UDC 547.597+547.918

STEROID SAPOGENINS OF ASPARAGUS PERSICUS

I. T. Tairov

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, pp. 326-327, 1969

This paper gives the results of a study of the saponins of Asparagus persicus Baker (Perisan asparagus, growing in Azerbaidzhan) which was begun previously [1].

In an investigation of the sapogenins by the method of Wall [2] and Rothrock [3], as modified by O. S. Madaeva [4, 5], we isolated a sapogenin with the composition $C_{27}H_{44}O_3$, mp 200-201° C, $[\alpha]_D^{20}$ −76.25 (c 1.8; chloroform) and obtained the acetyl derivative of the genin with mp 137-138° C $[\alpha]_D^{23}$ −70.5 (c 2.5; chloroform). From its constants behavior on paper chromatography and thin-layer chromatography and its absorption in the IR spectrum, this sapogenin has been identified as sarsasapogenin (IR spectra: 852, 900, 921, 987 cm^{-1}).

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9 January 1969

Narimanov Azerbaidzhan State Medical Institute

UDC 547.913:547.917

THE STRUCTURE OF HELIANTHOSIDE B

P. L. Cheban and V. Ya. Chirva

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, p. 327, 1969

We have isolated helianthoside B from a mixture of saponins of sunflower petals by chromatography on silica gel in the butan-1-ol-acetic acid-water (4 : 1 : 5) system. As in the case of helianthosides A and C [1, 2], the aglycone of the glycoside obtained is echinocystic acid and it contains the monosaccharides glucose, arabinose, xylose, and rhamnose in a ratio of 1 : 1 : 1 : 2.

By gas-liquid chromatography and chromatography in a thin layer of silica gel in the presence of reference samples, an acid hydrolysate of fully methylated helianthoside B was shown to contain, 2, 3, 4, 6-tetra-O-methyl-D-glucose, 2, 3, 4-tri-O-methyl-D-xylose, 3, 4-di-O-methyl-L-arabinose, and 2, 3-di-O-methyl-L-rhamnose. The periodate oxidation of the saponin confirmed the results of methylation.

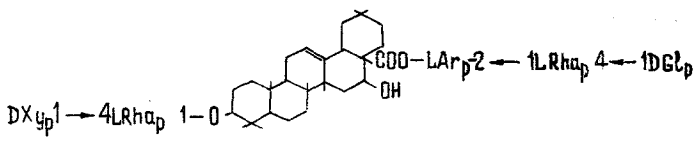
The aluminum hydride cleavage of the methylated helianthoside B gave an oligosaccharide identical with that obtained similarly from methylated helianthoside C [2].

Thus, the carbohydrate chain attached to the carboxyl group of echinocystic acid has the structure

$$DGl_p\,1 \rightarrow 4\,LR\mathit{ha}_p\,1 \rightarrow 2LAr_p\,1 \rightarrow .$$

The acid hydrolysis of the methylated glycoside obtained by the aluminum hydride cleavage gave 2, 3, 4-tri-O-methyl-D-xylose and 2, 3-di-O-methyl-L-rhamnose, which were identified by known methods.

The final structure of helianthoside B is as follows:



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4 February 1969

Institute of Chemistry AS Moldavian SSR

UDC 615.711.5:615.45

THE ISOLATION OF OLITORISIDE FROM THE SEEDS OF CORCHORUS OLITORIUS

M. T. Turakhodzhaev, M.-R. I. Shamsutdinov, and T. T. Shakirov

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, pp. 327-328, 1969

The cardiac glycoside olitoriside, which is used in medicine [1], is isolated from the seeds of Corchorus olitorius [2]. We have developed a new method for the isolation of olitoriside. The seeds of Corchorus olitorius, after cominution and defatting by extraction with gasoline, were extracted by steeping in 96% ethanol. The extract was concentrated and was treated twice with acetone to precipitate the sugars.

The acetonic solution was concentrated and was treated with ether to eliminate the residues of fatty and resinous substances. The extractive substances insoluble in ether were precipitated in the form of a viscous dark mass which was separated off and washed with ether.

The viscous mass was dissolved in water and common salt was added, and, to eliminate the monosides and inert substances, the solution was washed with chloroform several times. Then the olitoriside was exhaustively extracted from the aqueous solution with small portions of a mixture of chloroform and isopropanol (1 : 1 by volume). The chloroform-isopropanol extract was concentrated to small volume and filtered through a layer of alumina, and then an equal volume of water was added and the mixture was left in a thermostatted vessel at 36-37° C. As the isopropanol evaporated off, crystals deposited; these were separated off and washed with water.

The crystals obtained were dissolved in boiling acetone, and on cooling the olitoriside deposited.

The crystals of olitoriside that had deposited were recrystallized from aqueous ethanol (1 : 1 by volume). The yield of olitoriside was 0.13% of the weight of the raw material.

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25 February 1969

Institute of the Chemistry of Plant Substances AS UzSSR

UDC 615.43+615.32

APIGENIN AND ITS GLYCOSIDES FROM GRATIOLA OFFICINALIS

V. I. Litvinenko, L. I. Borodin, and N. V. Kurinnaya

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, pp. 328-329, 1969

By one- and two dimensional paper chromatography we have found in the herb drug hedgehyssop, collected in the phase of mass flowering, about ten substances of flavonoid nature. The flavonoids were separated on Kapron, and the individual compounds GF-1, GF-2, GF-3, and GF-4 were isolated.

Apigenin (GF-1), $C_{15}H_{10}O_5$, has mp 346-348° C (aqueous methanol), R_f in 15% acetic acid (system 1) 0.04, and in butan-1-ol-acetic acid-water (4 : 1 : 2) (system 2) 0.92; λ_{max} in ethanol 336, 270 mμ (log ε 4.40, 4.38); λ_{max}