FARNESIFEROL C FROM THE ROOTS OF Ferula szovitsiana

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UDC 547.587.53

Continuing a study of the coumarin composition of representatives of the family Umbelliferae, we have investigated Ferula szowitsiana. The hydroxycoumarin umbelliferone has previously been isolated from the seeds of this plant [1]. A preliminary investigation of the roots (PC) showed that they contained other coumarins besides the hydroxycoumarin found in the seeds.

To isolate the coumarins, the air-dry comminuted roots collected in the environs of Nakhichevan' (Nakhichevan' ASSR) were extracted with methanol, the solvent was evaporated, the residue was diluted with water, and the coumarins were extracted with ether. The total coumarins, consisting of five components, were chromatographed on a column of silica gel. They were eluted successively with petroleum ether, benzene, and ether. Two coumarins were isolated in the individual state: (I), $C_{24}H_{30}O_4$, mp 85-86°C; and (II), $C_9H_6O_3$, mp 232-233°C.

Substances (I) and (II) were identified by their physicochemical constants, elementary compositions, PC analysis, and UV, IR, and NMR spectra as farnesiferol C and umbelliferone, respectively [2, 3].

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Original article submitted January 15, 1976.

THE CRYSTAL STRUCTURE OF GUMMOSIN

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UDC 547.582

Because of contradictory information on the configuration of substituents at C_9 of farnesiferol A and its analogs [1-6], we have studied gummosin (the epimer of farnesiferol A at C_3) by x-ray structural analysis.

The crystals investigated belong to the space group $P2_12_12_1$ of the rhombic system; a = 21.364 (1), b = 13.4754 (6), c = 7.1429 (3) Å, $\alpha = \beta = \gamma = 90°$, V = 2056.3 (3) Å^3, M = 382.5, d_{meas} = 1.23; d_{calc} = 1.24 g/cm^3, Z = 4. The noncentrosymmetrical structure was interpreted by the direct method using the Rentgen-75 program [7] based on 1644 reflections with $|F|^2 > 3\sigma$ measured on a four-circle automatic diffractometer (copper radiation, graphite monochromator) and it was refined in the block-diagonal anisotropic approximation to R = 0.089 by the program of Chekhlov et al. [8].

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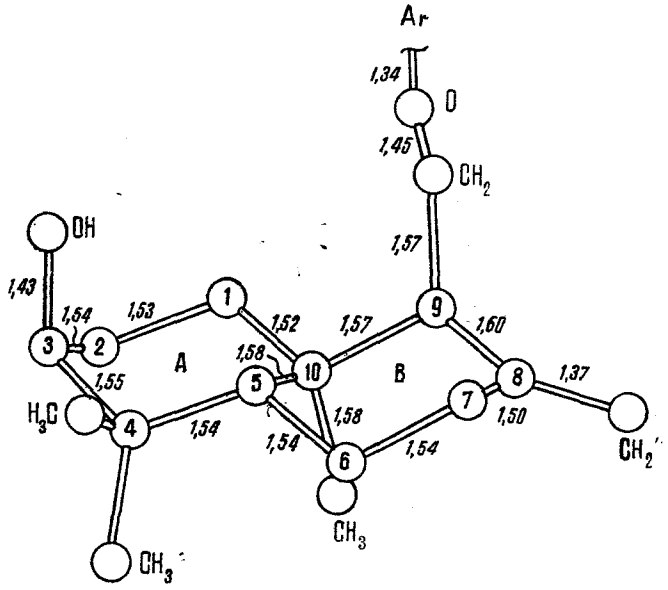
The x-ray structural investigations objectively established the stereochemistry of the molecule (Fig. 1) and showed that the $-CH_2-O-Ar$ group at C_9 is present in the β-axial orientation. This is in harmony with the conclusions of Perel'son et al. [5, 6]. The coumarin nucleus is planar. Rings A and B have the chair configuration ($^3C_{10}$ and $^5C_8$, respectively) and are trans-linked. The substituent OH at C_3 and the CH_3 group at C_{10} are axial. The molecules in the crystal are united by a weak $O-H\cdots O=$ bond 2.99 Å long. The lengths of the other bonds and valence angles are normal.



B. L. Tarnopol'skii (OIKhF Akad. Nauk SSSR) took part in the interpretation of the structure by the direct method.

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