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A NEW DIHYDROFUROCOUMARIN FROM Smyrniopsis aucheri

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UDC 947.918

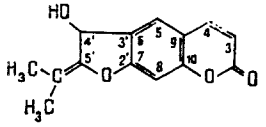
The roots of the Smyrniopsis aucheri Boiss. (smyrniopsis) collected in the flowering and fruit-bearing phase close to the village of Kuku in the Shakhbuz region of the Nachikhevan ASSR have been investigated. Thin-layer chromatography on Silufol showed that an ethanolic extract of the roots contained not less than 12 coumarin compounds. Five coumarins have previously been extracted from a plant of this species and characterized [1-3].

From an ethanolic extract, by chromatography on a column of silica gel, we isolated another seven coumarin compounds. On the basis of a study of their UV, IR, NMR, and mass spectra and physicochemical characteristics we have established the structure of one of them, which we have called nachsmyrin (I), $C_{14}H_{12}O_4$, M^+ 244, mp 135-136°C. The presence in the UV spectrum of absorption maxima at 220, 225, 247, 253, and 303 nm (log ε 3.99, 3.96, 4.17, 4.20, and 3.86, respectively) showed that the substance was a 7-O-substituted coumarin.

In the IR spectrum, absorption bands were observed at (cm^{-1}) 3500 (OH), 1730 (C=O), 1620, 1585, and 1455 (aromatic ring), which are characteristic for dihydrocoumarin derivatives [4]. The mass spectrum of the subtances contained the peaks of ions with m/z 244 (M^+), 227 (M^+–OH), 201 ($M^+ - =C\begin{smallmatrix}CH_3\\CH_3\end{smallmatrix}$) 198 ($M^+ - 2CH_3$, –O) 187, 158, 155, 131, 101. In the NMR spectrum (100 MHz, $CDCl_3$) of nachsmyrin signals were observed from two methyl groups at a double bond, 1.68 (6H, s, $2CH_3$); of a gem-hydroxylic, 2.93 (1H, s), and from the aromatic protons of a coumarin nucleus: 6.18 (1H, d, J = 10 Hz, H-3); 6.77 (1H, s, H-8); 7.19 (1H, s, H-5) and 7.61 (1H, d, J = 10 Hz, H-4).

When (I) was acetylated with acetic anhydride in pyridine, a monoacetate $C_{16}H_{14}O_5$, M^+ 286, mp. 121-122°C, was obtained, as was confirmed by a pronounced decrease in the absorption band of a hydroxy group in the IR spectrum, by the presence of a three-proton singlet at 2.02 ppm, and by a paramagnetic shift of the signal of the gem-hydroxylic proton in the NMR spectrum. The mass spectrum of the monoacetate contained the peaks of ions with m/z 286 (M^+) and 244 ($M^+ - 42$).

On the basis of the facts given above, and in the light of the biogenesis of similar coumarins in smyrniopsis, it may be concluded that nachsmyrin has the following structural formula



I

A study of the structures of the other coumarins that we isolated from this plant is continuing.

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FLAVONOIDS AND STEROLS OF Physochlaina physaloides

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UDC 547.972:547.926

The isolation of coumarins from the epigeal part of the Physochlaina physaloides has been reported previously. We have continued a study of the roots of this plant.

When the hexane fraction [1] was investigated by preparative chromatography on silica gel (in the hexane-acetone (95:5) by volume system), β-sitosterol (I), mp 134-136°C, M^+ 414, and β-sitosterol β-D-glucopyranoside (II), mp 265-267°C were isolated. They were identified by the methods of NMR and mass spectroscopy [2].

The column chromatography of chloroform and ethyl acetate extracts (chloroform–ethanol (97:3) system) yielded compounds (III) and (IV). On the basis of its PMR spectra, substance (III) was identified as liquiritigenin (7,4'-dihydroxyflavanone), and this was confirmed by the results of mass spectrometry and ^{13}C NMR.

Compounds (IV) proved to be a monoglycoside of quercetin. The PMR spectrum of its peracetate showed that the carbohydrate moiety was galactose (double resonance). The position of attachment of the sugar residue to the aglycon and the size of the oxide ring [3] were determined with the aid of ^{13}C NMR spectra, and in this way component (IV) was identified definitively as quercetin 3-O-β-D-galactopyranoside. Compound (III) had the formula $C_{15}H_{12}O_4$, mp 204-206°C (from ethanol), M^+ 256, and compound (IV) $C_{21}H_{20}O_{12}$, mp 230-234°C (from ethanol), $[\alpha]_{546}^{20}$ −94.4° (c 0.36; pyridine).

It is interesting to note that flavonoids having no oxygen function in the C-5 position (as in liquiritigen) are found fairly rarely in plants. Such compounds may serve as a good "marker" in the chemosystematics of the genus Solanaceae.

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