of the anthraquinone pigments and resins. Then it was dissolved in water and chromatographed on cellulose. The aqueous eluate from the cellulose was transferred to a Kapron column. The catechins were desorbed from the Kapron with methanol. The dried residue from the methanolic eluate was dissolved in water-saturated ether. The ethereal solution was chromatographed on a column of silica gel with ether as the mobile solvent and water as the stationary phase [2, 3]. After the ether had been driven off, the separated catechins were recrystallized from water and dried over phosphorus pentoxide at 140° C. The following catechins were isolated: (+)-catechin, (–)-epicatechin, and (–)-epicatechin gallate.

The physical constants, the number of pyrocatechin hydroxyls, the behaviour or chromatography, and the qualitative reactions of the catechins corresponded to published data.

The quantitative determination of the catechins was carried out spectrophotometrically directly on the paper after their separation by two-dimensional chromatography in the systems of solvents described and treatment with silver nitrate [3]. The measurements of the maximum optical densities of the colorations of the spots were carried out on a SF-4A spectrophotometer at a wavelength of 460 mμ. The concentrations were calculated from calibration curves constructed for the pure catechins.

The roots collected in June were found to contain 0.039% of (+)-catechin, 0.028% of (–)-epicatechin, and 0.078% of (–)-epicatechin gallate, while the seeds contained 0.117%, 0.065%, and 0.039%, respectively. The leaves contained only traces of these catechins.

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PRESENCE OF *l*-β-PHELLANDRENE IN THE TURPENTINE OF PINUS SILVESTRIS

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The possible presence of *l*-β-phellandrene in natural turpentines was first shown in 1948 [1]. It was reported that the head fraction of the dipentene isolated by the rectification of turpentine contained a component the properties of which resemble those of *l*-β-phellandrene.

Later [2], we found that this component is present in almost all industrial samples of natural turpentines from *Pinus silvestris* (Scotch pine).

In the present investigation it has been shown that this component of turpentine is in fact *l*-β-phellandrene.

The α-pinene fraction (60%) was distilled off from resin turpentine in an industrial rectifying column of the Borisov wood-chemical factory (Fig. 1a). The distillation residue (6000 g) was rectified in a column with an efficiency of 50 theoretical plates. Appropriate fractions (600 g, Fig. 1b) were redistilled in the same column and the fractions obtained (100 g, Fig. 1c) were distilled for a third time in a column with an efficiency of 100 theoretical plates. The *l*-β-phellandrene fraction (7.2 g, Fig. 1d) was purified on a PAKhV-OZ chromatograph at 150° C in a 9000 × 6mm column. Inza diatomite brick (grain size 0.25–0.50 mm) was used as the solid support; the stationary liquid phase was polyethyleneglycol 2000 (30% of the weight of the solid support) and the carrier gas was nitrogen.

The fraction (1.51 g, Fig. 1e) having n_D^{20} 1.4824, d_4^{20} 0.8413, $[\alpha]_D$ –24° was rechromatographed to give 90%-pure *l*-β-phellandrene (0.5 g, Fig. 1f) with n_D^{20} 1.4863, d_4^{20} 0.8412, $[\alpha]_D$ –18.6°, MR_D 46.49. UV spectrum: λ_{max} (in alcohol): 231 mμ (log ε 4.25); IR spectrum – see Fig. 2.

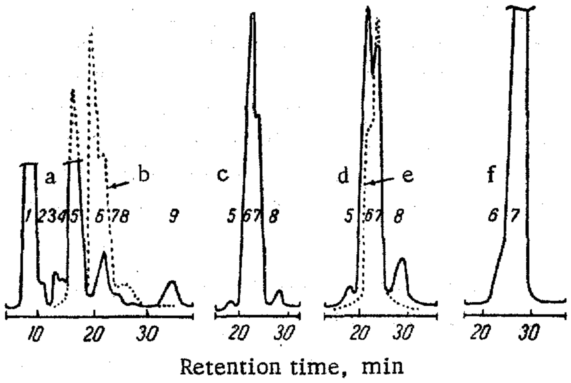

Fig. 1. Chromatograms: a) Turpentine from the Borisov wood-chemical factory; b) *l*-β-phellandrene fraction after the first rectification; c) *l*-β-phellandrene fraction after the second rectification; d) *l*-β-phellandrene fraction after the third rectification; e) *l*-β-phellandrene fraction isolated on a preparative chromatography; f) sample of *l*-β-phellandrene isolated by repeated chromatography. 1) α-Pinene; 2) camphene; 3) β-pinene; 4) myrcene; 5) Δ^3-carene; 6) dipentene; 7) *l*-β-phellandrene; 8) substance of undedetermined structure; 9) terpinolene.

Pure *l*-β-phellandrene [3] has n_D^{20} 1.4882, d_4^{20} 0.8409, $[\alpha]_D$ −15.8° MR_D 46.8; UV spectrum: λ_{max} (in ethanol) 232 mμ (log ε 4.67).

Chemical derivatives characteristic for *l*-β-phellandrene were obtained: from a fraction corresponding to Fig. 1d a nitrosite with mp 102–102.5° C, $[\alpha]_D$ −168° (c 6; chloroform); and from the fraction corresponding to Fig. 1e a nitrosochloride with mp 105–106.5° C, $[\alpha]_D$ −252° (c 5; chloroform).

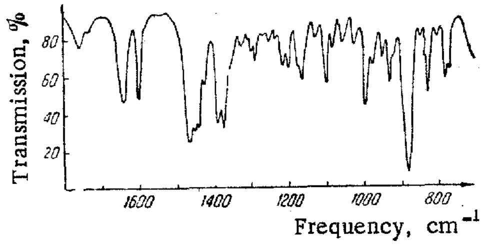

Fig. 2. IR spectrum of a sample of *l*-β-phellandrene isolated from turpentine.

All the turpentines from Pinus silvestris contained *l*-β-phellandrene in the following amounts: resin turpentines (Borisov, Gor'kii, and Neivo-Rudyanka factories) −1%; extraction turpentines (Vakhtan, Novo-Belitsa factories) −0.5%; and the dry-distillation turpentine of the Borisov factory −0.2%.

The analysis of the turpentine was carried out on a UA-1 instrument at 115° C in a 4000 × 4 column. Solid support was Inza diatomite brick (grain size 0.25–0.50 mm). The liquid phase was tricresyl phosphate (25% of the weight of the solid carrier). The carrier gas was hydrogen.

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