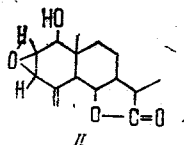


Previously [2], by comparing the NMR spectra of the hydroxylactone (II) and saponified badkhyzinin it was observed that (II) probably has the structure of the 11,13-dihydro derivative of saponified badkhyzin. In actual fact, saponification of the sesquiterpene lactone badkhyzidin [11] (11,13-dihydrobadkhyzinin) led to the hydroxylactone, which was identified by a comparison of its properties, melting point, and IR and NMR spectra. Consequently, the hydroxylactone has the structure (II):



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LACTONES OF *Ferula badghysi*

S. V. Serkerov

UDC 547.913.5 + 547.473.2

By chromatographic separation on a column of alumina of the resin from the roots of *Ferula badghysi* Korov. collected in the Turkmen SSR we have obtained four substances, provisionally denoted by the symbols (I-IV).

Substance (I), $C_{20}H_{24}O_5$, mp 139-140°C (needles from ethanol). The IR spectrum shows the presence of a γ -lactone ring (1770 cm^{-1}), and α, β -unsaturated ester group (1703 cm^{-1}), a conjugated ketone group (1670 cm^{-1}), and double bonds ($1640, 1610\text{ cm}^{-1}$). The presence of a conjugated group is also shown by the UV spectrum: $\lambda_{\text{max}} 252\text{ nm}$ ($\log \epsilon 4.12$).

The NMR spectrum (here and below δ scale, ppm, spectrometer Varian HA-100D) showed the following signals: d 1.38 ($J = 7\text{ Hz}$, 3H, CH_3); d, 1.95 ($J = 1.5\text{ Hz}$, 3H, $\text{CH}_3-\text{C}=\text{O}$); d/d, 2.25 ($J_1 = 7, J_2 = 1.5\text{ Hz}$, 3H, $\text{CH}_3-\text{CH}=\text{O}$); s, 2.30 (6H, $\text{CH}_3-\text{C}=\text{O}$); d, 3.7 ($J = 10\text{ Hz}$, 1H, $-\text{CH}-$); q, 4.55 ($J_1 = 10, J_2 = 7.5\text{ Hz}$, 1H, lactone proton); m, 5.55 ($J_1 = 10, J_2 = 10, J_3 = 4\text{ Hz}$, hemi-ester proton); 6.18 (2H, $-\text{CH}=\text{O}$ of an ester group).

Substance (II), $C_{20}H_{24}O_5$, mp 98-99°C (from aqueous ethanol). ν_{max} (cm^{-1}): 1765 (CO of a γ -lactone), 1708 (CO of an α, β -unsaturated ester), 1668, and 1640 (double bonds); $\lambda_{\text{max}} 216\text{ nm}$ ($\log \epsilon 3.75$).

Saponification gave tiglic acid and a hydroxylactone $C_{15}H_{18}O_{14}$ mp 185-186°C. Its NMR spectrum had the following signals: s, 0.40 (3H, $\text{CH}_3-\text{C}-$); t, 1.80 (3H, $\text{CH}_3-\text{C}=\text{O}$); d/t, 1.92 ($J = 7\text{ Hz}$, 3H, $\text{CH}_3-\text{CH}=\text{O}$); d, 3.25

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($J = 3$ Hz, 1H); and q, 3.43 ($J_1 = 5$, $J_2 = 3$ Hz, 1H)—protons in an oxide ring; d, 4.49 ($J = 5$ Hz, 1H)—hemi-ester proton; m, 5.78 (1 H)—vinyl proton of an ester; doublets of an exocyclic methylene group (5.26 and 5.29, $J = 2$ Hz); $\text{CH}_2 =$ in a lactone ring (d 4.98 and 6.12, $J = 4$ Hz, 1H each), and other signals.

Substance (III), $\text{C}_{20}\text{H}_{24}\text{O}_4$, mp 113–114°C (from aqueous ethanol). The IR spectrum had bands at 1765 and 1670 cm^{-1} (α, β -unsaturated ester), and 1650 and 1603 cm^{-1} (conjugated double bonds). UV spectrum, λ_{max} 232 and 224 nm ($\log \epsilon$ 4.32 and 4.33, respectively).

The NMR spectrum showed the doublets of a $\text{CH}_2 =$ group in a lactone ring (6.26 and 5.49, $J = 3.5$ Hz), the signals of olefinic protons (d, 6.28, $J = 10$ Hz; q, 5.78, $J_1 = 10$, $J_2 = 6$ Hz); the vinyl proton of an ester group (m, 6.04); an exocyclic $\text{CH}_2 =$ group (5.21, 5.46)— a $\text{CH}_2 - \overset{|}{\underset{|}{\text{C}}} -$ group (s, 0.85); vinyl methyls of an ester group (t, 1.80; d/t, 1.90, $J = 7$ Hz); and the protons of a hemi-ester group and a lactone ring (m, 4.85, 2H).

Substance (IV), $\text{C}_{11}\text{H}_{10}\text{O}_4$, mp 144–145°C. In the IR spectra there are strong bands of a δ -lactone ring (1710 cm^{-1}) and of a benzene ring (1620, 1580, 1510 cm^{-1}).

A direct comparison of the IR, UV, and PMR spectra, and also the absence of a depression of a mixed melting point with authentic samples, substances (I), (II), (III), and (IV) were identified as badkhyzin [1], badkhyzinin [2, 3], dehydrooopodin [4, 5], and scoparone [6], respectively.

The IR spectra were taken in paraffin oil, the UV spectra in ethanol, and the NMR spectra of (I) and (III) in deuteriochloroform and of (II) in benzene. TMS was used as internal standard: s) singlet; d) doublet; t) triplet; q) quartet; m) multiplet; d/t) doublet each component of which consists of a triplet.

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