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3,4-METHYLENEDIOXY-5-METHOXYPROPIOPHENONE AND TERPENOID COUMARINS OF Ferula caucasica

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We have investigated the roots of Ferula caucasia Korov. (Caucasian giant fennel) collected on the multicolored outcrops in the basin of the R. Chikil-chai (Azerbaidzhan SSR). The species F. caucasia, which is close to F. caspica Bieb., has not previously been studied chemically.

Three substances (I-III) were isolated from the resin of the roots by chromatography on a column of neutral alumina (4 × 110 cm, activity grade IV) with elution by hexane, hexane–chloroform (9:1) and (1:3), and chloroform. When fractions 42-44 (eluted by hexane–chloroform 1:3) were rechromatographed on a column of alumina (3.5 × 50 cm) with elution by hexane–chloroform (1:1), chloroform, and chloroform–ethyl acetate (9:1), another two substances (IV and V) were isolated in the individual state. The volume of the fractions collected was 100 ml.

Substance (I) – $C_{11}H_{12}O_4$, mp 91-92°C (from hexane) – was isolated from fractions 9-13 eluted by hexane–chloroform (9:1). Its IR spectrum showed absorption bands at (cm^{-1}) 1685 (CO of a ketone group conjugated with an aromatic ring); 1630, 1605, 1518 (C=C of a benzene ring); 2800 (methylenedioxy group), and 2860 (methoxy group).

The NMR spectrum contained the signals of a CH_3-CH_2-group (t, 1.10 ppm, 3 H, and q, 2.76 ppm, 2 H, J = 7 Hz), of a methylenedioxy groups (s, 5.91 ppm, 2 H), of a methoxy group (s, 3.83 ppm, 3 H), and of an aromatic ring (d, 6.94, and d, 7.07 ppm, 1 H each, J = 2 Hz).

Substance (II) – $C_{24}H_{32}O_5$, mp 212-214°C (from ethanol) – was isolated from fractions 49-53 eluted by hexane–chloroform (1:3). Its IR spectrum had the absorption bands of OH groups (3430 cm^{-1}), of the CO of an α-pyrone (1710 cm^{-1}), and of the C=C bonds of an aromatic ring (1620, 1560, 1520 cm^{-1}).

Substance (III) – $C_{24}H_{32}O_5$, mp 221-222°C (from ethanol) – was isolated from fractions 54-58 eluted by chloroform. In the region of characteristic frequencies of the IR spectrum were observed the bands of OH groups (3440, 3370 cm^{-1}), of the CO of an α-pyrone (1700 cm^{-1}), and of an aromatic ring (1620, 1560, 1510 cm^{-1}).

Substance (IV) – $C_{24}H_{28}O_4$, mp 184-185°C (from chloroform) – was eluted by chloroform when fractions 42-44 were rechromatographed. In the region of characteristic frequencies the IR spectrum of compound (IV) had the absorption band of a CO of an α-pyrone (1730 cm-1) and those of an aromatic ring (1620, 1520 cm^{-1}). OH groups were absent.

Substance (V) – $C_{29}H_{38}O_6$, mp 176-178°C (from ethanol) – was isolated from the fractions eluted by chloroform–ethyl acetate (9:1). The IR spectrum revealed the bands of OH groups (3350, 3500 cm^{-1}), of the CO group of a δ-lactone (1720 cm^{-1}), and of an aromatic ring (1625, 1513 cm^{-1}).

A direct comparison of the IR and NMR spectra, and also of the physicochemical constants, of substance (I-V) with those of 5-methoxy-3,4-methylenedioxypropiophenone (latifolone, crocatone) [1-4], ferucrin [5], isosamarkandin [6], badrakemone [7], and isosamarkandin angelate [8], respectively, showed the identity of the compounds compared.

Benzo-α-pyrone derivatives and farnesiferol A have been isolated previously from Caspian giant fennel, which is morphologically close to Caucasian giant fennel [9, 10].

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FLAVONOIDS OF *Trigonella grandiflora* AND *T. tenuis*

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We have studied the epigeal parts of *Trigonella grandiflora* Bunge and *T. tenuis* Fisch. collected in the flowering period in the Chimganskii gorge, Tashkent province.

The comminuted air-dry raw material was exhaustively extracted with 90% ethanol on the boiling water bath. The combined extracts were evaporated to 1/3 of their initial volume and were diluted with water and freed from lipophilic impurities with the aid of chloroform. The flavonoids were extracted with ethyl acetate, and the combined ethyl acetate extracts were concentrated almost to dryness.

The combined flavonoids were separated on a column filled with cellulose powder, the eluents being increasing concentrations of ethanol in water. *T. grandiflora* yielded two substances in the individual state.

Substance (I) – light yellow crystals with mp 260-265°C. In the UV spectrum, λ_{max} (nm): C_2H_5OH: 270, 302 sh., 336; CH_3COONa: 280, 300 sh., 380; $AlCl_3$: 277, 305, 350, 385; $AlCl_3$ + HCl: 278, 303, 344, 384; CH_3COONa + H_3BO_3: 271 sh., 344.

In the IR spectrum (cm^{-1}): 3390, 3260 (OH groups); 1657 (C=O group); 1612, 1570, 1505 (π-conjugation of an aromatic nucleus); 1040, 1010 (C bond for a glycoside); 990, 972, 856, 780, 751, 701 (distribution in a lateral phenyl radical).

Substance (I) did not undergo hydrolysis in the presence of dilute mineral acids. When it was hydrolyzed with Kiliani's mixture apigenin, D-glucose, and a very small amount of L-arabinose were formed.

Thus, according to UV and IR spectroscopy and the products of acid hydrolysis, substance (I) was a C-glycoside of apigenin. In a mixture with an authentic sample of the C-glycoside vitexin it gave no depression of the melting point, which enables it to be identified as 8-C-D-glucopyranosylapigenin (vitexin).

The same compound was isolated from *T. tenuis*.

Substance (II) – light yellow crystals with mp 255-257°C. In the UV spectrum, λ_{max}(nm): C_2H_5OH: 255, 267, 294 sh., 346; $AlCl_3$: 276, 302, 430; $AlCl_3$ + HCl: 265 sh., 276, 296 sh., 357, 384; CH_3COONa: 278, 325, 386; CH_3COONa + H_3BO_3: 264, 275, 340 sh., C_2H_5ONa: 286, 378 sh., 405. In contrast to substance (I), under the action of solution of basic lead acetate the spots on the chromatogram acquired an orange coloration. R_f 0.76 in the butan-1-ol–CH_3COOH–H_2O (4:1:5) system.

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