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POLYSACCHARIDES OF *Ungernia*.

XI. A STUDY OF THE GLUCOFRUCTANS OF *Ungernia vvedenskyi*

M. Kh. Malikova, D. A. Rakhimov, and E. S. Kondratenko

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In a preceding paper [1] we have reported the structure of ungeromannan-V isolated from the bulbs of *Ungernia vvedenskyi* Khamidkh. In the present communication we give informa-mation on the determination of the structure of a glucofructan from the same plant.

The comminuted air-dry raw material was first treated with ethanol (96% and 82%) in order to eliminate low-molecular-weight compounds and mono- and oligosaccharides, and it was then extracted with water and the ungeromannan-V [2] was precipitated by the addition of ethanol, the concentration of the latter being brought to 70%. The mother ethanolic solution was concentrated. The precipitate of ungeromannan-V and protein impurities was eliminated by the addition of a solution of neutral lead acetate, and the excess of the latter was eliminated with a saturated solution of sodium sulfate. Then the solution was de-ionized with KU-2 cation-exchange resin and evaporated to a syrup, and the latter was triturated with ethanol. This gave the total glucofructans with a yield of 1% on the air-dry raw material.

For purification, the combined glucofructans were dissolved in water and dialyzed. The dialyzed solution was evaporated and precipitated with ethanol. The yield of product was 20% on the initial combined glucofructans. In a hydrolysate, PC showed mainly fructose and glucose.

The glucofructan was a white hygroscopic amorphous powder readily soluble in water and in dimethyl sulfoxide, $[\alpha]_D^{20} - 65°$ (c 1; water). Gel filtration on Sephadex G-50 showed it to be homogeneous with a molecular weight of 2000, which corresponds to a degree of polymerization of 12.

The IR spectrum of the glucofructan has absorption bands at (cm^{-1}) 3600-3200 (hydroxy group), 940 (vibrations of a furanose ring), 880 (vibrations of a β-glycosidic bond), and 820 (vibrations of a pyranose ring). These absorption bands are characteristic for inulin [3].

Periodate oxidation and Smith degradation were carried out as described by Tomoda and Saton [4]. The consumption of periodate and the yield of formic acid per 1 mole of hexose unit were 0.99 and 0.07 mmoles, respectively. In a hydrolysate of the oxidation products, PC in the butan-1-ol—pyridine—water (6 : 4 : 3) system showed the presence of glycerol, which was identified by GLC in the form of the polyol acetate [5].

The isolation of glycerol is evidence in favor of a 2 → 1 bond between the monosaccharide units.

Methylation of the glucofructan was performed by Hakomori's method [6]. A permethylate was obtained which, after formolysis and hydrolysis, was studied by TLC in the methyl ethyl ketone—1% aqueous ammonia (30 : 4) system. 3,4,6-Tri-O-Me-D-fructose (the main product), 1,3,4,6-tetra-O-Me-D-fructose, and 2,3,4,6-tetra-O-Me-D-glucose were identified in the hydrolysate. The results of methylation were in agreement with those of periodate oxidation.

The partial hydrolysis of the glucofructan was carried out in 0.1% acetic acid at 100°C for 20 min. PC showed the presence of sucrose, in addition to fructose and glucose.

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Thus, the glucofructan isolated is a low-molecular-weight polysaccharide with a degree of polymerization of 12 consisting of glucopyranose and fructofuranose residues linked in the inulin manner (2 → 1 bonds). This is the first time that a glucofructan has been isolated from the genus *Ungernia*.

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POLYSACCHARIDES OF *Eremurus*.

XVII. THE STRUCTURE OF A GLUCOFRUCTAN FROM *Eremurus lactiflorus*

A. Dzhumamuratova, D. A. Rakhimov, and E. S. Kondratenko

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We have previously [1, 2] reported the structure of a glucomannan isolated from the tuberous roots of *Eremurus lactiflorus*. In the present paper we give the results of a study of a glucofructan from this plant.

The comminuted air-dry raw material was first treated with ethanol and was extracted with water at room temperature. The mother solution after the precipitation of a glucomannan from the aqueous extract [3] was concentrated and, to eliminate proteins and clarify it, it was treated with a solution of neutral lead acetate, the excess of which was precipitated with a solution of Na_2SO_4. To eliminate the low-molecular-weight compounds the solution was dialyzed in countercurrent. The dialyzed solution was evaporated to a syrup and was treated with acetone, which converted it into a powder. The yield of water-soluble carbohydrate was 2.48% (on the air-dry raw material). It consisted of a hygroscopic white powder readily soluble in water. In the products of complete acid hydrolysis (0.5 N H_2SO_4, 90°C, 0.5 h), PC showed the presence of mainly fructose, with traces of glucose. Consequently, the carbohydrate was a glucofructan (GF). The GF was separated on a column of Sephadex G-25 and G-50, which led to a homogeneous GF with a molecular weight of 1200 and a degree of polymerization of 7. It possessed no reducing capacity, and the ratio of fructose and glucose according to the ^{13}C NMR spectrum was 6 : 1, respectively. IR spectrum, λ_{max}^{KBr}, cm^{-1}: 3400 (OH); 880 (β-glycosidic bond), 820 (hexapyranose ring), and 940 (furanose ring) [4].

To determine the type of bond in the GF, it was methylated by Hakomori's method [5]. After formolysis and hydrolysis of the permethylate of the GF, by TLC on Silufol (methyl ethyl ketone—1% ammonia (30 : 4) system) and by the GLC of the trifluoroacetates of the corresponding polyols [6], using comparison with known samples, 3,4,6-tri-O-Me-fructose, 1,3,4,6-tetra-O-Me-fructose, and 2,3,4,6-tetra-OMe-glucose were identified.

The results of methylation were confirmed by those of periodate oxidation. The GF was oxidized by the method of Khodzhaeva and Ismailov [7], consuming 1 mole of $NaIO_4$ per monosaccharide unit. Glycerol was found by PC in the products of Smith degradation. The results of methylation and periodate oxidation permit the assumption for the glucofructan of a structure with 2 → 1 bonds between the hexose residues.

The ^{13}C NMR spectrum also showed the presence of 2 → 1 bonds in the GF. The spectrum contained peaks with chemical shifts corresponding to residues of β-2 → 1-bound fructofuranoside units (ppm):

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