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ISOIMPERATORIN FROM Phlojodicarpus sibiricus

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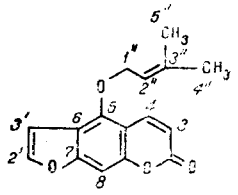
To confirm statements made previously concerning the instability of the chemical composition of plants of the genus Phlojodicarpus, we have investigated an ethanolic extract of Phlojodicarpus sibiricus Steph. ex Speng. K.-Pol., collected in the environs of Yakutsk in the flowering phase.

The whole plant was extracted with ethanol. The ethanolic extracts were concentrated to small volume and cooled, and the crystalline mixture of suksdorfin and 3'-O-acetyl-4'-2"-methylbutanoyl-cis-khellactone that deposited was separated off. It was established by PMR spectroscopy that the mixture of products isolated was quantitatively and qualitatively identical with the mixture of esters isolated from the plant growing in the Chita province [1]. The amount of esters was ∿1.5% on the weight of the dry plant.



The residual extract was diluted with water and was extracted successively with petroleum ether and dimethyl ether. Both extracts were treated with 0.5% KOH and were washed with water. The ethereal extract yielded umbelliferone, which was identified from its physicochemical constants. The petroleum ether extract was chromatographed on SiO_2 using petroleum ether as eluent. This gave a compound with the composition $C_{16}H_{14}O_4$, mp 107-108° (from benzene); λ_{max} (CH_3OH): 220, 248, 260, 267, and 309 nm. These results, and also the PMR spectrum, agreed with the characteristics of imperatorin [2]. The ^{13}C spectrum also agreed with the structure of the latter. The assignment of the signals was made on the basis of a comparison of the spectra of related substances and by the off-resonance method [3, 4]. Below, we give the chemical shifts of the carbon atoms of imperatorin in the ^{13}C NMR spectra taken in DMSO-d_6 (ppm relative to TMS):

C-2	160,1	s	C-6	113,9	s	C-3′	105,5 d
C-3	112,4	d	C-7	157,3	s	C-1″	69,4 t
C-4	139,6	d	C-8	93,6	d	C-2″	119,5 d
C-4a	106,8	s	C-8a	152,1	s	C-3″	139,0 s
C-5	148,6	s	C-2′	146,0	d	C-4″	18,0 q
						C-5″	25,5 q

The evaporated aqueous residue was chromatographed on SiO_2, and chloroform–methanol (9:1) eluted a crystalline compound (yield 0.15%) identical in its melting point and ^1H and ^{13}C

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NMR spectra with umbelliferone β-D-apiosyl-(1 → 6)-β-D-glucopyranoside, which has been isolated previously from Ph. sibiricus growing in the environs of Ulan-Bator [5].

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COUMARINS OF Artemesia vulgaris

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We have analyzed the epigeal and hypogeal parts of Artemesia vulgaris L. (mugwort wormwood), collected in the environs of Tomsk in the budding phase.

The comminuted raw material was extracted with 96% ethanol, and the ethanolic extract was concentrated in vacuum and diluted with water (1:1). The precipitate that deposited was separated off, and the filtrate was treated with chloroform. The resin remaining after the solvent had been evaporated off was subjected to preparative chromatography on "Leningradskaya S" (medium) paper impregnated with a mixture of formamide and acetone (1:3) [1]. Separation of the combined substances by descending chromatography in chloroform gave coumarin, scopoletin, and umbelliferone.

The further separation of the combined coumarins was performed by preparative rechromatography on "Leningradskaya S" paper that had been washed with a 5% solution of Trilon B and with distilled water and had been impregnated with 10% dimethylformamide in methanol, with butan-1-ol–acetic acid–water (4:1:5) as the mobile phase (system 1) [2], and on Silufol plates with the mobile phases ether–petroleum ether (3:1) (system 2) and hexane–ethyl acetate (3:1), acidified with acetic acid (system 3).

The substances were eluted from the chromatogram with 96% ethanol, and were obtained in the individual form of recrystallization from methanol.

From mugwort wormwood we isolated 18 substances of coumarin nature, and of them, on the basis of IR and UV spectra and mixed melting points with authentic samples we identified esculin, esculetin, umbelliferone, scopoletin, and coumarin. An analysis of the available literature showed a distinct concentration of scopoletin in the genus Artemesia [3].

The qualitative compositions of the coumarins from the epigeal and hypogeal parts of the plant in the budding phase were identical.

The amount of coumarins in the epigeal part of the plants was 1.9%, and that in the hypogeal part 1.2%. The determination was made by photoelectric colorimetry [4].

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