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THE PRESENCE OF ANHYDRIDES OF RESIN ACIDS IN ROSIN

I. I. Bardyshev and O. D. Strizhakov

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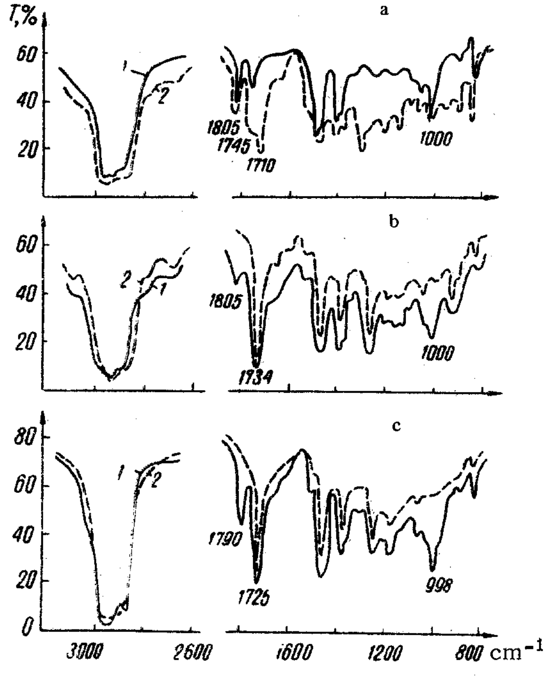
Anhydrides of resin acids (RA) are present in Finnish tall oil rosin [1] obtained by the rectification of the mixture of resin and fatty acids at a high temperature (up to 300° C). At such a temperature, the formation of RA anhydrides is a completely normal phenomenon.

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.

When pine oleoresin rosin of the Borisov Wood-Chemical Factory (acid no. 169.2; saponification no. 181.0) was chromatographed on Al_2O_3, a mixture of neutral substances was obtained with the absorption bands in the IR spectrum characteristic for anhydrides (1805, 1734, and 1000 cm^{-1}) [3], which disappeared after the saponification of this mixture with 0.5 N ethanolic KOH at 40° C (figure, b). Pure abietic acid was extracted from the saponification product. Under the conditions mentioned, esters of the resin acids (if they are present in the mixture) are not saponified and therefore the acid was produced by the saponification of the anhydride.

Tall oil rosin from the Mari Pulp and Paper Combine (acid no. 140.7; saponification no. 167.3) was extracted with petroleum ether. After the elimination of the ether, the extract was chromatographed three times on Al_2O_3 as a result of which a mixture of neutral substances containing RA anhydrides was obtained (figure, c). From the products of the saponification of this mixture were isolated acids identified by the GLC method [4] as abietic and dehydroabietic acids.

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URSOLIC ACID IN SORBUS AUCUPARIA

A. G. Shavva, L. G. Matyukhina, and I. A. Saltykova

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In a search for a convenient material for studying the biosynthesis of triterpenes we have investigated the composition of the leaves and stems of Sorbus aucuparia L. collected in September 1968 in the environs of Leningrad. From the acid fraction of a chloroform extract we isolated ursolic acid with mp 283–283.5° C for the identification of which we prepared a number of derivatives by the usual methods: the acetate with mp 287–289° C, the methyl ester with mp 167.5–168° C, and its acetate with mp 245–246° C. The melting points and specific rotations of the compounds obtained were compared with literature data [1, 2].

The acid isolated and its derivatives gave no depression of the melting point in admixture with the authentic compounds, and the IR spectra of the methyl ester of the acid and its acetate coincided with those of corresponding derivatives of ursolic acid. The results of the elementary analysis of the compounds correspond to the calculated figures. The neutral compounds isolated previously from the bark of this plant [3] were not detected in the leaves.

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