COMPONENTS OF THE ROOTS OF XANTHOGALUM SACHOKIANUM

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From the roots of Xanthogalum sachokianum collected in the Kubin region of the Azerbaidzhan SSR we have isolated compound I, with the composition $C_{17}H_{18}O_6$, mp 127–128° C (from methanol), $[\alpha]_D^{20}$ −34.4° (c 0.9, ethanol), mol wt 318 (mass spectrometry).

The UV spectrum of I (λ_{max} 231, 251, 259, and 296 mμ, log ε 4.33, 4.36, 4.36, and 4.06) shows that the substance belongs to the chromone group [1–3]. Its IR spectrum has absorption bands at (cm^{-1}) 1737 (—C=O of an ester), 1650, 1638, and 1589 (vibrations of the C=O and —C=C— bonds of γ-pyrone and aromatic rings).

The saponification of I gave compound II, with the composition $C_{15}H_{16}O_5$, mp 199–201° C (from methanol), mol wt 276 (mass spectrometry), and an acid identified by paper chromatography as acetic acid. In the IR spectrum of II the band at 1737 cm^{-1} disappeared and the peak of a free hydroxyl group appeared at 3530 cm^{-1}.

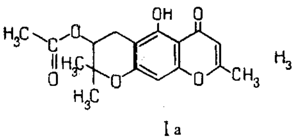
According to elementary analysis, the molecule II has a second hydroxy group which, judging from the positive reaction of the substance with ferric chloride, has a phenolic nature. A diffuse absorption band in the 2500–2750-cm^{-1} region belonging to a phenolic hydroxyl shows the formation of an intramolecular hydrogen bond [3], which is also confirmed by the presence of a one-proton singlet at 12.88 ppm in the NMR spectrum of the hydrolysis product. The presence of the latter in the NMR spectrum of the initial compound as well (δ 12.93) shows that it contains a phenolic hydroxyl. The table gives the NMR spectra of I and II and the assignment of the signals.

Table

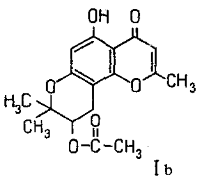
Sub stance	C_2–Me (3H)	H_3 (1H)	C_5–OH (1H)	H_8 (1H)	$C_2$1–Me (6H)	$H_3$1 (1H)	$H_3$1–Ac (3H)	$H_4$1 (2H)
I	2,30 s	5,95 s	12,93 s	6,25 s	1,26 s	5,05 t	2,05 s	2,85 m
II	2,26 s	5,95 s	12,88 s	6,25 s	1,35 1,38 s	3,61 m	— —	2,76 m

Note. s) singlet, t) triplet, m) multiplet.

The results obtained permit structure Ia or Ib to be proposed for compound I.



Ia



Ib

Structure Ia corresponds to natural hamaudol 3'-acetate, $C_{17}H_{18}O_6$, mp 125–126.5° C, isolated from Angelica polymorpha Maxim. [4], and compound II to hamaudol, $C_{15}H_{16}O_5$, mp 198–200° C, isolated from the roots of A. japonica A. Gray [5] and obtained by the hydrolysis of seselirin [6]. A direct comparison of the physical constants and the IR and NMR spectra of the product of the hydrolysis of seselirin and I, and also the absence of a depression of the melting point of a mixture, shows that they are identical. Thus, compound I is 3'-acetoxy-2',2'-dimethyl-3',4'-dihydropyrano-5',6':6,7-(5-hydroxy-2-methyl)chromone (Ia).

In addition to hamaudol 3'-acetate, the roots yielded a substance with the composition $C_{29}H_{50}O$, mp 139–140° C, which was identified by its IR spectrum and a mixed melting point test as β-sitosterol.

The mass spectrometric determination of the molecular weight was performed by P. I. Zakharov and the microanalyses were carried out by E. A. Nikonova.

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