

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 $\text{C}_{16}\text{H}_{10}\text{O}_8$, M^+ 330, mp 328–330°C, λ_{max} 250, 359, 370 nm ($\log \epsilon$ 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].

LITERATURE CITED

1. M. M. Tadzhibaev and K. L. Lutfullin, *Khim. Prirod. Soedin.*, 571 (1985).
2. L. Jurd, *J. Am. Chem. Soc.*, **81**, 4610 (1959).
3. M. A. M. Nawwar, J. Buddrus, and H. Bauer, *Phytochemistry*, **21**, 1577 (1982).
4. A. Arnone, L. Camarda, L. Merlini, and G. Nasini, *J. Chem. Soc., Perkin Trans. I*, 2118 (1977).
5. J. Jurd, *J. Am. Chem. Soc.*, **81**, 4606 (1959).
6. B. P. Moore, *Aust. J. Chem.*, **17**, 901 (1964).
7. A. Chatterjee, M. Chakrabarty, and A. K. Ghosh, *Indian J. Chem.*, **15B**, 564 (1977).

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

T. V. Bukreeva and B. A. Shukhobodskii

UDC 547.972

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] showed 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, $[\alpha]_D^{22}$ –64.3° (c 0.014; ethanol). UV spectrum $\lambda_{\text{max}}^{\text{ethanol}}$ 255, 266 sh., 364 nm ($\log \epsilon$ 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

V. L. Komarov Botanical Institute of the USSR Academy of Sciences, Leningrad. Translated from *Khimiya Prirodnykh Soedinenii*, No. 5, pp. 755–756, September–October, 1987. Original article submitted February 28, 1987; revision submitted May 28, 1987.

(sh), 1377, 1350 s, 1315 v.s, 1260 s, 1237 s, 1205 v.s, 1175 v.s, 1143 m, 1107 v.s, (sh), 1095 v.s, 1082 s (sh), 1052 s, 1016 m, 1003 m (sh), 999 m, 942 v.s, 897 v.s, 873 w, 843 m, 830 w, 814 w, 801 m, 728 m, 714 m (in paraffin oil).

^{13}C NMR spectrum, δ , (DMSO): C-2 147.0 s^a, C-3 136.6 s, C-4 176.2 s, C-5 160.9 s, C-6 98.6 d, C-7 164.2 s, C-8 93.7 d, C-9 156.4 s, C-10 103.3 s, C-1' 125.4 s, C-2' 115.4 d, C-3' 146.6 s^a, C-4' 146.1 s^a, C-5' 116.1 d, C-6' 119.8 d, C-1'' 101.6 d, C-2'' 73.5 d, C-3'' 76.2 d^a, C-4'' 70.0 d, C-5'' 77.4 d^a, C-6'' 61.4 t (^a - assignment made ambiguously).

The features of the ^{13}C NMR spectrum that we obtained of the glycoside isolated and literature figures for spiraeoside from plants of the genus *Allium* [7] are very close. The values of the signals of the carbon atoms of quercetin 3'-glucoside - C-2 146.3, C-3 136.2, C-1' 122.1, C-2' 116.7, C-3' 145.0, C-4' 149.1, C-5' 116.1, C-6' 123.3 - calculated in the light of the glycosylation effect with the use of corrections [7] found in a comparison of the chemical shifts of the signals of the carbon atoms of luteolin and luteolin 3'-glucoside differ substantially from the values of the signals of the carbon atoms of the glycoside that we had isolated. The chemical shifts of the signals of the carbohydrate moiety of the glycoside were close to those for methyl O- β -D-glucopyranoside [8]. On the basis of the ^{13}C NMR spectrum, the main flavonoid glycoside from the leaves of European meadowsweet has been identified as quercetin 4'-O- β -D-glucopyranoside, spiraeoside [2].

LITERATURE CITED

1. P. Casparis. Pharm. Acta Helv., 21, 341 (1946); Chem. Abstr., 42, 6990c (1948).
2. L. Hörhammer and R. Hänsel, Arch. Pharm., 287, 36 (1954).
3. V. L. Shelyuto, V. I. Glyzin, V. N. Filipchik, L. P. Smirnova, and A. I. Ban'kovskii, Khim. Priir. Soedin., 113 (1977).
4. A. Schneegans and I. G. Gerock, Jahresber. Pharm., 27, 164 (1892).
5. B. Pasich, Acta Pol. Pharm., 10, No. 1, 67 (1953).
6. E. Steinegger and P. Casparis, Pharm. Acta Helv., 21, 154-173 (1945), Chem. Abstr., 42, 4578g (1948).
7. K. R. Markham, B. Ternai, R. Stanley, H. Geiger, and T. J. Mabry, Tetrahedron, 34, 1389 (1978).
8. S. Seo, Y. Tomito, K. Tori, and Y. Toshimura, J. Am. Chem. Soc., 100, No. 11, 3331 (1978).

FLAVONOIDS OF THE SEEDS OF *Genista aetnensis*

N. A. Artamonova, G. K. Nikonov,
K. Nusipbekova, and V. A. Nosul'chak

UDC 547.972

Studying the seeds of *Genista aetnensis* DC (Aetna woadwaxen), family Fabaceae, growing in the village of the Kara-Kala, Turkmen SSR, by paper chromatography we have detected not less than 10 substances of flavonoid nature. Information on the presence of flavonoids in the epigeal part of Aetna woadwaxen is given in [1, 2].

In the present communication we give the results of an investigation of an ethanolic extract from the seeds of this plant. The concentrated methanolic extract was chromatographed on a column of silica gel L using as eluent benzene containing from 0 to 50% of ethyl acetate. Four substances were isolated which were identified from their physicochemical properties, IR, UV, and PMR spectra [3], and the products of alkaline fusion as known flavonoids: kaempferol 4'-methyl ether, isorhamnetin, and tamarixetin.

This is the first time that these compounds have been isolated from the seeds of Aetna woadwaxen.

Institute of Chemical Sciences of the Kazakhstan SSR Academy of Sciences, Alma-Ata.
Translated from Khimiya Prirodnykh Soedinenii, No. 5, pp. 756-757, September-October, 1987.
Original article submitted March 30, 1987.