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# ESTERS OF THE ROOTS OF *Ferula pallida*

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Studying the roots of *Ferula pallida* Eug. Kor. growing in the semidesert (the mountains of Alym-Tau, 70 km north west of Tashkent), we directed our attention to the fact that their composition differs from that of plants growing in the subalpine zone (environs of the village of Zarkent). By repeated chromatography we isolated eight esters from a methanolic extract. Four of them [$C_{23}H_{32}O_5$, mp 129-130°C (I); $C_{22}H_{30}O_4$, mp 121-122°C (II); $C_{23}H_{32}O_5$, mp 93-95°C (III); and $C_{22}H_{30}O_3$ (oil) (IV)] were identified by their IR and NMR spectra as the previously known ferutin [1], ferutinin [1], teferin [2] and teferidin [3], respectively. Nevertheless, among them new substances giving a red coloration with vanillin/sulfuric acid were present in predominating amount. They had the following characteristics: $C_{26}H_{34}O_7$, mp 109-110°C, $M^+$ 446, $[\alpha]_D^{25}$ +272.7° (c 1.1; methanol), $R_f$ 0.2 [TLC on Silufol, hexane–ethyl acetate (2:1) system], red coloration (V); $C_{20}H_{34}O_4$, mp 133-135°C (from methanol) $[\alpha]_D^{25}$ +48.1° (c 0.84; methanol), $R_f$ 0.25, dark green coloration (VI); $C_{25}H_{38}O_7$, mp 109-110°C, $[\alpha]_D^{25}$ +51.5° (c 0.97; methanol), $R_f$ 0.1, green coloration (VII); and $C_{20}H_{34}O_4$, mp 120-121°C, $[\alpha]_D^{25}$ –21.4° (c 1.07; methanol), $R_f$ 0.40, orange coloration (VIII). These substances are new, and we have called them taulin, tauferin, taufedin, and taufenin, respectively.

They were hydrolyzed by the action of caustic soda. Their IR spectra showed absorption bands of the carbonyl of the ester of an α,β-unsaturated acid in the 1700-$cm^{-1}$ region. The UV spectra of (VI) and (VIII) had maxima at 216 nm (double bond conjugated with a carbonyl group) and those of (V) and (VII) at 362 nm (2,4,5-trimethoxybenzoic acid chromophore).

The NMR spectra of taulin had singlets at 3.41 ppm (2 H) and 3.78-3.66 ppm (3 H and 6 H, respectively) due to the 3,4,5-trimethoxybenzoic acid residue, a broadened singlet at 6.03 ppm and a multiplet at 6.25 ppm (olefinic protons), singlets at 1.75 and 1.50 ppm of 3 H each (methyls on double bonds, a doublet at 1.00 ppm, J = 7 Hz (6 H, methyls of an isopropyl grouping), and a broadened singlet at 5.80 ppm (hemiacyl proton). When (V) was saponified with caustic soda, the above-mentioned trimethoxybenzoic acid and an alcohol with the composition $C_{15}H_{24}O_3$ were formed. With this composition and with two double bonds, the alcohol must have a bicyclic, most probably, guaiane structure. Tauferin is also an ester of the acid mentioned, with an alcohol $C_{15}H_{28}O_3$. Its NMR spectrum contains, in addition to the signals from the acid, a broadened doublet at 5.16 ppm, J = 10 Hz (hemiacyl proton), singlets at 1.36 and 1.21 ppm (3 H each, tertiary hemihydroxyl methyl groups), and doublets at 0.82 and 0.94 ppm, J = 7 Hz (3 H each, methyls of an isopropyl grouping). It contains no double bonds and, consequently, has a bicyclic structure (apparently also a guaiane structure).

According to their spectra (NMR, mass, IR) characteristics, tauferin and taufenin are esters of angelic acid and of saturated sesquiterpene alcohols with the composition $C_{15}H_{28}O_3$. The NMR spectrum of tauferin shows the following signals: singlets at 1.98 and 1.85 ppm (1.5 and 4.5 H) and multiplet at 6.05 ppm (methyls and olefinic proton of angelic acid), quartet at 5.0 ppm, $J_1$ = 10 Hz, $J_2$ = 3 Hz (hemiacyl protons), singlets at 1.20 and 1.15 ppm (3 H each, tertiary methyl groups), and doublets at 0.94 and 0.84 ppm, J = 5 Hz (3 H each,

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methyls of an isopropyl grouping). With this composition and no double bonds, the alcohol must have a bicyclic structure.

The NMR spectrum of taufenin shows, in addition to the signals of the protons of the acid residue, singlets at 0.88 ppm (3 H, angular methyl) and 1.18 ppm (3 H, hemihydroxyl tertiary methyl), doublets at 0.95 and 0.85 ppm, J = 6 Hz (3 H, methyls of an isopropyl group), and quartet at 4.94 ppm, $J_1$ = 10 Hz, $J_2$ - 3 Hz (hemiacyl proton).

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# LACTONES OF *Inula helenium*

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In a study of the inflorescences, leaves, roots, and rhizomes of *Inula helenium* L. we found that they contained lactones.

As was shown by preliminary paper-chromatographic investigations using specific reagents, the epigeal organs contained lactones fluorescing bright blue in UV light, which were assigned to benzo-α-pyrone derivatives [1].

The compounds detected were isolated from the epigeal part of the plant by a method described elsewhere [2]. For this purpose, ethanolic extracts were concentrated and the concentrates were mixed with polyamide sorbent, which was then dried and deposited on columns of polyamide. The columns were eluted with benzene, benzene with gradually increasing concentrations of chloroform, chloroform, and chloroform—ethanol. The benzene fractions yielded, after crystallization from chloroform—ethanol (9:1) a substance (I) with the empirical formula $C_{10}H_8O_4$, mp 202-204°C; and the fractions eluted by chloroform +5% of ethanol yielded substance (II) with the composition $C_9H_6O_3$, mp 230-232°C (from methanol).

By a comparison of IR and UV spectra, a mixed melting point, and a comparison of $R_f$ values on chromatography in various systems and fluorescence in UV light, the substances isolated were identified as scopoletin [1] and umbelliferone [2, 3].

No derivatives of benzo-α-pyrone nature were detected in the hypogeal organs. Thin-layer chromatography [fixed layer of silica gel; benzene—methanol—ethyl acetate (12:3:1) system; chromogenic agent a 1% solution of vanillin in concentrated sulfuric acid] showed the presence of no less than five substances of terpenoid nature in purified ethanolic extracts of the roots and rhizomes [4].

By the usual methods of adsorption chromatography on alumina using as eluents mixtures of petroleum ether and benzene in various ratios we isolated four substances with mp 76-78°C 112-114°C, 133-134°C, and 174-176°C which were identified by direct comparison with authentic samples as, respectively, alantolactone, isoalantolactone, dihydroalantolactone, and dihydroisoalantolactone [5].

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