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FLAVONOIDS OF THE BUDS OF Populus deltoides

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UDC 547.972

There is only one communication in the literature on the flavonoid composition of the eastern or Canadian poplar, Populus deltoides Marsh, family Salicaceae, from the bark of which was isolated 6-C-methyldihydroquercetin and its glycosides [1].

Freshly gathered buds of eastern poplar, obtained in March, 1988, in the Kuibyshev botanical garden, were extracted with aqueous alcohol, and the extract obtained was evaporated in vacuum to a viscous residue, which was chromatographed on a column of silica gel using as eluents mixtures of hexane and chloroform and of ethanol and chloroform in various proportions. This permitted the isolation of ten flavonoid substances, which were assigned to the flavanones (I and II), the flavanonols (III), the flavones (IV-VI), and the flavonols (VII-X).

For the identification of the substances isolated we used UV, PMR, and mass spectroscopies, and also direct comparison with authentic samples [compounds (I), (II), and (IV-X)].

Pinostrobin (I) (5-hydroxy-7-methoxyflavanone) – white acicular crystals with the composition $C_{16}H_{14}O_4$ (M^+ 270), mp 96-98°C (from chloroform–petroleum ether).

Pinocembrin (II) (5,7-dihydroxyflavanone) – light yellow crystals with the composition $C_{15}H_{12}O_4$ (M^+ 256), mp 193-195°C (acetone).

The flavanone natures of compounds (I) and (II) followed from their UV spectra (λ_{max} 289 nm) [2] and their PMR spectra, each of which contained the signals of a proton at C-2 in the form of a doublet of doublets with SSCCs 4 and 12 Hz (chemical shift ~5.5 ppm) and the signals of protons at C-3 in the form of two doublets of doublets with the SSCCs 12 and 17 Hz for the axial proton at 3.2 ppm and 4 and 17 Hz for the equatorial proton at 2.8 ppm.

Analysis of the spectral characteristics, and also a comparison of physicochemical constants permitted the identification of compounds (I) and (II) as 5-hydroxy-7-methoxy- and 5,7-dihydroxyflavanones, respectively.

Alpinone (III) (3,5-dihydroxy-7-methoxyflavanone) – white acicular crystals with the composition $C_{16}H_{14}O_7$ (M^+ 286), mp 176-179 (ethanol). Compound (III) was the flavanonol analogue of pinostrobin (I), as followed from its PMR spectrum (deuteroacetone), which contained, in particular, two doublet signals with SSCCs of 17 Hz at 3.24 and 2.29 ppm belonging to the H-2 and H-3 protons, respectively. The flavanonol nature of compound (III) was also confirmed by its UV spectra (λ_{max} 290 nm) [2], and also by the characteristic peak of the M–17 ion with m/z 269 (68%) in its mass spectrum [1].

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0009-3130/90/2604-0466$12.50

The presence in the PMR spectrum of compound (III) of the singlet signal of the proton of the 5-OH group at 11.98 ppm and also the presence in the mass spectrum of the peak of an ion A_1 with m/z 166 (81%) indicated the methylation of the 7-OH group [1].

Chrysin (IV) (5,7-dihydroxyflavone) – yellow crystals with the composition $C_{15}H_{10}O_4$ (M^+ 254), mp 282-285°C (ethanol).

Tectochrysin (V) (5-hydroxy-7-methoxyflavone) – yellow acicular crystals, $C_{16}H_{12}O_4$ (M^+ 268), mp 144-147°C (from chloroform–petroleum ether).

Apigenin (VI) (4',5,7-trihydroxy flavone) – light yellow crystals with the composition $C_{15}H_{10}O_5$ (M^+ 270), mp 341-343 °C (aqueous ethanol).

Galangin (VII) (3,5,7-trihydroxyflavone – light yellow crystals with the composition $C_{15}H_{10}O_6$ (M^+ 270), mp 220-223°C (ethanol).

Kaempferol (VIII) (3,4',5,7-tetrahydroxyflavone) – yellow crystals with the composition $C_{10}H_{10}O_6$ (M^+ 286), mp 285-287°C (aqueous ethanol).

Quercetin (IX) (3,3',4',5,7-pentahydroxyflavone) – yellow crystals with the composition $C_{15}H_{10}O_7$ (M^+ 302), mp 302-304°C (chlf.–MeOH).

Isorhamnetin (X) (3,4',5,7-tetrahydroxy-3'-methoxyflavone) – yellow crystals with the composition $C_{16}H_{12}O_7$ (M^+ 316), mp 301-304°C (chlf.–MeOH).

Each of the flavonoids (VII-X) contained a free 3-OH group, while compound (X) also had a methoxy group (presence in the PMR spectrum of a singlet three-proton signal at 3.96 ppm. The methylation of the 3'-OH group in compound (X) was shown by its UV spectra, where the presence of sodium acetate caused a bathochromic shift of the short-wave band (free 7-OH group) and the presence of sodium methanolate caused a decrease in the intensity of the long-wave band [2], and also by its mass spectrum, which contained the peak of a B_2 ion with m/z 151 (24%) [1].

It is interesting that we have isolated compounds (I), (II), and (IV-VIII) previously from buds of the balsam or southern poplar, while compounds (IX) and (X) have been described only for the buds of the black poplar [4].

So far as concerns alpinone (III), this is the first time that it has been isolated from a plant of the genus Populus L.

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