6. Kaempferol 3-O-β-D-glucoside 2"-gallate, $C_{28}H_{24}O_{15}$, mp 227-229°, $[\alpha]_D^{20}$ −84° (c 1.0; methanol).

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FLAVONOIDS, PHENOLIC ACIDS, AND HYDROXYCOUMARINS FROM THE FRUIT OF VARIOUS SPECIES OF THE GENUS Berberis

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Continuing a study of the fruits of five species of barberry (Family Berberidaceae) [1, 2], we have investigated the phenolic compounds.

The dry comminuted fruits of Berberis vulgaris L., B. coreana Palib., B. sieboldii Miq., B. integerrima Bunge, and B. sphaerocarpa Kar. et Kir. (20-50 g of each species), freed from seeds, were extracted with 80% ethanol three times in the water bath at the boiling point of the solvent in a flask with a reflux condenser. In each case, the combined extract was filtered, evaporated to an aqueous residue, and mixed with Woelm DC polyamide (FRG). After this, the mixture obtained was dried at 60°C, ground, and filled into a column. The substances were eluted with distilled water and with 10, 50, and 96% ethanol. The fractions collected were chromatographed by the two-dimensional ascending method on Filtrak FN 12 paper in two solvent systems: 1) butan-1-ol–CH_3COOH (glacial)–H_2O (3:1:1) and 2) 15% acetic acid (second direction). Some of the fractions were combined and evaporated to dryness, and the residues were dissolved in 50 ml of 80% ethanol. These solutions were used for hydrolysis and the subsequent identification of the phenolic compounds.

The hydrolysis of ethanolic extracts of the fruits of five species of barberry showed that the flavonoid glycosides were represented by five aglycones – quercetin, isorhamnetin, kaempferol, apigenin, and luteolin, which we identified chromatographically with authentic samples and by spectral analysis [3, 4].

The separation of the phenolic substances was carried out in the same solvents by paper chromatography. It was established that the fruits of these species of barberry contained 15 compounds of flavonoid nature and one compound of hydroxycoumarin nature, together with four phenolic acids [3, 5].

The individual components of the phenolic substances and the hydroxycoumarin compound were obtained by preparative paper chromatography of the ethanolic extracts in the systems given above. The spots of the substances were eluted from the chromatograms with 96% ethanol. From the result of the Bryant cyanidin reaction, substances 1-11 were assigned to the glycosides and 12-15 to the flavonoid aglycons. These substances were identified by acid hydrolysis, paper chromatography with markers, and specific color reactions, and also by a study of their spectral characteristics in the UV region with diagnostic reagents [6].

The phenolic carboxylic acids and the hydroxycoumarin compounds were studied from their fluorescence in UV light (λ 254 and 360 nm) in the presence of ammonia and in its absence,

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by some color reactions, and by their hydrolysis products, and also with the aid of spectral methods and paper chromatography with authentic samples [7].

On the basis of the results obtained, the structures of seven substances were established, these being identified as rutin, hyperin, isoquercitrin, quercitrin, caffeic acid, chlorogenic acid, and esculetin. The other compounds were present in minor amounts but the aglycons of six of them were determined – quercetin, kaempferol, isorhamnetin, apigenin, and luteolin.

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FLAVONOIDS OF Astragalus macropterum

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In the epigeal part of Astragalus macropterum (bird-egg pea) C. D. collected in the Tadzhik SSR (environs of the village of Ramit) in the flowering period (May-June, 1985), more than 15 polyphenolic compounds, 10 of which have been assigned to the flavonoids, have been detected by paper chromatography.

The flavonoids were exhaustively extracted with 70-96% ethanol, the ethanol was distilled off in vacuum, and the residue was purified with chloroform. The purified aqueous residue was treated with ethyl acetate. The ethyl acetate was distilled off and the residue was chromatographed on a column of polyamide sorbent. Four flavonoids (I-IV) were isolated. On the basis of the Bryant test [1], substances (I), (II), and (III) were assigned to the aglycons, and (IV) to the glycosides.

Substance (I): $C_{15}H_{10}O_5$, mp 349-350°C; the melting point of its acetate (183-185°C) was similar to that of apigenin triacetate. Phloroglucinol and p-hydroxybenzoic acid were identified in the products of alkaline degradation. Compound (I) was identified as apigenin.

Substance (II): $C_{15}H_{10}O_6$, mp 275-277°C; temperature of the acetate 181-183°C. By comparison with an authentic sample, compound (II) was identified as kaempferol.

Substance (III): $C_{15}H_{10}O_7$, mp 308-309°C, was identified as quercetin.

Substance (IV): $C_{21}H_{20}O_{10}$, mp 256-258°C, $[\alpha]_D^{20}$ −50.5° (c 0.1; ethanol). On acid hydrolysis, the aglycon apigenin and glucose were detected. UV spectrum, λ_{max}, nm: 270, 340; $+CH_3COONa$, 270, 340 [2]. The absence of a bathochromic shift for the glycoside under the influence of sodium acetate indicated the attachment of the sugar moiety at C_7. Compound (IV) was identified as apigenin 7-O-β-D-glucoside.

Two compounds of flavonoid nature (V) and (VI) were isolated from the aqueous residue with the aid of preparative paper chromatography.

Substance (V) was identified as rutin (quercetin 3-O-rutinoside), $C_{27}H_{30}O_{16}$, mp 190-191°C (aqueous ethanol); $[\alpha]_D^{20}$ −32.2° (c 0.3; methanol); λ_{max} 260, 360 nm [3].

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