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FLAVONOIDS FROM *Cephalaria grossheimii*

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We have continued research on chemical components from representatives of the family Dipsacaceae Lindl. (teasel) [1, 2], one species of which, *Cephalaria grossheimii* Bobr., is endemic to Azerbaidzhan [3]. According to Cherepanov [4], this species is synonymous with *C. kotschyi* Boiss et Hoh. However, this contradicts the literature on the phytochemistry of these plants. Thus, roots of *C. kotschyi* contained the alkaloids gentianine, gentianadine, and gentianaine [5] whereas alkaloids were not observed in those of *C. grossheimii*.

Flowers of *C. kotschyi* contained the flavonoids kaempferol, hyperoside, quercimeritrin, cinaroside, cephaside, and cephacoside [2]. The flavonoid composition of *C. grossheimii* has not been studied.

Therefore, we studied the flavonoid composition of *C. grossheimii* flowers. Flowers of this plant contained oleanolic acid in addition to apigenin, hyperoside, quercimeritrin, cinaroside, and palustroside. It can be seen that the flavonoid composition of flowers differed significantly for the two compared plants. The recent literature is consistent with this [6, 7].

Dry flowers of *C. grossheimii* (2 kg) that were collected near Rozgov, Lerik Region, Republic of Azerbaidzhan, during full flowering (beginning of July 2007) were extracted with ethanol (96%, 1:8) for 1 d. The extract was decanted. The extraction was repeated two more times using a fresh portion of ethanol (1:6) each time. The extracts were combined and evaporated in a rotary evaporator (fraction 1). The processed raw material was extracted a fourth time with ethanol (80%, 1:8) for 1 d. The extract was filtered and evaporated to a watery residue (fraction 2).

Fraction 1 was treated with water (300 mL), shaken, and extracted successively with $CHCl_3$, EtOAc:hexane, and EtOAc.

Purification of the $CHCl_3$ extract over Al_2O_3 and recrystallization afforded oleanolic acid, $C_{30}H_{18}O_3$, mp 305-306°C (EtOH), $[\alpha]_D^{20}$ +79 ± 2° (*c* 1.2, MeOH). The crystals were white; soluble in alcohols, $CHCl_3$, and Py; and insoluble in H_2O.

The EtOAc:hexane extract was evaporated. Recrystallization from aqueous EtOH gave apigenin, $C_{15}H_{10}O_5$, mp 342-343°C. The crystals were light-yellow, soluble in alcohols, and insoluble in H_2O.

The EtOAc extract was dried over Na_2SO_4 and evaporated to dryness. The solid was dissolved in MeOH (50 mL) and left at room temperature for 2 d. The resulting crystals were filtered off and recrystallized from MeOH to afford hyperoside, $C_{21}H_{20}O_{12}$, mp 230-232°C. UV spectrum (λ_{max}, MeOH, nm): 361, 305 sh, 277; +$AlCl_3$: 434, 342 sh, 275. Light-yellow crystalline powder, soluble in alcohols and DMF, insoluble in $CHCl_3$. Acid hydrolysis produced quercetin (64%) and D-galactose.

The mother liquor was evaporated to dryness. Recrystallization of the solid from acetone afforded quercimeritrin, $C_{21}H_{20}O_{12}$, mp 250-252°C, $[\alpha]_D^{20}$ −58 ± 2° (*c* 0.3, Py:MeOH). UV spectrum (λ_{max}, MeOH, nm): 370, 256; +CH_3COONa: 350, 256. Acid hydrolysis produced quercetin (65%) and D-glucose.

The acetone solution was evaporated to dryness. Recrystallization of the solid from aqueous EtOH afforded cinaroside, $C_{21}H_{20}O_{11}$, mp 230-232°C. UV spectrum (λ_{max}, EtOH, nm): 352, 266 sh, 256; +CH_3COONa: 352, 268 sh, 258. Acid hydrolysis produced luteolin (66%) and D-glucose.

A precipitate that formed in fraction 2 after 1 d was filtered off. The filtrate was evaporated to a small volume and extracted with *n*-BuOH. The extract was evaporated. Recrystallization of the solid from aqueous EtOH afforded palustroside, $C_{27}H_{30}O_{15}$, mp 172-173°C, $[\alpha]_D^{20}$ −50 ± 2° (*c* 0.3, DMF), soluble in DMF and aqueous alcohols, slightly soluble in EtOH, insoluble in EtOAc [8].

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The isolated compounds were identified by comparison with authentic samples and using PMR and ^{13}C NMR spectra obtained on a Bruker AM-300 spectrometer.

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