

STEROID SAPONINS.

V. AGAVASAPONIN C FROM THE LEAVES OF *Agava americana*

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In a methanolic extract of the leaves of *Agava americana* by thin-layer chromatography in various systems of solvents we have detected nine glycosides which we have called in the order of the increase in their polarity agavasaponins A, B, C, D, E, F, G, H, and I.

By chromatography on a column of silica gel in the chloroform-methanol-water (65:25:10) system, from the combined saponins we have isolated pure agavasaponin C with mp 275°C,  $[\alpha]_D^{20}$  -55° (c 1.08; dimethylformamide).

The aglycone obtained by the acid hydrolysis of agavasaponin C (5% H<sub>2</sub>SO<sub>4</sub>, 16 h, +110°C) was identified by mass spectrometry (M<sup>+</sup> 430), by its melting point and  $[\alpha]_D$  value and IR spectrum as hecogenin.

In a filtrate of the hydrolyzate by paper chromatography in the butanol-pyridine-benzene-water (5:1:3:3) system and by gas-liquid chromatography (GLC) of the aldonitriles of the sugars we identified galactose (Gal) and glucose (Glc) in a ratio of 1:2. Partial hydrolysis (2% H<sub>2</sub>SO<sub>4</sub>, 6 h, 100°C) gave two progenins.

After the acid hydrolysis of these substances, the sugar of the less polar progenin was found to be Gal while the other contained Gal and Glc in a ratio of 1:1.

To prove the type of bond, the agavasaponin C was methylated by the method of Kuhn and Purdie [1, 2]. Methanolysis of the permethylated saponin [72% HClO<sub>4</sub> in absolute methanol (1:10) at 100°C, 5 h] yielded 2,3,4,6-tetra-O-methyl-D-glucose, identified by GLC in the presence of markers.

The mass spectrometry of the deuteromethylsugars [3], and also high-resolution NMR spectrometry showed the presence in them of 2,3,6-tri-O-methyl-D-glucose and 2,3,6-tri-O-methyl-D-galactose. The results that we obtained agree well with those given in the literature [4] (PMR spectrum of the sugars taken in CCl<sub>4</sub>); the chemical shifts of the CH<sub>3</sub> groups of the 2,3,6-methylated sugars given in parts per million - ppm - relative to HMDS as internal standard were 3.15, 3.44, and 3.49.

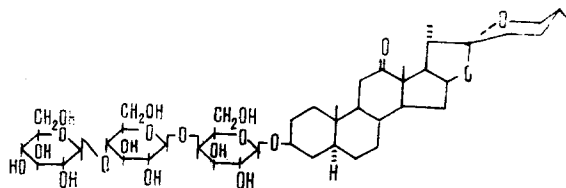
The configurations of the glycosidic centers of the sugars in the saponin were shown by the NMR spectra of the methylated progenins and of the initial glycoside.

The results obtained for the methylated monoside, bioside, and triside show β-glycosidic linkages in the carbohydrate moiety of the saponin (the spin-spin coupling constant for the anomeric proton was 8 Hz and the chemical shifts were 4.2, 4.3, and 4.8 ppm, respectively [5]; in the case of an α-glycosidic link, the value of the SSCC would be 4.2 Hz or less [6]).

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Summing the results obtained, the above formula can be put forward for the structure of agavasaponin C.

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