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A CRYSTALLINE SUBSTANCE FROM ARTEMISIA RUTIFOLIA

R. I. Evstratova and P. V. Chugunov

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From the green epigeal part of Artemisia rutifolia Steph. et Spreng. collected on August 5, 1967 in the flowering phase (Central Asia, region of the Turkestan range) we have isolated a colorless crystalline substance with the composition $C_{15}H_{18}O_5$, mp 235° C (decomp., from ethanol).

Chromatography in a thin layer of Al_2O_3 (activity grade IV) in the benzene–methanol (9 : 1) system gave a clear spot with R_f 0.55, revealed with a 1% solution of $KMnO_4$ in 1% H_2SO_4. IR spectrum, λ_{max}, cm^{-1}: 3520, 3580, 3620 (OH), 1765 (γ-lactone), 1660 and 820 (C=C). In the NMR spectrum there are two methyl singlets in the 1.23 and 1.63 ppm regions, two doublet at 5.39 and 6.0 ppm (J = 3 and 4 Hz) (an exocyclic methylene in conjugation with the carbonyl of a γ-lactone), and a quartet at 4.50 ppm (lactone proton).

The composition of the substance, its IR and NMR spectra, and its solubility on heating in alkalis shows that it is a sesquiterpene lactone.

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LACTONES FROM ARTEMISIA TENUISECTA

Sh. Z. Kasymov and G. P. Sidyakin

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The epigeal part (5 kg of leaves and flower heads) of Artemisia tenuisecta Nevski, collected in August 1968 in the Tashkent region, were extracted with chloroform. The concentrated extract was dissolved in 500 ml of ethanol, 400 ml of water was added, and the mixture was extracted with chloroform. The concentrated chloroform extract (25 g) was chromatographed on alumina (activity grade IV, 600 g). Elution with benzene gave a substance $C_{15}H_{18}O_3$, mp 168–170° C, identical with α-santonin [1].

Elution with benzene–methanol (19 : 1) gave a substance $C_{15}H_{22}O_4$ with mp 230–231° C; mol. wt. 266 (mass spectrometry); IR spectrum, cm^{-1}: 3470 (OH), 1750 (γ-lactone), 1650 and 810 (double bond). The NMR spectrum has a singlet at δ 0.82 ppm ($C-CH_3$, tertiary), a doublet at 1.20 ppm ($HC-CH_3$), and a singlet at 1.15 ppm [$C(OH)-CH_3$, tertiary]. The spectral characteristics show that the substance isolated is a sesquiterpene lactone of the type of selinane. Acetylation with acetic anhydride in pyridine gave an acetyl derivative $C_{17}H_{24}O_5$, mp 217–218° C. The IR spectrum of the latter retained the absorption band of a hydroxyl group. Consequently, the lactone is identical with mibulactone, as is shown by the results of a direct comparison of the IR spectra and by the melting point of a mixture of samples of these substances [2].

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THE PRESENCE OF ANHYDRIDES OF RESIN ACIDS IN ROSIN

I. I. Bardyshev and O. D. Strizhakov

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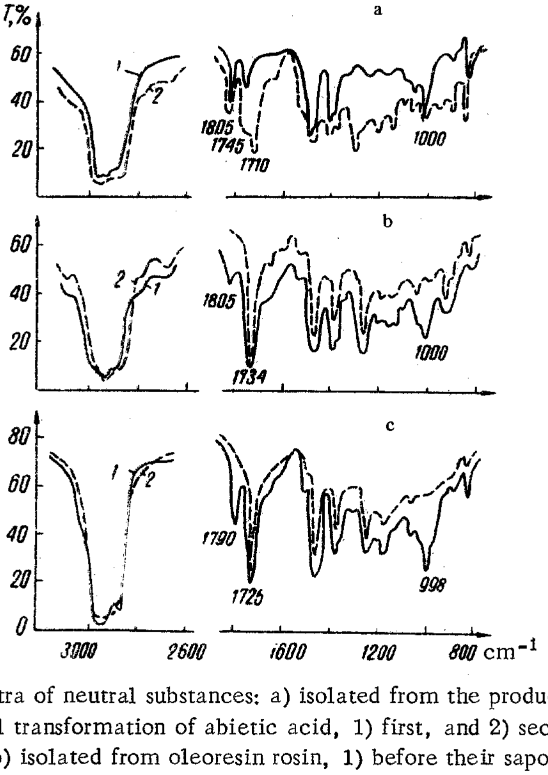
Anhydrides of resin acids (RA) are present in Finnish tall oil rosin [1] obtained by the rectification of the mixture of resin and fatty acids at a high temperature (up to 300° C). At such a temperature, the formation of RA anhydrides is a completely normal phenomenon.

We have shown that RA anhydrides are present in small amounts (0.2% and less) in the oleoresin rosin of the Scotch pine (P. silvestris L.) obtained at 170–220° C. Moreover, heating pure abietic acid at 340° C gave its anhydride. On this basis it may be assumed that the RA anhydrides are formed in the industrial treatment of the oleoresin for rosin.

Abietic acid (acid no. 185.5; mp 173.5–174° C; $[\alpha]_D^{\text{ethanol}}$ −114.8°) was heated at 340° C for 2 hr (in an atmosphere of CO_2). The product was chromatographed on Al_2O_3 (activity grade II.) Four fractions were isolated from the eluates. In fraction 1 (figure, a) the products of the decomposition of abietic acid (abietene, abietin, etc.) were concentrated, while fractions 2–4 contained the abietic acid anhydride, which was isolated from them in the pure form by crystallization after the elimination of the free acid with the calculated amount of 1% NaOH solution (acid no. 0; saponification no. 187.4; mp 153–155° C; IR spectrum, cm^{-1}: 1805, 1745, and 1000 cm^{-1} [2, 3]).



IR spectra of neutral substances: a) isolated from the product of the thermal transformation of abietic acid, 1) first, and 2) second fractions; b) isolated from oleoresin rosin, 1) before their saponification; 2) after their saponification; c) the same for tall oil rosin.