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SPIROSTANOL GLYCOSIDES OF Yucca gloriosa

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The total steroid glycosides, in amounts of 5% [1] and 10%, respetively, have been obtained from the air-dry leaves and flowers of mound lily yucca <u>Yucca gloriosa L.</u> (family <u>Agavaceae</u>) growing in Tbilisi in an experimental field for medicinal plants of the Institute of Pharmacochemistry of the Georgian SSR Academy of Sciences. As a result of their repeated chromatography on column of silica gel (KSK 50/90) with elution by the solvent system chloroformmethanol-water (65:35:8), individual glycosides were obtained: from the flowers - glycoside 1 with a yield of 0.15% (on the weight of the air-dry raw material, mp 302-304°C, and glycoside 2 in an amount of 0.12% (on the weight of the air-dry raw material), mp 258-262°C. Two individual glycosides have also been obtained from the total glycosides of the leaves, and from their physicochemical constants and mobilities in TLC they proved to be identical with glycosides 1 and 2 from the flowers. Glycoside 1 from the leaves melted at 298-299°C (the yield amounting to 0.13%), while glycoside 2 had mp 262-264°C (isolated in an amount of 0.11%).

All the glycosides isolated gave a positive reaction with the Sannie reagent [2] and a negative reaction with the Ehrlich reagent [3]. Their IR spectra contained absorption bands in the 850-980 cm<sup>-1</sup> region that are characteristic for a spiroketal grouping, which showed their spirostanol nature.

As a result of the acid hydrolysis of the glycosides (0.1 g each) with 2 N HCl (5 ml,  $100^{\circ}$ C, 5 h) an aglycon was isolated in all cases the physicochemical properties of which corresponded to tigogenin [4]. In the carbohydrate moieties of the glycoside under investigation (after evaporation of the filtrate) glucose, galactose, and rhamnose were identified by the TLC method in the solvent system butanol-methanol-water (5:3:1) (revealing agent: o-toluidine salicylate). The hydrolysates were reduced with sodium tetrahydroborate at room temperature (12 h) and were then acetylated in a mixture of acetic anhydride (2 ml) and pyridine (2 ml) at room temperature (12 h). By the GLC method, in comparison with authentic samples, the acetates of rhamnitol, duicitol, and sorbitol were identified in a ratio of 1:1:3 for glycoside 1 and 1:1:4 for glycoside 2.

The PMR and <sup>13</sup>C NMR spectra of the glycosides under investigation were taken on WM-250 and AM-300 instruments (Bruker) with working frequencies of 250 MHz for protons and 63 MHz for carbon, and 300 and 75 MHz for solutions of the glycosides in pyridine at various temperatures. The results obtained showed the identiyf of glycosides 1 and 2 with yuccaloesides B adn C which we have isolated previously from the leaves of aloe yucca <u>Yucca aloifolia L. [5]</u>. Thus, glycoside 1 was the 3-O-{[O- $\alpha$ -L-rhamnopyranosyl-(1+4)-O- $\beta$ -D-glucopyranosyl-(1+3)]-[O- $\beta$ -D-glucopyranosyl(1+2)]-O- $\beta$ -D-glucopyranosyl-(1+4)- $\beta$ -D-galactopyranoside} of (25R)-5 $\alpha$ -spirostan-

I. C. Kutateladze Institute of Pharmacochemistry Academy of Sciences of the Georgian SSR, Tbilisi. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 295-296, March-April, 1991. Original article submitted May 28, 1990. 3 $\beta$ -ol, and glycoside 2 was the 3-O-{[O- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 4)-O- $\beta$ -D-glucopyranosyl-(1 $\rightarrow$ 3)]-[O- $\beta$ -D-glucopyranosyl-(1 $\rightarrow$ 3)-O- $\beta$ -D-glucopyranosyl-(1 $\rightarrow$ 2)]-O- $\beta$ -D-glucopyranosyl-(1 $\rightarrow$ 4)- $\beta$ -D-galac-topyranoside} of (25R)-5 $\alpha$ -spirostan-3 $\beta$ -ol.

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MONITORING THE PURITY OF ANTIGEPOLIN WITH THE AID

OF CHROMATOGRAPHY IN A THIN LAYER OF SORBENT

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Antigepolin (the ethobromide of polymethacryloyllupinine) has been recommended by the N. I. Pirogov 2nd Moscow State Medical Institute and the laboratory of the Pharmacological Institute of Bioorganic Chemistry Academy of Sciences of the Uzbek SSR as a drug for eliminating the side effect of heparin (1, 2].

Since the starting substance for the synthesis of antigepolin, methacryloyllupinine, is not an inert substance for the organism, the necessity arises for checking the purity of the drug. For the best separation of the drug and determining sensitivity we used plates with a fixed layer of type LS 5/40  $\mu$  silica gel (Czechoslovakia) and the following set of solvent systems: benzene-dioxane (9:1.5), benzene-methanol (5:2 and 5:1), acetone-dioxane-water (0.5:8:0.5), ethanol-chloroform (1:1, 2:1, and 1:2), and methanol-chloroform (2:1 and 1:2). The best separation was achieved in the methanol-chloroform (2:1) system.

Chromatography was conducted by the ascending method in a cylindrical chamber with dimensions of  $80 \times 200$  mm. At the start line  $100 \ \mu g$  (0.01 ml) of a solution fo the drug in ethanol was deposited with a micropipette. On the same line, at a distance of 2 cm, was deposited 1  $\mu g$  (0.05 ml) of a marker solution of methacryloyllupinine in ethanol. The plate was visualized with Dragendorff reagent. The conditions of chromatography that had been selected enabled antigepolin to be distinguished from methacryloyllupinine. The minimum detectable amount of the marker was 1  $\mu g$  and that of the drug 1.1  $\mu g$ .

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