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DEHYDRATION IN TERPENOID COUMARINS

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Continuing a study of the reaction of trifluoroacetic acid (TFA) with terpenoid coumarins of the iresane series [1], we have investigated coumarins of the samarcandin series containing methyl and hydroxy groups in the C-2 position: samarcandin (I) [2], samarcandone (II) [2], nevskin (III) [3, 4], nevskone (IV) [3, 4], ferucrin (V) [5], and ferucrinone (VI).

A study of the end products showed that during the reaction compounds with double bonds in the rings were formed. Thus, the reaction of TFA with (IV) [3, 4] led to conferone (VII) [6]. This transformation is well shown in the ¹H NMR spectrum, in which the signals of the protons of the initial product have disappeared and the signals of the protons of (VII) have appeared (Fig. 1). Conferone is also formed by the reaction of TFA with (II) [2], which confirms the equatorial orientation of the substituent at C-1 in compounds (II) and (IV) [3-7].

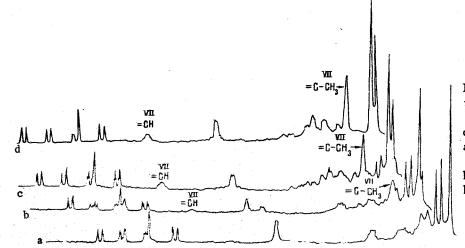
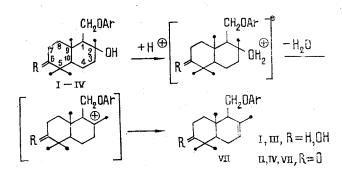


Fig. 1. ¹H NMR spectrum of samarcandone (a, b, c) and conferone (d) in trifluoroacetic acid. Spectrum (c) was recorded three hours after the sample had been dissolved.

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The formation of a product with an endocyclic double bond takes place via a carbocation formed after the splitting out of a molecule of water from the initial compound protonated on the OH group at C-2.



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FLAVONOID AGLYCONES OF THE ROOTS OF Rhaponticum carthamoides

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Continuing a study of the phenolic compounds from plants of the family Compositae [1, 2], by means of paper chromatography we have established that the roots and rhizomes of *Rhaponticum carthamoides* (Willd.) Iljin, collected in October, 1977, on the experimental plantation of the Central Botanical Garden of the Academy of Sciences of the Belorussian SSR, contained not less than ten flavonoid aglycones.

To establish the nature of the aglycones the combined glycosides (an amorphous powder from the ethyl acetate fraction, the yield of which was 0.5% calculated on the air-dry raw material) was hydrolyzed with a 10% solution of hydrochloric acid with heating on the boiling water bath for 5 h. The aglycones were extracted with diethyl ether. The ethereal extracts were washed with distilled water to neutrality and dried with anhydrous sodium sulfate. After elimination of the organic solvent, the dry residue was again dissolved in ether. The powder obtained after the elimination of the ether again was dissolved in 80% ethanol.

The combined aglycones were separated into their individual components by two-dimensional chromatography on Filtrak FN-16 paper in the following solvent systems: 1) chloroform acetic acid (3:2), and 2) 40% acetic acid [3, 4]. Preparative paper chromatography and elution with ethanol yielded the individual components (I-IV), which were identified by comparison

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