

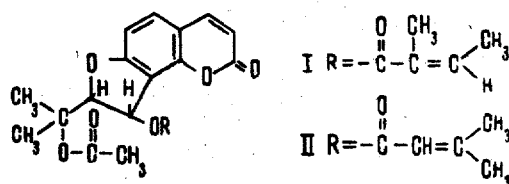
BRIEF COMMUNICATIONS

THE STRUCTURE OF PEUCENIDIN

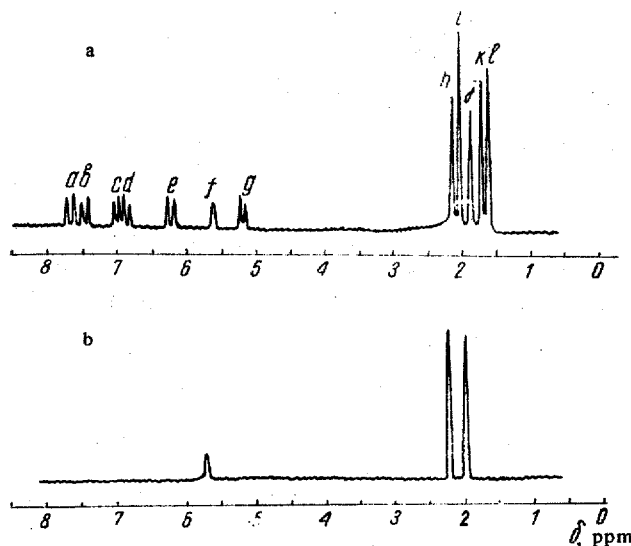
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One of us has previously reported [1] the isolation and the determination of the structure of a new dihydrofuro-coumarin, peucenidin, for which structure I was proposed on the basis of its chemical properties. The results of a study of the NMR spectrum of peucenidin have confirmed the main conclusions of the chemical experiments and have shown that this substance contains a residue not of tiglic acid but of senecioic (β , β -dimethylacrylic) acid.



The NMR spectrum of peucenidin (figure) was taken in deuteriochloroform at a frequency of 100 MHz. The chemical shifts of the signals were determined in relation to tetramethylsilane as internal standard taken as 0.



NMR spectra. a) Peucenidin; b) senecioic (β , β -dimethylacrylic) acid. In CDCl_3 .

The presence of the doublets a, b, c, d, and e, due to protons in positions 4, 5, 6, and 3 of the coumarin nucleus, confirms the structure of peucenidin as a 7,8-disubstituted coumarin [2]. The doublets c and g must be assigned, respectively, to protons 4' and 5' of the dihydrofuran ring. The singlets i, k, and l with an intensity of three proton units each relate to the methyl of the acetyl group and to two nonequivalent methyl groups attached to a quaternary carbon atom. Peaks f, h, and j are due to the second acid residue. The signals h and j of methyl groups on a double bond are somewhat broadened because of allyl interaction with the vinyl proton, whose signal f also has complex splitting with a spin-spin interaction constant of 1.2 Hz.

The facts given show that the second acid residue in peucenidin is of β , β -dimethylacrylic acid. The results of a comparison of the NMR spectrum of peucenidin with the spectrum of β , β -dimethylacrylic acid (see figure) confirm this conclusion. Thus, peucenidin has the structure II.

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FLAVONOIDS OF RHEUM TATARICUM. V

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Continuing an investigation of the seeds and leaves of Rheum tataricum L. fil. [1], we have isolated the total flavonoids. Their separation from a concentrated methanolic extract was achieved on fine Kapron powder. After repeated chromatography of the individual fractions, we obtained quercetin with mp 313°-314° C, isoquercitrin with mp 240°-241° C, meratin with mp 182°-183° C, and rutin with mp 190°-190.5° C.

To determine the flavonoids in the raw material quantitatively [2], they were chromatographed in the ethyl acetate-formic acid-water (10:2:3) system. The spots were cut out and eluted with 1% aqueous ammonium chloride [3]. The optical densities of the eluates were measured with a SF-4A spectrophotometer at a wavelength of 415 mμ [4, 5]. The concentrations were calculated from calibration curves constructed for the pure flavonoids.

The seeds of Rheum tataricum contain 0.05% of quercetin, 0.066% of isoquercitrin, 0.044% of meratin, and 0.072% of rutin, and the leaves contain 0.15%, 0.30%, 0.22%, and 1.45%, respectively.

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FLAVONOLS OF HIBISCUS AND A HYBRID OF HIBISCUS WITH THE COTTON PLANT

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Continuing our investigation [1] of the flavonoids of the hybrid Hibiscus 3209 and 2332, we have isolated a second flavonol.

The products of acid and enzymatic hydrolysis and of peroxide oxidation were compared chromatographically on paper. It was found that the aglycone is identical with quercetin. The sugar was revealed by mixtures of diphenylamine