

Thus, the analysis of the peptides isolated from A-128-OP acids after the action of N-BS that has been performed gives grounds for assuming that the A-128-OP acid has the following amino-acid sequence:  $\Delta$  Asp  $\rightarrow$  Ser  $\rightarrow$   $\Delta$ - Abu  $\rightarrow$  Thr  $\rightarrow$  Ala  $\rightarrow$  Gly  $\rightarrow$  t-3-Hyp  $\rightarrow$   $\Delta$ - Trp  $\rightarrow$   $\beta$  MeTrp  $\rightarrow$   $\beta$  HyLe  $\rightarrow$  c-3-Hyp-OH, which is somewhat refined as compared with that given previously [3]. Details of the experiment on the cleavage of the A-128-OP acid will be published later.

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#### POLYPHENOLS OF *Myricaria alopecuroides*

#### III. HYDROLYZABLE TANNING SUBSTANCES

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UDC 547.477.982

Continuing an investigation of the polyphenols of *Myricaria alopecuroides* Schrenk., family Tamaricaceae Lindl. [1], by acid hydrolysis of the combined ellagotannins (5% HCl, 100°C) followed by extraction with ether and ethyl acetate and chromatography of the ethyl acetate extract on Sephadex LH-20 we have obtained two cleavage acids.

Acid 1,  $C_{14}H_{10}O_{10} \cdot 2H_2O$  (the results of elementary analysis correspond to the calculated figures), mp (decomp.) 277°C,  $R_f$  0.65 in BAW (40:12.5:29; system 1) and 0.65 in 15%  $CH_3COOH$  (FN-3; system 2). It forms a deep blue coloration with 1% ferrous ammonium alum (FAA). The reaction for bound ellagic acid is negative [2]. With concentrated  $H_2SO_4$  it gives a positive reaction for the formation of xanthone [3]. The IR spectrum shows the absorption bands of hydroxy groups, a carbonyl group, a benzene ring, and an ether bond (3300, 3000-2600, 1650, 1535, 1610, 1035  $cm^{-1}$ ).

The methylation of acid 1 with an ethereal solution of diazomethane gave a heptamethyl derivative, mp 113°C,  $C_{21}H_{24}O_{10}$ , mol. wt. 436 (mass spectrometrically). The IR spectrum has the absorption bands of methoxy groups, an ester bond, a benzene ring, and an ether bond (2930, 1730, 1600, 1100  $cm^{-1}$ ). The NMR spectrum has the signals of three aromatic protons in the form of a superposed singlet and doublet at  $\delta$  7.22 ppm and a doublet at 6.71 ppm ( $J = 2$  Hz), and also the signals of the protons of seven methoxy groups at 3.84 ppm (12H) and 3.83 ppm (9H).

The hydrolysis of the heptamethyl derivative with 2 N NaOH gave a pentamethyl ether, mp 247°C. Titration of the pentamethyl ether showed the presence of two carboxy groups.

On the basis of results obtained, the acid 1 was identified as dehydrodigallic acid [4].

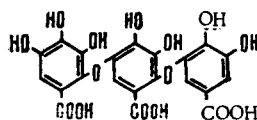
Acid 2,  $C_{21}H_{10}O_5 \cdot 2H_2O$  (the result of elementary analysis corresponded to the calculated figures), mp (decomp.) 281-283°C,  $R_f$  0.48 in system 1 and 0.60 in system 2. With a 1% solution of FAA it formed a deep blue coloration. The reaction for bound ellagic acid was negative [2] and with concentrated  $H_2SO_4$  it gave a positive reaction for the formation of xanthone [3]. IR spectrum: 3400, 3000-2600, 1690, 1610, 1050  $cm^{-1}$ .

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Methylation with diazomethane gave a decamethyl derivative with mp 117°C,  $C_{31}H_{33}O_{15}$ , mol. wt. 646 (mass spectrometrically). IR spectrum: 2930, 1730, 1600, 1100  $cm^{-1}$ . NMR spectrum (ppm): signals of four aromatic protons in the form of a doublet with a singlet at  $\delta$  7.23, singlet at 6.80, doublet at 6.72 ( $J = 2$  Hz), and the signals of the protons of ten methoxy groups at 4.00 (3H), 3.86 (12H), 3.70 (12H), and 3.52 (3H). On hydrolysis with 2 N NaOH, a heptamethyl ether was obtained with mp 277°C. Titration of the heptamethyl ether showed the presence of three carboxy groups.

On the basis of the results obtained, acid 2 is a dehydrotrigallic acid not previously described in the literature and has the structure



The NMR spectra were taken in IOKÉ of the Academy of Sciences of the Kazakh SSSR and the mass spectra in the Lenin Tashkent State University.

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#### NEW ESTERS OF UGAMDIOL FROM THE ROOTS OF *Ferula involucrata*

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UDC 547.913.5+547.587

By column chromatography on silica gel we have isolated from a methanolic extract of the roots of *Ferula involucrata* Eug. Kor. two new esters which we have called involucrin (I) and involucrinin (II).

**Substance (I)**,  $C_{27}H_{38}O_8$ , mp 154-155°C,  $[\alpha]_D^{24} -33.4^\circ$  (c 0.89; chloroform),  $M^+ 490$ , dissolves readily in ether, chloroform, and ethanol and is insoluble in petroleum ether and water. Its UV spectrum has  $\lambda_{max}$  270 nm ( $\log \epsilon$  4.01), which shows the presence of a benzene nucleus in the molecule. In the IR spectrum there are absorption bands at 1750 and 1720  $cm^{-1}$  (ester carbonyls) and at 1600 and 1510  $cm^{-1}$  (aromatic nucleus). On saponification with caustic soda, involucrin is cleaved forming 3,4,5-trimethoxybenzoic acid and ugamdol [1], which were identified by mixed melting points, IR spectroscopy, and chromatography in a thin layer of cellulose.

The facts given show that involucrin is a diester of ugamdol. This is confirmed by the NMR spectrum of involucrin [signals at 5.50 and 4.90 ppm (2H) corresponding to two geminal protons], and also by a comparison of the positions of the signals of the geminal and hemihydroxyl protons in the NMR spectra of the diester, the monoester, and ugamdol itself.

To establish the positions of the acid residues, we performed mild hydrolysis (5%  $Na_2CO_3$  at room temperature). A substance was obtained with mp 124-125°C which was identified by a mixed melting point as ugaferin, i.e., a monoester of ugamdol with 3,4,5-trimethoxybenzoic acid at the  $C_6$  position, and acetic acid was detected chromatographically in the hydrolyzate.

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from *Khimiya Prirodnikh Soedinenii*, No. 1, p. 132, January-February, 1976. Original article submitted April 15, 1975.

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