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ELLAGIC ACID DERIVATIVES FROM *Euphorbia ferganensis*

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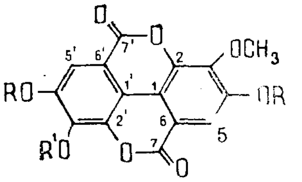
Scopoletin and ethyl gallate have been isolated previously from the roots of *Euphorbia ferganensis* B. Fedtsch. [1]. Continuing the investigation of this plant, from the ethyl extract fraction of an ethanolic extract of the roots we have isolated two more compounds of phenolic character by chromatography on a column of silica gel in the benzene—ethanol (7:3) solvent system.

Compound (I) has the composition $C_{15}H_8O_8$, mp 303-305°C, ν_{max}^{KBr} (cm^{-1}); 3450-3030 (OH), 1725-1707 (C=O), 1615, 1590, 1510 (C=C bond). Its UV spectrum had maxima at 254, 358* (inflection), and 372 nm (log ε 3.58, 3.91, 3.96) and was similar to the spectrum of 3,3'-di-O-methylellagic acid [2]. The PMR spectrum of (I) (C_5D_5N, δ, ppm. 0 — HMDS) showed a three-proton singlet at 4.08 ppm ($Ar\text{-}OCH_3$), two one-proton singlets at 7.88 and 7.94 ppm (H-5 and H-5'), and a broadened signal at 7.52-8.06 ppm apparently due to the protons of phenolic hydroxy groups. In actual fact, when the spectrum was taken with the addition of trifluoroacetic acid (TFA) the signs of these protons, exchanging with the OH protons of the TFA, shifted downfield and appeared in the form of a broadened singlet at 8.4 ppm.

In the mass spectrum of compound (I) in addition to the 100% peak of the molecular ions with m/z 316 there were the peaks of ions with m/z 301 $(M - CH_3)^+$, 287 $(M - CHO)^+$, 273 $(M - CH_3 - CO)^+$, 259, 231, and others.

The acetylation of substance I gave a triacetyl derivative (Ia) with mp 251-252°C (2.20 ppm, 9H, s) while methylation with diazomethane led to the trimethyl derivative, (Ib), $C_{18}H_{14}O_8$, mp 343-344°C (decomp.), M^+ 358; ν_{max}^{KBr} 1741, 1616, 1577, 1497 cm^{-1}. In its physical and spectral properties, substance (Ib) was identical with the tetramethyl derivative of ellagic acid [3]. Consequently, the compound (I) was a monomethyl derivative of ellagic acid.



I. R=R'=H
Ia. R=R'=$COCH_3$
Ib R=R'=CH_3
II. R=H, R'=CH_3

As mentioned above, the UV spectrum of (I) was close to that of 3,3'-di-O-methylellagic acid and differed considerably from that of 4,4'-di-O-methylellagic acid. Furthermore, the signal of the carbon atoms of the $Ar\text{-}OCH_3$ group appeared in the ^{13}C NMR spectra of (I) at 60.8 ppm, which is characteristic for a sterically hindered methoxy group in a benzene ring [4]. This corresponds to the C-3 and C-3' positions of ellagic acid, but in biew of the symmetry of the molecule the above-mentioned positions are equivalent. Thus, compound (I) was the 3-O-methyl derivative of ellagic acid. This substance has not been described in the literature.

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The assignment of the signals of the ^{13}C carbon nuclei in the ^{13}C NMR spectrum was made on the basis of a comparison of the CSs of the carbon atoms of (I) with the analogous values for 3,3'-di-O-methylellagic acid [3] and from a comparative study of the ^{13}C NMR spectra obtained under the conditions of complete and partial decoupling from protons. The results of the assignment are given below:

C	δ, ppm	C	δ, ppm
1	111.7	1′	111.9
2	140.0	2′	136.9
3	139.8	3′	139.8
4	151.9	4′	148.2
5	111.2	5′	110.2
6	112.2	6′	107.0
7	158.6	7′	158.7
		OCH_3	60.8

Compound (II) has the composition $C_{16}H_{10}O_8$, M^+ 330, mp 328-330°C, λ_{max} 250, 359, 370 nm (log ε 3.53, 3.86, 3.92), and on methylation with diazomethane formed substances (Ib). On the basis of a study of its UV and mass spectra and a comparison of its physical constants with those given in the literature, this compound was identified as 3,3'-di-O-methylellagic acid [3, 5]. A similar substance has been detected previously in *Euphorbia formosana* [5, 6], while 3,3',4-tri-O-methylellagic acid has been isolated from *Euphorbia tirucalli* [7].

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STRUCTURE OF THE MAIN FLAVONOID GLYCOSIDE FROM THE FLOWERS OF *Filipendula ulmaria*

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The main flavonoid glycoside has previously [1-3] been isolated from the flowers of *Filipendula ulmaria*, and contradictory opinions have been expressed about its structure. The first discoverers [1] suggested that the compound, which they called spiraeoside, was quercetin 4'- or 3'-glucoside. Hörhammer et al., [2] shosed that spiraeoside was quercetin 4'-glucoside. Other workers [3] identified the main flavonoid glucoside that they had isolated as quercetin 3'-O-β-D-glucopyranoside.

We have studied the chemical composition of the flowers of *Filipendula ulmaria* collected in the Priozersk region of Leningrad province, in the Okulovka region of Novogorod province, in the Putstoshka region of Pskov province, and in the Vologod province.

An ethanolic extract of the flowers was dissolved in hot water and the solution was treated successively with chloroform, ethyl ether, ethyl acetate, and butanol. Salicylic and gallic acids and quercetin, which are known for the flowers of this plant [4-6] were isolated from the first two fractions and identified by their melting points and IR spectra.

The ethyl acetate fraction, by precipitation with chloroform followed by crystallization from ethanol-chloroform. yielded yellow acicular crystals with mp 212°C, $[\alpha]_n^{22}$ −64.3° (c 0.014; ethanol). UV spectrum $\lambda_{max}^{ethanol}$ 255, 266 sh., 364 nm (log ε 4.14. 4.14, 4.15), ν_{max}, cm^{-1}; 3340 v.s., 3270 v.s (sh), 1657 v.s., 1620 s (sh), 1598 v.s., 1560 m, 1510 v.s, 1465, 1435 m

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