PHENOLIC GLYCOSIDES OF THE ROOTS OF Salix pentandroides

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We have previously reported the isolation from the leaves of Salix pentandroides A Skv. of the glycosides hyperoside, quereimeritrin, and salicin [1]. Continuing a study of the phenolic compounds, from the bark of this species we have isolated two phenolic glycosides.

The finely ground fresh bark (1.5 kg) from the branches of plants two to five years old collected in June in the Teberda State Reserve on the banks of the R. Amanaus were exhaustively extracted with ethanol—methanol (1:1). The alcoholic extracts were concentrated in vacuum at $50-60^{\circ}$ C to small volume. The residue was dissolved in water and was purified with chloroform, and tannin substances and other polyphenolic compounds were precipitated with a 10% aqueous solution of basic lead acetate. After the removal of the precipitate, the phenolic glycosides were extracted from the aqueous residue with ethyl acetate, and the extract was evaporated to small volume and placed in the refrigerator. After two days, white plates had crystallized out of a substance with the composition $C_{15}H_{20}O_8$, mp 179-181°C (water-saturated ethyl acetate), $[\alpha]_D^{20}-39^{\circ}$ (c 1.48; water; l 0.2 dm). UV spectrum: $\lambda_{\rm max}$ 267 nm (log ϵ 3.0); $\lambda_{\rm max}^{\rm NaOH}$ 269 nm (log ϵ 2.96); melting point of the acetate 130-132°C (aqueous methanol).

Saligenin (2-hydroxybenzyl alcohol), D-glucose, and acetic acid were found in the products of acid hydrolysis of the compound isolated. On alkaline hydrolysis in 1% caustic potash solution for 20 min [2], equimolecular amounts of the glycoside salicin (saligenin 2-O- β -D-glucopyranoside) and acetic acid were obtained. The glycoside under investigation was not cleaved by the enzyme emulsin.

The position of attachment of the acetyl group was determined by exhaustive methylation with methyl iodide in dimethylformamide in the presence of silver oxide followed by acid hydrolysis [3]. 2,3,4-Trimethylglucose was found in the hydrolysis products. Consequently, in the glucose molecule the hydroxyl in position 6 was acetylated, and the glycoside under consideration is saligenin 2-O- β -D-(6-acetyl)glucopyranoside (fragilin) [4].

The ethyl acetate filtrate after the removal of the fragilin was deposited on a column of cellulose (h 60 cm, diameter 7 cm) and eluted with butan-1-ol-xylene-water (4:6:8), 50-ml fractions being collected [5]. No phenolic glycosides were detected in the first fractions; from the 23rd to the 33rd the glycoside fragilin was eluted, and after this a mixture of fragilin and salicin. Fractions 42-56 gave salicin (saligenin 2-O- β -D-glucopyranoside) with mp 200-201°C (water-saturated ethyl acetate); $[\alpha]_D^{20}$ -63° (c 1.24; water; l 0.2 dm). UV spectrum: $\lambda_{\rm max}$ 269 nm (log ϵ 2.12); melting point of the acetate 132-134°C (aqueous methanol).

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