methanol. The homogenate was filtered, and the residue was extracted several times with 80% methanol on the boiling water bath. The filtrate and the extracts were combined and were evaporated in vacuum until the methanol had been eliminated, and the aqueous residue was treated repeatedly with chloroform. More than 10 flavonoid compounds were detected in the resulting extract by two-dimensional paper chromatography (direction I: butan-1-ol–acetic acid–water (4:1:5); direction II: 2% acetic acid). The fractionation of the combined flavonoids was carried out on a column of polyamide sorbent. Water and increasing concentrations of methanol in water (from 10 to 100%) were used as eluents. The flavonol fraction, eluted from the polyamide with 65% methanol, was separated on a column of microcrystalline cellulose (with elution by water), and the flavanone fraction, which was eluted from the polyamide with 95% methanol, was separated on a column of Sephadex LH-20 (with elution by methanol). This gave two substances (I and II).

The positions of the main absorption maxima in the UV spectra of substances (I) and (II) characterized them as flavonol and flavanone derivatives [2]. By PC in various solvent systems the products of the acid hydrolysis [3] of substances (I) and (II) were shown to include quercetin (compound I) and hesperetin (compound II) and glucose and rhamnose (compounds I and II). The oxidative degradation [4] of substances (I) and (II) gave the disaccharide rutinose (6-O-α-L-rhamnosyl-D-glucose). The performance of qualitative reactions [5] and of spectral investigations with ionizing and complex-forming reagents [2] showed that the rutinose was attached to the aglycone of substance (I) in the C-3 position and to the aglycone of substance (II) in the C-7 position.

The physicochemical constants obtained, the spectral indices (UV and IR spectra), and a chromatographic comparison with authentic samples permitted compounds (I) and (II) to be identified as rutin and hesperidin.

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FLAVONOIDS OF Solidago canadensis AND S. virgaurea

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Continuing an investigation of goldenrod [1], we give the results of a study of two species of the plant Solidago canadensis L. Canada goldenrod) and S. virgaurea L. (Eurpean goldenrod), family Asteraceae (Compositae), collected at the beginning of the flowering period.

The dried and comminuted flowers and leaves of S. canadensis were extracted with 95% ethanol, the extract was evaporated until the solvent had been driven off completely, and the residue was diluted with water. To free the flavonoids from accompanying substances, the extract was treated successively with chloroform, butanol, and ethyl acetate. The flavonoid substances were separated on polyamide sorbent with elution by ethanol–chloroform (1:4) and were then crystallized from water–acetone (1:1).

Substances (I-III) were isolated from the ethyl acetate extract. From their qualitative reactions, physicochemical properties, and UV spectra with diagnostic reagents [2], and also by chromatography with markers, the substances isolated were identified as a number of aglycones of the flavonol group.

Substance (I) – composition $C_{15}H_{10}O_6$; mp 275-277°C; UV spectrum: 371, 272 nm (in methanol)* – was 3,4',5,7-tetrahydroxyflavone (kaempferol).

Substance (II) – composition $C_{15}H_{10}O_7$; mp 311-314°C. The UV spectrum (370, 257 nm, in methanol) did not differ from that of 3,3',4',5,7-pentahydroxyflavone (quercetin).

Substance (III) – composition $C_{16}H_{12}O_7$; mp 305°C (decomp.); UV spectrum: 368, 254 nm (in methanol) – was identified as 3,4',5,7-tetrahydroxy-3'-methoxyflavone (isorhamnetin).

The separation on polyamide of the butanolic extract yielded substances (IV-VII) which, according to the preliminary results of a chemical study, were flavonoid biosides. D-glucose and L-rhamnose were detected in the products of the hydrolysis with 5% hydrochloric acid of substance (IV-VII), together with the aglycones kaempferol, isorhamnetin, rhamnetin (3,3',4',5-tetrahydroxy-7-methoxy flavone), and quercetin, respectively.

From their UV spectra with diagnostic reagents it was established that in all the substances isolated the sugar components were present in position 3 in the form of biosides. Rhamnose was the terminal sugar residue. All the substances isolated were hydrolyzed by the enzyme rhamnodiastase, which confirmed the β configuration of the glycosidic center of the glucose.

Substance (IV) – composition $C_{27}H_{30}O_{15}$; mp 173-175°C; UV spectrum, 350, 266 nm (in methanol) – was identified as kaempferol 3-O-β-D-glucorhamnoside.

Substance (V) – composition $C_{28}H_{32}O_{16}$; mp 171-173°C; UV spectrum: 353, 255 nm (in methanol) – consisted of isorhamnetin 3-O-β-D-glucorhamnoside.

Substance (VI) –composition $C_{28}H_{32}O_{16}$; mp 184-187°C; UV spectrum: 357, 257 nm (in methanol) – was identified as rhamnetin 3-O-β-D-glucorhamnoside.

Substance (VII) – composition $C_{27}H_{30}H_{16}$; mp 188-192°C; UV spectrum: 358, 258 nm (in methanol) – was identical with rutin (quercetin 3-O-β-D-rutinoside).

*The absorption maxima in the UV spectra of substances (I-VII) are given.

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