SAXALIN – A NEW FUROCOUMARIN FROM THE ROOTS OF *Angelica saxatilis*

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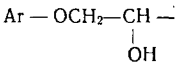
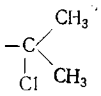
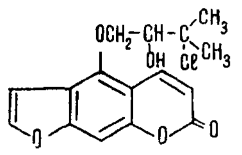
We have previously [1] reported the isolation from the roots of Angelica saxatilis Turcz. of five lactones of the furocoumarin group – isoimperatorin, imperatorin, bergapten, xanthotoxol, and (+)-oxypeucedanin hydrate. On eluting the chromatographic column with diethyl ether, we obtained a neutral substance $C_{16}H_{15}O_5Cl$ with mp 159-161°C (Kofler) (from methanol), R_f 0.59, soluble in chloroform and methanol, and insoluble in water. UV spectrum: λ_{max} 223, 250, 266, 309 nm (log ε 4.40, 4.24, 4.21, 4.15). IR spectrum, ν_{max}, cm^{-1}: 3480 (OH group), 3140, 825 (C–H bond of a furan ring), 1711 (C=O of an α-pyrone ring), 1625, 1578 (aromatic nucleus).

The features of its spectra and its chemical properties show that the compound obtained is a new chlorine-containing furocoumarin, which we have called saxalin.

The NMR spectrum of the lactone (in deuteroacetone, on a HA-100D, 100 MHz instrument, from TMS taken as 0), there were doublets at 8.33 and 6.21 ppm, J=10 Hz, due to the H_4 and H_3 protons of the coumarin nucleus, doublets at 7.83 and 7.23 ppm, J=2.5 Hz, due to the $H_{5'}$ and $H_{4'}$ protons of the furan ring, and a singlet at 7.15 ppm due to the H_8 proton. These facts confirm that saxalin is a 5-substituted furocoumarin. Signals at 4.92, 4.50, and 4.10 ppm (total intensity 4 H) relate to the protons in a  $Ar-OCH_2-\underset{OH}{\underset{|}{CH}}-$ grouping (a quartet at 4.92 ppm with an intensity of two proton units corresponds to one of the protons of the methylene group and the hydroxyl, a quartet at 4.50 ppm to the other proton of the methylene group, and a signal at 4.10 ppm to the methine proton). Two singlets at 1.64 and 1.68 ppm (with an intensity of 3 H each) are due to the protons of geminal methyl groups on a tertiary carbon atom  $-\underset{Cl}{\underset{|}{C}}\!\!<^{CH_3}_{CH_3}$. The downfield shift of the signals from the gem-dimethyl grouping as compared with the spectra of known compounds – oxypeucedanin (δ 1.32 and 1.39) and oxypeucedanin hydrate (δ 1.28 and 1.32) [2] – is due to the electron-accepting influence of the chlorine.

The presence of an alcoholic hydroxy group in the molecule of saxalin was confirmed by the preparation of an acetate with mp 147-150°C (Kofler) (from methanol) in the IR spectrum of which the OH absorption band at 3480 cm^{-1} had disappeared. In the NMR spectrum of saxalin acetate (in $CHCl_3$), a singlet at 2.14 ppm (3H) is due to the protons of one acetyl group, and the unchanged position of the signals of the gem-dimethyl grouping (1.64 and 1.68 ppm) as compared with the spectrum of the starting material shows the acetylation of a hydroxy group on the second carbon atom of the side chain.

On the basis of the facts presented, saxalin has the most probable structure of 5-(3-chloro-2-hydroxy-3-methylbutoxy)furo-3′,2′:6,7-coumarin.



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A spot with the same R_f value was detected feebly in the initial extract, which gives grounds for considering saxalin to be a native compound.

Chlorine-containing coumarins were previously unknown; saxalin is the first compound of this type.

M. E. Perel'son took part in the interpretation of the NMR spectrum.

LITERATURE CITED

1. L. G. Avramenko, G. K. Nikonov, and M. G. Pimenov, Khim. Prirodn. Soedin., 5, 436 (1969).
2. M. E. Perel'son, Tr. VILR [Proc. of the All-Union Institute of Medicinal Plants], 15, 60 (1969).