UDC 547.972

FLAVONOIDS OF ARTEMISIA TRANSILIENSIS

T. K. Chumbalov and O. V. Fadeeva

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, p. 439, 1969

Continuing a study of the flavonoid composition of Artemisia transiliensis Poljak [1], we have isolated four flavonoid aglycones from an ethereal extract. Flavones and flavonols in which the OH group at $C_{(3)}$ is substituted are stable to oxidation in alkaline solutions and can be obtained on acidification [2]. Using this method, the ethereal extract was treated successively with saturated $NaHCO_3$ solution and with a 10% solution of Na_2CO_3. The alkaline solutions were acidified and extracted with ether. From the bicarbonate extract by preparative paper chromatography using benzene–acetic acid–water (125 : 72 : 3) as solvent, we isolated quercetin 3-methyl ether with mp 259° C and a substance, II, with mp 255–256° C (from 70% ethanol), λ_{max} 355, 266, 255 mμ. In the cyanidin reaction, substance II gave a crimson coloration showing its flavonol nature. Spectral studies in the UV region with ionizing and complex-forming reagents showed the presence in the aglycone II of free OH groups at $C_{(7)}$, $C_{(3')}$ and $C_{(4')}$. In an alkaline melt of the substance paper chromatography showed the presence of catechol and protocatechuic acid. IR spectroscopy showed that the aglycone II contains a methoxy group (2960, 2850 cm^{-1}) located at $C_{(3)}$. Demethylation of the substance led to the formation of 7, 8, 3', 4'-tetrahydroxyflavonol with mp 310–312° C [3]. Methylation of the aglcyone with diazomethane yielded a pentamethoxyflavone with mp 149–150° C.

Thus, on the basis of the chemical and spectral studies substance II has been characterized as 7, 8, 3', 4'-tetrahydroxy-3-methoxyflavone. It proved to be new, and we have called it transilitin. From the sodium carbonate solution we isolated genkwanin with mp 283° C and acacetin with mp 260–261° C.

REFERENCES

1. T. K. Chumbalov and O. V. Fadeeva, KhPS [Chemistry of Natural Compounds], 3, 281, 1967.
2. L. H. Briggs and R. H. Locker, J. Chem. Soc., 3137, 1951.
3. F. E. King and W. Bottomley, J. Chem. Soc., 1399, 1954.

26 March 1969

Kirov Kazakh State University

UDC 547.972

A FLAVONOID DIGLYCOSIDE FROM ACONITUM ORIENTALE

I. I. Samokish, A. L. Shinkarenko, and V. A. Makarov

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, pp. 439–440, 1969

In the leaves of Aconitum orientale Mill., collected in the region of the Teberdina reserve, we have found by paper chromatography no less than four flavonoid compounds.

By chromatography on a column of Kapron with desorption by 25–35% ethanol we isolated glycoside I with mp 187–189° C, $[\alpha]^{20}_D$ −245° (c 0.5; methanol). λ_{max} 344, 165 mμ (methanol); CH_3COONa λ_{max} 344, 165 mμ; $CH_3COONa + H_3BO_3$ λ_{max} 344, 265 mμ; $AlCl_3$ λ_{max} 405, 350, 277 mμ; $AlCl_3 + HCl$ λ_{max} 342, 275 mμ; CH_3ONa λ_{max} 390, 265 mμ.

The aglycone (yield 50%) with mp 273–275° C (acetyl derivative with mp 180–182° C) was identified as kaempferol on the basis of its IR and UV spectra, chromatographic behavior, and absence of a depression of a mixture with an authentic sample. The carbohydrate component of glycoside I is L-rhamnose (melting point of the osazone 178–180° C).

On the stepwise acid hydrolysis, a 7-monoside with mp 232–233° C, $[\alpha]^{20}_D$ − 166° (c 0.4; methanol) was obtained as an intermediate product; alkaline hydrolysis [1] led to the formation of a 3-monoside with mp 173–175° C, $[\alpha]^{20}_D$ −165° (c 0.2; methanol). By physicochemical analysis, the conversion products of the diglycoside were characterized as kaempferol 7-O-α-L-rhamnofuranoside and kaempferol 3-O-α-L-rhamnofuranoside.

The IR and UV spectra and an analysis of the conversion products, together with the absence of a depression of the melting point with an authentic sample, enabled glycoside I to be identified as kaempferol 3-O-α-L-rhamnofuranoside-7-O-α-L-rhamnofuranoside, which has been reported previously under the names "kaempferitrin" and "lespedin" [2, 3].

28 March 1969

Pyatigorsk Pharmaceutical Institute

UDC 547.972

LUTEOLIN FROM THE LEAVES OF DIGITALIS CILIATA

E. P. Kemertelidze

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, p. 440, 1969

In the preparation of cardiac glycosides from the leaves of Digitalis ciliata Trautv. we isolated a yellow crystalline substance [1] giving all the reactions for flavonoids. In the cyanidin test, an octanol-extractable orange-red pigment was formed, which shows the aglycone nature of the compound [2]. When the substance was subjected to paper chromatography in the butanol–acetic acid–water (4 : 1 : 5) system it gave a single spot, while in each of the systems ethyl acetate–formic acid–water (10 : 2 : 3) and benzene–ethyl acetate–acetic acid (74.5 : 23.5 : 2) systems it gave two spots. On them the main component appeared at the level of an authentic sample of luteolin, and a small spot in the region of apigenin.

To separate the combined flavonoids into the individual compounds we chromatographed them on a polyamide sorbent. Pure luteolin was isolated by washing the column with a mixture of chloroform and ethanol (1 : 1). After its recrystallization from dilute ethanol, long yellow acicular crystals, $C_{15}H_{10}O_6$, with mp 330–332° C were obtained. It gave no depression with standard luteolin. The acetate of the substance melted at 226–231° C. The IR and UV spectra of the flavonoid and its acetate coincided completely with literature data for luteolin and its acetate [3, 4].

REFERENCES

1. E. P. Kemertelidze, KhPS [Chemistry of Natural Compounds], 1, 379, 1965.
2. E. T. Brrejant, J. Am. Pharm. Assoc., 39, 481, 1956.
3. I. P. Kovalev and E. V. Titov, Infrared Absorption Spectra of Some Groups of Natural Compounds (Atlas of Spectra) [in Russian], Khar'kov, 1966.
4. V. I. Litvinenko and N. P. Maksyutina, KhPS [Chemistry of Natural Compounds], 1, 420, 1965.

23 April 1969

Kutateladze Institute of Pharmacochemistry AS Georgian SSR

UDC 547.972

LUTEOLIN 7-GLUCOSIDE FROM CAMPANULA LACTIFLORA

S. F. Dzhumyrko, E. T. Oganesyan, and A. L. Shinkarenko

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 5, pp. 440–441, 1969

The chromatography of an ethanolic-aqueous extract from the leaves of Campanula lactiflora M. B. in the BAW (4 : 1 : 5) system showed the presence of three substances of a flavonoid nature. One of them, with the composition $C_{20}H_{20}O_{11}$, mp 256–258° C (from ethanol), $[\alpha]_D^{20}$ −58° (c 0.528; methanol–pyridine (3 : 2)), mol. wt. 259, is a flavone glycoside as was shown by the results of color reactions.

UV spectrum: λ_{max} 352, 255 mμ; $\lambda_{max}^{AlCl_3}$ 400, 275 mμ; $\lambda_{max}^{CH_3COONa}$ 355, 258; $\lambda_{max}^{CH_3COONa+H_3BO_3}$ 380, 258 mμ; $\lambda_{max}^{CH_3ONa}$ 407, 265 mμ; $\lambda_{max}^{AlCl_3+HCl}$ 390, 275 mμ. The elementary composition found corresponds to that calculated.