## THE CATECHINS OF POLYGONUM CORIARIUM

Sh. Yu. Islambekov, A. K. Karimdzhanov, A. I. Ismailov, and A. S. Sadykov Khimiya Prirodnykh Soedinenii, Vol. 4, No. 3, p. 191, 1968

Continuing our study [1] of the tanning substances of the roots of P. coriarium by partition chromatography on a silica gel column, we have isolated four catechins with mp 210-211° C, 216-217° C, 170° C, and 236-237° C.

A study of the products of the alkaline cleavage of these substances showed that the first three catechins form phloroglucinol and gallic acid and the last one phloroglucinol and protocatechuic acid.

Enzymatic hydrolysis with tannase affected only the catechin with mp 210-211° C, splitting it into (--)-epigallocatechin and free gallic acid.

The IR spectra of the four catechins have broad bands at 3430-3280 cm<sup>-1</sup> (closely adjacent OH groups), while the spectrum of the catechin with mp 210-211° C has additional maxima at 1710 cm<sup>-1</sup> and 1140 cm<sup>-1</sup> (ester linkage).

On the basis of what has been said and the results of a comparison with the catechins from tea (by paper chromatography and by mixed melting points), the compounds isolated from P. coriarium have been identified as (-)-epigallocatechin gallate, (-)-epigallocatechin, (±)-gallocatechin, and (-)-epicatechin.

## REFERENCE

1. A. S. Sadykov, A. K. Karimdzhanov, A. I. Ismailov, and Sh. Yu. Islambekov, Nauchnye trudy TashGU, 2, no. 286, 51, 1966.

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## TRICYCLENE IN TURPENTINES FROM PINUS SILVESTRIS

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There is no information in the literature on the amount of tricyclene in turpentines from various species of Pinus. We have shown that tricyclene is an invariable component of all types of turpentines from the Scotch pine.

To detect tricyclene in pine oleoresin turpentine (Fig. 1, a) we distilled off the  $\alpha$ -pinene fraction (60%, Fig. 1, b) and fractionated it in a column with an efficiency of 50 theoretical plates. The tricyclene fraction (9.3%, Fig. 1,c) was oxidized with 5% aqueous potassium permanganate at 10° C and the residue (0.04%, Fig. 1, d) was purified in a PAKhV-03 preparative gas-liquid chromatograph at 130° C in a column (5000 × 8mm) containing deactivated Inza diatomite brick (grain size 0.03-0.5 mm) impregnated with dinonyl phthalate (30% of the weight of the brick). This gave 0.003% of tricyclene C<sub>10</sub>H<sub>16</sub> (Fig. 1, e) with mp 60° C; a mixture with an authentic sample of tricyclene melted at the same temperature.

The results of direct comparison of the IR spectra of the tricyclene that we isolated and a pure sample [1] show their complete identity (Fig. 2).

We have shown by analytical GLC that all types of turpentines from Pinus silvestris contain tricyclene: oleoresin turpentine 0.2%; extraction turpentine 0.3%; dry distillation turpentine 0.8%.

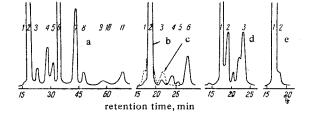


Fig. 1. Chromatograms of pine oleoresin turpentine (a); technical  $\alpha$ -pinene (b); tricyclene fraction isolated by a fractional distillation of technical  $\alpha$ -pinene (c); tricyclene fraction after its oxidation with KMnO<sub>4</sub> (d); and a sample of tricyclene purified on a PAKhV-03 (e). Peaks: a) tricyclene; 2) α-pinene; 3) camphene; 4) \(\beta\)-pinene; 5) \(\beta\)-myrcene; 6) 3-carene; 7) 1 + d1-limonene; 8) 1-8-phellandrene;

9) p-cymene; 10)  $\gamma$ -terpinene; 11) terpinolene.