FLAVONOIDS OF THE EPIGEAL PART OF Scutellaria glabrata

M. P. Yuldashev, É. Kh. Batirov, and V. M. Malikov

Continuing an investigation of flavonoids of plants of the genus *Scutellaria* growing in Central Asia [1], we have studied the phenolic components of the epigeal part of *Scutellaria glabrata* Vved [2].

The comminuted air-dry raw material (400 g), gathered in the flowering period (July 26, 1990) in the Shakhristan pass (Turkestan range, Tadzhikistan Republic), was exhaustively extracted with ethanol at room temperature. The combined extract was evaporated in vacuum, and the concentrated residue was diluted with water and treated successively with petroleum ether, chloroform, ethyl acetate, and butanol. After the solvents had been distilled off, 4 g of chloroform fraction and 5 g of ethyl acetate fraction were obtained.

The chloroform fraction was chromatographed on silica gel in a chloroform – hexane gradient system. Compounds (I)-(IV) were isolated.

Chromatography of the ethyl acetate fraction on silica gel in solvents consisting of chloroform and methanol in various ratios led to the isolation of flavonoids (II) and (V). We used UV, PMR and mass spectra for the identification of the flavonoids.

Chrysin (5,7-dihydroxyflavone) (I) — light yellow crystalline substance with the composition $C_{15}H_{10}O_4$ (M⁺ 254), mp 289-291°C, $\lambda_{max}^{\text{ethanol}}$ 270, 310 nm. In the mass spectrum, in addition to the peak of the M⁺ 254 ion, there were diagnostic peaks of ions with m/z 152 and 102. According to the PMR and mass spectra, ring A contained two hydroxy groups, in positions 5 and 7, while ring B was unsubstituted [3].

Wogonin (5,7-dihydroxy-8-methoxyflavone) (II) — yellow crystalline substance with the composition $C_{16}H_{12}O_5$ (M⁺ 284, mp 202°C, λ_{max} 247, 277, 319 nm. The PMR spectrum revealed the signals of the protons of an OCH₃ group (3.85 ppm, s) and of H-3 (6.70 ppm, s) and H-6 (6.90 ppm, s), of the protons of an unsubstituted ring B (7.41 ppm, 3H, m, and 7.98 ppm, 2H, m) and of a 5-OH group (13.12 ppm, br.s). The mass spectrum of flavonoid (II) contained the peaks of ions with m/z 284 (M⁺), 269 (M - CH₃)⁺ (100%), 241 (M - CH₃ - CO), 167, 139, 105, 102, and others.

Skullcapflavone I (2',5-dihydroxy-7,8-dimethoxyflavone) (III) — light yellow crystalline substance with the composition $C_{17}H_{14}O_6$, mp 259-261°C, λ_{max} 272, 343 nm. The UV spectrum of the substance was characteristic for 5-hydroxyflavones. The mass spectrum showed peaks with m/z 314 (M⁺), 299 (M – CH₃)⁺ (100%), 271 (M – CH₃ – CO), 181, 153, 121, and 113. The ion peaks observed showed the presence of one hydroxy and two methoxy groups in ring A and of one hydroxy group in ring B. The 100% peak of the ion with m/z 299 showed the presence of a methoxy group in position 8 [4]. PMR spectrum in DMSO-d₆: 3.75 and 3.84 (s, each, 2 × OCH₃), 6.56 (s, H-3), 6.97 (s, H-6), 6.87-7.91 (m, H-3', 4', 5', 6'), and 12.91 ppm (br.s, 5-OH). In addition of sodium acetate did not lead to a bathochromic shift in the UV spectrum of the compound under consideration [5].

2',5,6'-Trihydroxy-7,8-dimethoxyflavone (IV) — yellow crystalline substance, composition $C_{17}H_{14}O_7$ (M⁺ 330), mp 284-285°C, λ_{max} 265.5, 339 nm. The PMR spectrum contained the signals of protons at 3.64 and 3.80 (s, each, 2 × OCH₃), 6.50 (s, H-3), 6.67 (d, 8 Hz, H-3', 5'), 6.83 (s, H-6), 7.16 (t, 8 Hz, H-4'), and 13.20 ppm (br.s, 5-OH). According to its mass and PMR spectra, substance (IV) contained two methoxy groups and three hydroxy groups. The maximum peak of the ion with m/z 315 (M – CH₃)⁺ in the mass spectrum indicated the presence of an 8-OCH₃ group, and the peaks of ions with m/z 138 and 134 showed the presence of two hydroxy groups in ring B. According to the PMR spectrum, they occupied positions 2', and 6' [6, 7].

Institute of Chemistry of Plant Substances, Academy of Sciences of the Uzbekistan Republic, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 3, pp. 471-472, May-June, 1993. Original article submitted December 28, 1992.

Chrysin 7-O-\beta-Glucuronide (V) — light yellow crystals with the composition C₂₁H₁₈O₁₀, with mp 223-225°C, λ_{max} 269, 306 nm. Acid hydrolysis formed chrysin and D-glucuronic acid.

The flavonoid under consideration was identified by its UV and PMR spectra and by direct comparison with a specimen isolated from *S. ramosissima*.

REFERENCES

- 1. M. P. Yuldashev, É. Kh. Batirov, and V. M. Malikov, Khim. Prir. Soedin., 178 (1992).
- 2. Flora of the USSR [in Russian], Izd. Akad. Nauk SSSR, Moscow-Leningrad, Vol. XX (1954), p. 124.
- 3. T. P. Popova, V. I. Litvinenko, V. G. Gordienkov, and D. A. Pakaln, Khim. Prir. Soedin., 730 (1976).
- 4. M. Goudard, J. Farve-Bonvin, P. Lebreton, and J. Chopin, Phytochemistry, 17, 145 (1978).
- 5. T. Horie, M. Tsukayama, M. Masumura, et al., Bull. Chem. Soc. Jpn., 52, 2950 (1979).
- 6. T. Tomimori, Y. Miyaichi, Y. Imoto, et al., Yakugaku Zasshi, 104, 529 (1984).
- 7. Y. Kikuchi, Y. Miyaichi, Y. Yamaguchi, et al., Chem. Pharm. Bull., 39, 199 (1991).