The elementary analysis corresponds to the calculated figures for both the composition given previously, $C_{21}H_{20}O_{10}$, and also the new one, $C_{23}H_{22}O_{11}$.

The isolation of methyl (apigenin 7-galactosid)uronate has been reported by Ahmed et al. [2], and these authors considered the substance to be native and not a consequence of extraction with methanol. We do not exclude the possibility that the plant contains free apigenin 7-glucosiduronic acid which is converted into the ethyl ester during extraction with ethanol on heating.

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FLAVONOIDS OF Atraphaxis muschketovii

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We have previously [1-3] reported an investigation of the flavonoid composition of the leaves of two species of Atraphaxis of the 14 found in the territory of Kazakhstan — Atra-phaxis pyrifolia and Atraphaxis frutescens, belonging to the subgenus Tragopyrun. Atra-phaxis muschketovii Krassn., which is widely distributed in the mountains of the Trans-Ili Ala-Tau belongs to the same subgenus. A bioecological investigation of some species of the family Polygonaceae has shown that this species is characterized by a high flavonoid content in the leaves. The flavonoid complex was isolated and separated in the same way as the flavonoids of Atraphaxis pyrifolia, the total flavonoids being obtained by extracting the leaves with acetone. The extract was evaporated and the residue was treated with a small amount of hot water and was extracted successively with benzene, ether, and ethyl acetate. From the ethyl acetate extract by chromatography on silica gel (with elution by chloroform-methanol in various proportions) followed by purification on polyamide, two flavonoid glycosides were isolated: I) with mp 158-160°C, $[\alpha]_D^{25}$ —118.1° (c 0.4; methanol; R_f 0.80 [I-BAW (4:1:5)] and 0.78 (II-15% CH₃COOH), and (II) with mp 183-185°C, $[\alpha]_D^{25}$ —110.5° (c 0.29; methanol), R_f 0.58 (I) and 0.63 (II).

On the basis of the results of physicochemical analysis (qualitative reactions, acid and alkaline hydrolysis, alkaline cleavage, acylation, and methylation, and IR, UV, and NMR spectroscopy) and a comparison with the glycosides that we isolated previously from the leaves of $Atraphaxis\ pyrifolia\ [5]$, glycoside (I) was identified as 8-acetoxy-3,3',4',5-tetrahydroxy-7-methoxyflavone 3-0- α -L-rhamnopyranoside, and (II) as 3,3',4',5,8-pentahydroxy-7-methoxyflavone 3-0- α -L-rhamnopyranoside. Quantitative determinations of these flavonoids were performed by the spectrophotometric method after the chromatogram had been treated with a 1% ethanolic solution of AlCl₃. Found: glycoside (I) - 0.32%; (II) - 0.19% (calculated on the absolutely dry raw material). In the ethereal extract, after isolation by the bicarbonate method, by paper chromatography [6] and a comparison with markers we identified p-hydroxybenzoic, protocatechuic, and caffeic acids. It follows from a comparison of the chemical compositions of the three species of Atraphaxis mentioned that Atraphaxis pyrifolia and Atraphaxis muschketovii have the same composition and differ considerably from Atraphaxis frutescens, the polyphenol complex of which shows the quercetin and kaempferol glucosides that are characteristic for the Polygonaceae family.

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STRUCTURE OF PATULOSIDE

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We have previously [1] reported the isolation from Campanula patula L. of a new flavone glycoside with the composition $C_{20}H_{18}O_{10}$, mp 211-213°C, $[\alpha]_D^{20}$ -67° (c 0.3; MeOH), which was characterized as luteolin 7-0- β -D-xylofuranoside (patuloside). However, in the course of a further investigation of this compound, doubt arose relative to the furanose form of the sugar. The basis for this was features of the NMR spectroscopy of patuloside derivatives.

The NMR spectrum of the trimethylsilyl ether in CCl₄ (100 MHz, 0 - TMS) showed the signals of six aromatic protons: multiplet at 7.22-7.50 ppm (H-2',6'), doublets at 6.83 ppm, J = 8 Hz (H-5'), 6.56 ppm, J = 2.5 Hz (H-8), and 6.24 ppm, J = 2.5 Hz (H-6), and a singlet at 6.28 ppm (H-3). A doublet at 4.82 ppm (J = 6.5 Hz) shows the β configuration of the anomeric proton of the xylose. The other five protons of the pentose formed a multiplet in the 3.3-3.9 ppm region.

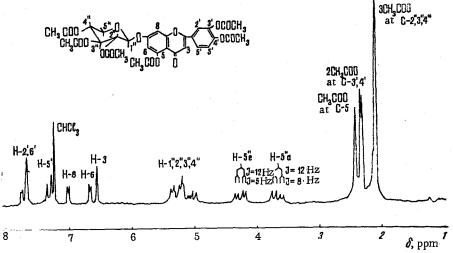


Fig. 1. NMR spectrum of patuloside hexaacetate in CDCl₃ (100 MHz, internal standard PMS).

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