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FRACTIONATION OF THE PECTIN SUBSTANCES OF *Eremurus regelii*. XXII.

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We have previously studied the structure of a galacturonan isolated by partial acid hydrolysis of the pectin substances (PSs) from the leaves of *Eremurus regelii* Vved. In the present communication we give the result of an investigation of homogeneous fractions isolated from the PSs.

The pectin substances from the leaves of *E. regelii* were polydisperse in gel chromatography on Sephadex G-100. To obtain homogeneous fractions of the PSs, alkaline saponification was carried out with caustic soda (0.15 g/100 ml) at room temperature for 18 h. After neutralization with 18% of HCl solution, a gel-like precipitate deposited which was washed with 80% and 96% methanols and formed fraction (I) (yield 65%). The mother solution was dialyzed, concentrated, and precipitated with methanol (1:2) giving 6% of fraction (II). The characteristics of the fractions obtained by alkaline saponification are given below:

Fraction	$[\alpha]_D^{20}$, deg	Monosaccharide composition, moles						
		Rham	*Ara*	*Xyl*	*Man*	*Gal*	*Glc*	*GalUA*
I	+249 (*c* 0,25; H_2O)	14	3,7	1.3	Tr.	4.6	Tr.	+
II	+200 (*c* 0.5; H_2O)	7,5	5.8	3.7	Tr.	19.6	4.9	+

To determine the monosaccharide composition, samples were hydrolyzed with 2 N H_2SO_4 in tubes at 100°C for 48 h and were subjected to PC (1-butanol–pyridine–water (6:4:3) system; revealing agent: aniline hydrogen phthalate). The GLC of samples in the form of the acetates of the corresponding aldonitriles [1] were recorded on a Tsvet-101 instrument with a flame-ionization detector under the following conditions: steel column (200 × 0.3 cm), 5% of Silicone XE-60 on Chromaton NAW (200-250 mesh), thermostat temperature 210°C, evaporator temperature 270°C, carrier gas helium at the rate of 75 ml/min.

To obtain homogeneous fractions of pectic acid, a 1% solution of fraction (I) was treated with 1 N NaOH (4 ml) and then a 2 M solution of CH_3COONa (6 ml) was added dropwise and the mixture was left at +4°C for 18 h. The precipitate that had deposited (fraction A) was centrifuged off and was washed with 80% and 96% methanols (the yield of fraction A was 14%). The supernatant was treated with 2 M CH_3COONa solution (3 ml) and the resulting precipitate (fraction B) was separated off and washed with 80% and 96% methanols (the yield of fraction B was 3.75%). The mother solution was dialyzed, concentrated, and precipitated with methanol (1:2), giving a 40.5% yield of fraction C. The homogeneity of the fractions obtained (A, B, and C) was confirmed by gel chromatography on Sephadex G-100. Their characteristics are as follows:

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*Deceased.

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Fraction	$[\alpha]_D^{20}$, deg (c 0,5; H_2O)	Uronic anhydride, % [2]	Monosaccharides composition, moles			
			Rham	*Ara*	*Xyl*	*Gal*
A	+150	73	20	3,6	2,3	3,7
B	+190	65,8	14,8	1	1	1,5
C	+165	69,9	19,49	4,1	4,1	4,2

Periodate oxidation was carried out in a neutral medium. The consumption of sodium perchlorate was 0.2 mole per mole of anhydrohexose unit for fraction A, 0.205 for fraction B, and 0.33 for fraction C. On Smith degradation followed by PC (1-butanol—pyridine—water (6:4:3) system; revealing agent: periodate—$KMnO_4$—benzidine), rhamnose, arabinose, galactose, xylose, glycerol, and erythritol, and also galacturonic acid were detected.

The comparatively low consumption of periodate and the presence of unoxidized monosaccharides indicates a branched structure for the pectin.

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3-DEOXYNON-2-ULOSONIC ACID IN THE CAPSULAR POLYSACCHARIDE FROM *Klebsiella ozaenae*

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The enterobacterium *Klebsiella ozaenae* is the causative agent of a disease of the upper respiratory tracts, ozaena. A polysaccharide has been isolated from the capsule of the microorganism by mechanical degradation and has been purified by ion-exchange chromatography.

The acetylated products of the optimized methanolysis [1] of the polysaccharide were separated by GLC on the liquid phases OV-225, QF-1, and NPGS. A study of the composition of the mixture of methyl glycoside acetates so obtained by the GLC-MS method (QF-1) showed the presence of two minor components: mannose (R_a = 4) and glucosamine (R_a = 5); and of two major components: glucose (R_a^* = 57) and methyl 3-deoxynon-2-ulosonate (I) (R_a^* = 34). The latter, (I) was identified on the basis of the results of a study of mass spectra in comparison with the spectrum of the deuterium analog (II) and results on the breakdown under electron impact from the octoanalogs [2].

The mass spectrum of (I) in the characteristic region (above m/z 150) was due to the decomposition of molecular ions (M^+) of three types (1, 2, and 3) into five large fragments: P, A, E, B, and D. This fragmentation was accompanied by the loss of ketene and acetic acid molecules and of acetyl and acetoxy radicals, which gave a complex mass-spectral pattern.

The splitting out of the substituents of the pyranose ring S_1 and S_2 from the branched C_2 and C_6 atoms of the M^+ ions of types 2 and 1 explains the high content of oxonium ions of the P, A, and E series. In the spectrum of the deuterium analog (II) the ions of series D, P, and A are responsible for peaks 3 m.u., and the ions of series B and E 6 m.u., higher than in the spectrum of (I). Among the fragments mentioned, only ions B and D arise as the result of the breakdown of the pyranose ring of (I) and form an inconsiderable fraction of the ion

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*Sum of the R_a values of the anomers and forms.

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