THE CHEMICAL COMPOSITION OF THE EXTRACTIVE

SUBSTANCES OF Saussurea controversa

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Saussurea controversa DC (syn. S. discolor auct. non DC), family Asteraceae, is a perennial plant growing in Eastern Siberia. The epigeal part of the plant is used in folk medicine for the treatment of pulmonary diseases, in glaucoma, as a hemostatic, and as an antirheumatic agent [1].

We have investigated the epigeal part of S. conversa (without peduncles) collected in the period of budding and partial flowering at the beginning of August, 1991, in Irkutsk province. The dried and comminuted raw material was exhaustively extracted successively with methanol and a 1:1 mixture of methanol and water. The concentrated methanolic and aqueous methanolic extracts (~ 10.6 and $\sim 15.6\%$, respectively, of the weight of the dry raw material) were fractionated with the aid of solvents of increasing polarity: hexane, chloroform, ethyl acetate, and n-butanol.

The total substances extracted by hexane and chloroform were chromatographed on columns of silica gel in a hexane—acetone system with the proportion of the latter increasing from 0 to 100%. Elution of the column with hexane yielded a fraction of the $C_{23}-C_{31}$ alkanes, and hexane—acetone (19:1) gave the free sterols β -sitosterol, stigmasterol, and campesterol. The hydrocarbons and the sterols were identified by the method of chromato-mass spectrometry under conditions described previously [2]. An increase in the proportion of acetone in the eluting mixture to 30-40% led to the isolation of β -sitosterol β -D-glucopyranoside, mp 296-298°C [2].

The fractions of the *S. controversa* extract that were soluble in ethyl acetate and n-butanol were chromatographed on polyamide in chloroform—methanol and water—methanol gradient systems. As a result, three compounds were isolated.

Compound (I) $-C_{15}H_{10}O_7$, MS, m/z 302, mp 308-310° 256, 370 nm [3] was identified as quercetin.

Compound (II) — $C_{27}H_{30}O_{17}$, FAB-MS, m/z 373 (M + H)⁺, mp 192-194°, $\lambda_{max}^{CH_3OH}$ 259, 360 nm [3] was identified as rutin.

Compound (III) — $C_{17}H_{24}O_9$, FAb – MS, m/z 373 (M + H)+; 211 (M — hexose + J)+, mp: 188-190°C (alcohol), $\lambda_{max}^{CH_3OH}$ 266 nm [4]; ¹³C NMR (CH₃OH-d₄), δ , ppm : 135.60 (C-1); 105.88* (C-2, 6); 154.65 (C-3, 5); 136.30 (C-4); 131.57 (C-7); 130.40 (C-8); 63.86 (C-9); 105.70* (C-1'); 76.06 (C-2'); 78.65** (C-3'); 71.70 (C-4'); 78.15** (C-5'); 62.94 (C-6'); 57.39 (2 × OCH₃); ¹H NMR (5000 MHz in CH₃OH-d₄), δ ppm: 674 (2H, s, H-2, 6), 6.53 (1H, dt, 15.8; 1.5; H-7), 6.31 (1H, dt, 15.8; 5.6; h-8), 4.21 (2H, dd; 5.7; 1.5; H-9 ab), 3.84 (6H, s, 2 × OCH₃), 4.86 (1H, d; 7.6; H-1'), 3.47 (1H, m, H-2'), 3.41 (2H, m, H-3', 4'); 3.21 (1H, ddd; 5.2; 3.5; 2.6; H-5'); 3.66 (1H, dd, 12; 5.2; H-6a'); 3.78 (1H, dd; 12; 2.6; H-6b'). The assignment of the chemical shifts of the carbon atoms in the ¹³C NMR spectrum was made on the basis of a comparison of information for similar structures [5], and that of the chemical shifts of the protons with the use of 2D COSY spectra for (III). On the basis of what has been said above, compound (III) was identified as syringin (eleutheroside B) [4]. This substance was also isolated from the water-soluble residue of the extract by chromatography on silica gel in the chloroform—methanol—water (63:23:3) system.

This is the first time that all these substances have been described for S. controversa.

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