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GLYCOSIDES OF PATRINIA INTERMEDIA

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We have previously obtained from the roots of Patrinia intermedia Roem et Schult. a triterpene glycoside, patrinoside C [1]. In addition, accompanying glycosides were found which gave no color reaction with antimony trichloride.

From a mixture containing triterpene glycosides we isolated one of them, interoside B, in the form of the acetyl derivative. For this purpose a methanolic extract of the roots of the plant was freed from reserve sugars by percolation through moist alumina, and acetylated with acetic anhydride in pyridine, and the total acetates so obtained were separated by chromatography on silica gel. In contrast to the acetates of the triterpene glycosides, the acetate of the new glycoside had a sharp melting point: 179°–180° C, $[\alpha]_D^{20}$ −76 ±8° (c 5; chloroform). Found, %: C 51.22; H 5.61. Its yield was about 1% of the methanolic extract of the roots. The cryoscopic method of determining the molecular weight gave a value of the order of 2200–2250.

In contrast to the acetate of patrinoside C, the IR spectrum of the acetate of the glycoside obtained has, in addition to the frequencies of an acetyl group at 1760 (C=O) and 1250 (C–O–C) cm^{-1}, absorption bands at 1615, 1490, 960, and 800 cm^{-1}, on the basis of which it may be assumed that the aglycone of the glycoside has an aromatic nature [2]. The hydrolysis of the acetate with mineral acid gave a crystalline aglycone with mp 78°–80° C $[\alpha]_D^{20}$ −68.5 ±3° (c 3.94; chloroform). Found: C 63.97; H 6.17. D-Glucose, mp of the osazone 205°–207° C, was identified in the filtrate by paper chromatography. The lower melting points and the percentages of carbon and hydrogen also indicated that the aglycone was not a triterpene compound.

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CONVERSION OF ERYSIMOSIDE INTO k-STROPHANTHIN-β

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The axial hydroxyl in the digitoxose residue of erysimoside (at C-3) acetylates more slowly than all the secondary equatorial hydroxyls of the glucose residue. By making use of this property, we have synthesized k-strophanthin-β in the following way.

A solution of 2 g of erysimoside in 20 ml of pyridine was treated with 10 ml of acetic anhydride and left at 22° C for 50 min. Then the reaction mixture was diluted with 100 ml of chloroform and was neutralized in the cold with 2 N sodium carbonate solution. The chloroform layer was separated off, washed with water (4 × 15 ml), and evaporated in vacuum. The residue was transferred to a 50-ml flask, 15 g of silver oxide and 30 ml of methyl iodide were added, a glas-covered stirring rod was inserted, the flask was sealed, and methylation was carried out for 1 hr at 56° C with a magnetic stirrer switched on.

At the end of the reaction the solution was filtered, the precipitate was washed with chloroform, and the filtrate was evaporated. The residue was dissolved in 30 ml of methanol, mixed with 4 ml of methanol saturated with gaseous ammonia, and left at room temperature for 17 hr. The solution was evaporated in vacuum and the resulting mixture of cardenolides was chromatographed on 80 g of alumina with chloroform– ethanol as eluant. The eluates containing pure k-strophanthin-β were combined and evaporated and the glycoside was crystallized from water.