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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-O-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 CCl_4 (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.

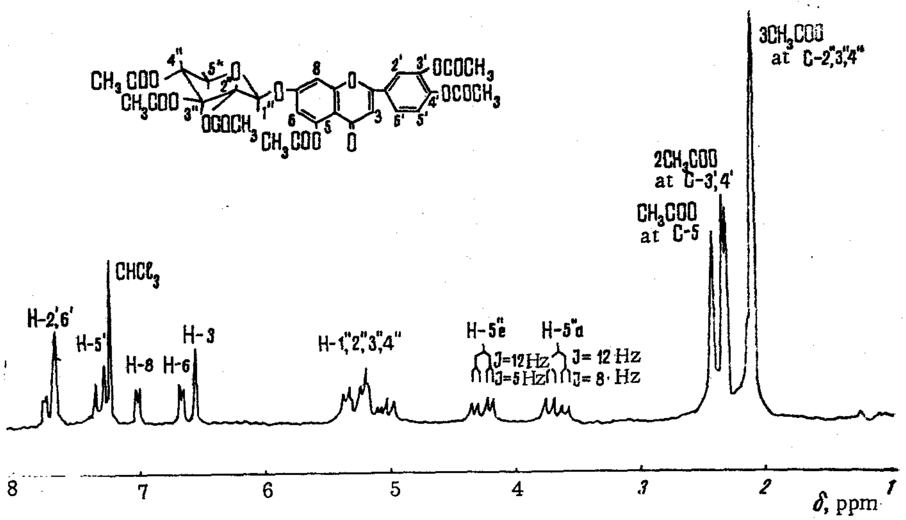

Fig. 1. NMR spectrum of patuloside hexaacetate in $CDCl_3$ (100 MHz, internal standard PMS).

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Acetylation of patuloside gave a hexaacetate with the composition $C_{32}H_{30}O_{16}$, mp 229-231°C, $[\alpha]_D^{20}$ $-45.4°$ (c 0.528; chloroform), ν_{CO} 1780, 1760, 1660 cm^{-1}.

The pyranose form of the sugar is shown by the multiplicity of the signals of the methylene protons in the NMR spectrum of the acetate (Fig. 1) and by the large shift between the H-5"a and the H-5"e signals. The spin–spin coupling constants of the anomeric proton in the TMS ether and of the proton at C-5" in patuloside acetate agree well with the $^4C_1$ conformation of D-xylopyranose in these compounds.

To confirm the pyranose form of the sugar, the glycoside residue was methylated by Hakomori's method [2] and the full methyl ether obtained was subjected to hydrolysis, which gave 2,3,4-tri-O-methyl-D-xylose, identified by TLC on silica gel and by GLC in the presence of a marker.

Thus, the results obtained show that patuloside has the structure of luteolin 7-O-β-D-xylopyranoside.

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DZHAMIRONE – A NEW KETONE FROM THE ROOTS OF *Ferula dshaudshamyr*

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From a methanolic extract of the roots of *Ferula dshaudshamyr* Eug. Kor., by chromatography on a column of type KSK silica gel we have isolated an oily substance with the composition $C_{23}H_{32}O_3$, M^+ 356, R_f 0.78 [petroleum ether–ethyl acetate (4:1)], readily soluble in organic solvents and insoluble in water which we have called dzhamirone. Its UV spectrum [λ_{max} 230, 275, and 312 nm (log ε 3.90, 4.16, and 3.85, respectively)] is characteristic for a 2,4-dihydroxybenzoyl chromophore. IR spectrum, ν_{max}, cm^{-1}: 3350 (phenolic hydroxy group), 1630 (carbonyl of an aryl alkyl ketone) [1], and 1610 and 1520 (aromatic nucleus). The molecule of this substance contained two phenolic hydroxyls, which was confirmed by the formation of a diacetate (M^+ 440).

The NMR spectrum of the substance has the signals of three aromatic protons – doublet at 7.50 ppm (J = 10 Hz, 1 H), quartet at 6.22 ppm (J_1 = 10 Hz, J_2 = 2 Hz, 1 H), and doublet at 6.25 ppm (J = 10 Hz, 1 H), and also a singlet at 12.86 ppm (1 H), corresponding to the proton of a phenolic hydroxy group bound by a hydrogen bond with a ketone carbonyl [1]. A multiplet in the 5.00-ppm region (3 H) corresponds to three olefinic protons. In the strong field there are signals at 1.54 (6 H), 1.56 (3 H), and 1.58 ppm (3 H), corresponding to four methyl groups on double bonds.

Thus, dzhamirone is an aryl alkyl ketone the terpenoid part of which has the composition $C_{15}H_{25}$.

On hydrogenation over PtO_2 in ethanol, dzhamirone absorbed three moles of hydrogen. This showed that the terpenoid part of the molecule contains three double bonds. In the NMR spectrum of the hydrogenation product (M^+ 362), the signals of the three olefinic protons and of the methyl groups on double bonds had disappeared, and a doublet had appeared at 0.86 ppm (J = 7.5 Hz, 12 H). With the given composition and with three double bonds, the terpenoid substituent can only have an acyclic structure.

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