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THE STRUCTURE OF AKIFERININ

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Continuing a study of esters of Ferula akitschkensis B. Fedtsch ex K.-Pol., by separating the mother liquor after the isolation of akichenin, akiferin, ferutin, and ferutinin on a column of silica gel, we have isolated a substance with the composition $C_{24}H_{34}O_6$ (I) (M^+ 418), with mp 176-177°C, $[\alpha]_D^{20}$ +73.1° (c 0.82; $CHCl_3$). The substance is readily soluble in alcohols, chloroform, and acetone, sparingly soluble in ether and petroleum ether, and insoluble in water. The UV spectrum of (I) showed maxima at 262 and 295 nm (log ε 4.09, 3.76) which are characteristic for a 3,4-dihydroxybenzoyl residue.

The IR spectrum of the substance has absorption bands of an aromatic nucleus (1520, 1590, 1610 cm^{-1}), of an ester group (1235, 1705 cm^{-1}), and of a hydroxy group (3550 cm^{-1}). A comparison with the literature of the physicochemical constants and spectral characteristics that we found for the substance showed that it is new. We have called it akiferinin.

The PMR spectrum of (I) (JNM-4-100 MHz, $CDCl_3$, 0 – HMDS) (Fig. 1) contains the signals of secondary (0.75, 0.85 ppm, d, J=7.5 Hz, 3H each) and tertiary (1.22, 1.44 ppm, s 3H each) methyl groups, of methoxy groups in an aromatic nucleus (3.82, 3.84 ppm, s, 3H each), and of a hemiacyl proton (5.35 ppm, sextet, $J_1=J_2=$ 10 Hz; $J_3=2.5$ Hz). Signals were also observed at 6.85 ppm (d, J=9.5 Hz, 1H), 7.45 ppm (q, $J_1=9.5$; $J_2=2.5$ Hz, 1H), and 7.35 ppm (d, J=2.5 Hz, 1H) due to the protons of a 3,4-dimethoxybenzoic (veratric) acid residue.

When akiferinin was subjected to alkaline hydrolysis, the acidic fraction of the hydrolyzate yielded an acid with the composition $C_9H_{10}O_4$ (II) with mp 192-193°C which was identified as veratric acid [3], and the neutral fraction yielded a sesquiterpene alcohol with the composition $C_{15}H_{26}O_3$ (III) with mp 113-114°C identical with the diol obtained in the hydrolysis of tenuferin, tenuferinin, and tenuferidin [4].

Thus, akiferinin is an ester of the sesquiterpene diol (III) with veratric acid, and structure (I) is proposed for it.

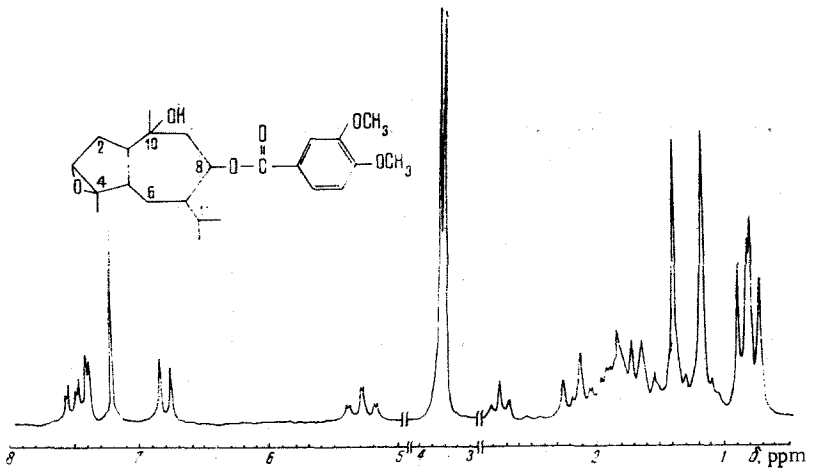

Fig. 1. NMR spectrum of akiferinin.

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SAPOGENINS OF THE ROOTS OF Gypsophila bicolor

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We have previously [1-3] reported the presence of triterpene saponins in the roots of Gypsophila bicolor Freyn et Sint (family Caryophyllaceae) growing in Azerbaidzhan. In the second paper we give the results of an investigation of the sapogenins of this plant.

The saponins were extracted from the roots of G. bicolor with water-saturated n-butanol with heating. The butanolic extract was filtered hot and was then cooled to room temperature. The saponins that deposited were separated off, washed repeatedly with n-butanol, and dried. This gave the combined saponins in the form of a nonhygroscopic white powder with a faint greyish tinge.

In a study of its quantitative composition by chromatography on paper and in a thin layer of silica gel in various systems, the presence of four individual triterpene glycosides – A, B, C, and D – was detected.

The combined saponins so obtained were hydrolyzed with 10% H_2SO_4 solution at 90°C for 10-12 h. The precipitate was filtered off and washed with water, and the saponins were extracted with chloroform. Chromatographic analyses in the toluene–ethanol (10:2), benzene–ethanol (10:1), and chloroform–methanol–ethyl acetate (2:1:3) systems showed that the extract obtained included the two saponins A and B.

The sapogenins were separated on a column of Al_2O_3 (activity grade II) (elution with toluene).

After recrystallization from ethanol, the composition of sapogenin A was $C_{30}H_{46}O_4$, mp 269-271°C, $[\alpha]_D^{20}$ + 90° (c 1.0; ethanol). The acetate of sapogenin A had mp 176-178°C.

From its physicochemical constants and spectral characteristics in the IR region and on the basis of comparative chromatographic investigations with authentic samples, it was established that sapogenin A was gypsogenin.

Sapogenin B, with the composition $C_{30}H_{46}O_4$, had mp 330-335°C (from methanol), $[\alpha]_D^{20}$ +40° (c 0.5; ethanol). The IR spectrum showed absorption bands in the region of 3400 cm^{-1} (OH) and 1735-1740 cm^{-1} (C=O of a γ-lactone and of an aldehyde).

The results that we obtained agree with those given in the literature [4, 5] for gypsogenin lactone, and show that sapogenin B was gypsogenin lactone.

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