# THE COUMARINS OF ARTEMISIA PERSICA

#### Sh. Z. Kasymov and G. P. Sidyakin

#### Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, p. 319, 1969

The plant was collected in the flowering phase (June 1968) in the Kurama range of the Tashkent region. By treatment of the epigeal part with chloroform, 6.1% of extractive substances were obtained and when these were treated with 5% sodium bicarbonate solution a coumarin derivative  $C_{10}H_8O_4$  was isolated. With diazomethane, the substance formed a methyl ether. The coumarin was identified as scopoletin.

On continuing extraction of the plant with methanol, we obtained an additional 13.1% of extractive substances. When the concentrated extract was treated with acetone, the mixture was converted into a solid mass readily soluble in water and methanol but insoluble in ether, benzene, and chloroform. This mass contained no scopoletin, but after hydrolysis with 10% hydrochloric acid for 12 hr, without heating, scopoletin was detected by thin-layer chromatography on silica gel.

For separation, the pulverulent mixture was acetylated with acetic anhydride in pyridine (48 hr without heating). After the elimination of the acetylating reagents, the mixture of acetylation products was dissolved in chloroform and the solution was washed with water and evaporated, and the residue was chromatographed on alumina (activity grade III). Elution with a mixture of benzene and chloroform (1:1) gave an acetyl derivative with mp 169-170°C (from methanol) having the composition  $C_{24}$  H<sub>26</sub>O<sub>13</sub>. The UV spectrum of this compound had maxima at 228, 290, and 340 mµ and the IR spectra lacked absorption bands of an OH group but exhibited a broad band at 1720-1760 cm<sup>-1</sup> (-O-C-CH<sub>3</sub> and a  $\delta$ -lactone ring) and bands at 1610 and 1570 cm<sup>-1</sup> (aromatic C=C bond), and at 830 and 890 cm<sup>-1</sup> (tetrasubstituted benzene ring). The NMR spectrum had signals at  $\delta$  2.00 ppm (C-CO-CH<sub>3</sub>, 9 protons),  $\delta$  2.07 ppm (C-CO-CH<sub>3</sub>, 3 protons),  $\delta$  3.79 ppm (aromatic OCH<sub>3</sub>, 3 protons), a doublet at  $\delta$  4.18 ppm (-CH-CH<sub>2</sub>-O-CO, 2 protons) (J = 6 Hz), a multiplet at  $\delta$  5.24 ppm (-CH-CH-, 4 protons), and a doublet at  $\delta$  6.30 ppm (J = 10 Hz) (olefinic proton at C<sub>3</sub>). An olefinic proton at C<sub>4</sub> also appeared in the form of a doublet at  $\delta$  7.67 ppm (J = 16 Hz). Singlets at  $\delta$  7.05 and 6.8 ppm are due to aromatic protons in positions 5 and 8.

From the products of the hydrolysis of the acetyl derivative with 10% hydrochloric acid we isolated scopoletin, and glucose was detected on a paper chromatogram with a reference sample (butanol-acetic acid-water, (4:1:5); spots revealed with aniline phthalate).

The hydrolysis of the acetyl derivative with barium hydroxide yielded scopolin (scopoletin 7-glucoside) [2, 3], which has been found in the genus Artemisia for the first time.

The NMR spectra were taken by M. R. Yagudaev.

#### REFERENCES

1. K. S. Rybalko, I. A. Gubanov, and V. I. Vlasov, Med. prom. SSSR, 2, 19, 1964.

2. W. Brandt, Chem. Zbl., vol II, p. 1199, 1915.

3. W. Rihel, A. Hunger, and T. Reichstein, Helv. Chim. Acta, 36, 434, 1953.

14 March 1969

Institute of the Chemistry of Plant Substances AS UzSSR

UDC 547.819

# SCOPOLETIN AND B-SITOSTEROL FROM ARTEMISIA BRACUNCULUS

## A. Mallabaev, I. M. Saitbaeva, and G. P. Sidyakin

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, p. 320, 1969

From the epigeal part of <u>Artemisia dracunculus</u> L. [1], collected in the budding and flowering stage in the neighborhood of the village of Nura (Alai valley, TadzhSSR) by chloroform extraction we have isolated 5% of total extractive substances. When the chloroform extract was treated with ether, a hydrocarbon  $C_{29}H_{60}$  with mp 63-64° C identical with the compound from <u>Artemisia absintium</u> [2] was obtained.

The ethereal solution was separated into acidic, phenolic, and lactone fractions. The acidic fraction was chromatographed on KSK silica gel. On elution with ether, acicular crystals with mp  $201-202^{\circ}$  C deposited which, on the basis of their IR spectrum, R<sub>f</sub> value, and a mixed melting point were identified as the coumarin scopoletin. Then the unsaponifiable neutral fraction was chromatographed on alumina. From a methanolic eluate we isolated a substance with the composition C<sub>29</sub>H<sub>50</sub>O, mp 139-140° C (from acetone), which gave the Liebermann-Burchard reaction for sterols.

By comparing the IR spectra and  $R_f$  values and by means of a mixed melting point test the substance was identified as  $\beta$ -sitosterol. Scopoletin and  $\beta$ -sitosterol have not previously been found in <u>Artemisia dracunculus</u>.

# REFERENCES

1. M. I. Goryaev, V. S. Bazalitskaya, and P. P. Polyakov, The Chemical Composition of Wormwood [in Russian], Alma-Ata, 1962.

2. I. S. Akhmedov, Sh. Z. Kasymov, and G. P. Sidyakin, KhPS [Chemistry of Natural Compounds], 5, 57, 1969.

### 18 March 1969

Institute of the Chemistry of Plant Substances AS UzSSR

UDC 547.97.2

# FLAVONOIDS OF ARMORACIA RUSTICANA AND BARBAREA ARCUATA

N. S. Fursa, V. I. Litvinenko, and P. E. Krivenchuk

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, p. 320, 1969

In the epigeal parts of <u>Armoracia rusticana</u> Gaerth. Mey et Scherb. and <u>Barbarea arcuata</u> Rchb. by one-dimensional and two-dimensional paper chromatography and qualitative reactions [1] we have detected not less than five and eight flavonoid substances, respectively. The total flavonoids were separated on a column of Kapron. On elution with aqueous methanol, methanol, and mixtures of chloroform and methanol and of acetone and water, two individual compounds were isolated from the leaves of <u>A. rusticana</u> (X-1 and X-2) and three from the flower clusters of <u>B. arcuata</u> (C-1, C-2, C-3).

As a result of alkaline cleavage and acidic and enzymatic hydrolysis and the features of the IR and UV spectra with ionizing and complex-forming reagents [2-4], X-1, with mp  $275-277^{\circ}$  C was identified as kaempferol, X-2 with mp  $311-313^{\circ}$  C as quercetin, C-1 with mp  $306-309^{\circ}$  C as isorhamnetin, C-2 with mp  $166-176^{\circ}$  C as isorhamnetin  $3-\beta$ -D-glucopyranoside, and C-3 provisionally as isorhamnetin  $3-\beta$ -D-glucosyl- $6-\beta$ -D-glucoside.

## REFERENCES

- 1. V. A. Bandyukova, Rast. res., 1, no. 4, 1965.
- 2. T. A. Geisman, The Chemistry of Flavonoid Compounds, Pergamon Press, NY, 107, 1962.

3. I. P. Kovalev and V. I. Litvinenko, KhPS [Chemistry of Natural Compounds], 1, 233, 1965.

4. V. I. Litvinenko and N. P. Maksyutina, KhPS [Chemistry of Natural Compounds], 1, 420, 1965.

4 February 1969

#### Zaporozh'e Medicinal Institute

Khar<sup>•</sup>kov Chemical and Pharmaceutical Scientific-Research Institute

UDC 547.972

#### PHENOLIC COMPOUNDS OF RHODODENDRON LUTEUM

N. F. Komissarenko and I. G. Levasheva

Khimiya Prirodnykh Soedinenii, Vol. 5, No. 4, p. 321, 1969

We have previously reported the isolation from <u>Rhododendron luteum</u> Sweet (pontic azalea) growing in the Ukrainian Poles'e the flavonoids quercetin, hyperoside, and avicularin [1]. From the same species growing in the Caucasus myricitrin