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COMPOSITION OF THE RESINOUS SUBSTANCES OF CONIFEROUS NEEDLES.

II. ACIDS FROM THE RESINOUS SUBSTANCES OF THE NEEDLES

OF *Pinus silvestris*

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In a study of the composition of pine needles, the resinous substances isolated from it were separated into individual groups of substances [1]. Among them is a group of acids (2.2% of the neutral fraction of the resinous substances). We give the results of an investigation of the composition of the acids by the GLC method. The acids were isolated by two methods: 1) column chromatography on silica gel; and 2) the acid-alkali method [2]. Table 1 gives the results of the identification of the acids (the acids isolated by the acid-alkali method are denoted by dots and their percentage yields are not given).

On chromatography, the acids isolated from the neutral fraction gave 28 peaks, and those isolated from the extract by the acid-alkali method gave nine peaks.

Among the acids methylatable by diazomethane, C₁₀-C₂₄ saturated and unsaturated, and also aromatic, acids were detected. According to Table 1, under the conditions of separation selected the acids issued in double peaks and in the acids isolated by the acid-alkali method very small amounts of myristic and vanillic acids were detected.

Of the unsaturated acids, the highest percentage was represented by linoleic, which is present together with oleic acid to some degree in the group of substances combined under the name of vitamin F usually forming the bulk of the acids of extracts of coniferous needles. Of the aromatic acids, syringic and veratric were present in very small amount, and acids of the cinnamic type (vanillic and caffeic) were also detected.

The presence of high-boiling acids (above C₂₄) in the resinous substances was established, but in view of the absence of pure substances they were not identified.

The methyl esters of the acids were identified. Separation was carried out on an LKhM-72 chromatogram with a thermal conductivity detector with a three-meter stainless-steel column having a diameter of 4 mm of which 2 m was filled with Chromaton N-AW (0.25-0.315 mm) upon which Apiezon L had been deposited in an amount of 20% of the weight of the solid support and 1 m was filled with chromaton N-AW (0.43-0.60 mm) upon which Apiezon L had been

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TABLE 1. Acids of the Resinous Part of *Pinus silvestris*

Acid	Amount, %	
	of the extract	of the combined acids
Adipic	0,18	8,18
Capric + phthalic	0,29	13,18
Undecanoic	0,01	0,45
Lauric	0,07	3,17
Sebacic	0,18	8,18
Myristic	—	—
Vanillic	—	—
Caffeic	0,0004	0,018
Margaric	0,017	0,770
Syringic + veratric	0,067	3,040
Oleic	0,030	1,360
Linoleic	0,230	10,450
Stearic + behenic	0,180	8,180
Lignoceric	0,110	4,990
Unidentified acids	0,840	38,180
Total	2,200	100,0

deposited in an amount 30% of the weight of the solid support. The carrier gas was helium at a rate of flow of 120 ml/min. The column was heated from 110°C at a rate of 2.5°C/min, and the temperature of the detector was 275°C. The qualitative composition of the acids isolated was determined by the method of adding pure substances and the quantitative composition by the method of internal normalization.

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MELISSIC ACID AND β -SITOSTEROL FROM *Morina kokanica*

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We have previously reported the isolation from the leaves of *Morina kokanica*, family Morinaceae of hentriacontane, rutin, and ursolic acid. Continuing a study of the leaves, we have isolated another two substances. For this purpose, a chloroform extract of the raw material was passed through a column of silica gel and it was washed with petroleum ether and then with mixtures of petroleum ether and diethyl ether with increasing concentrations of the latter. Elution of the column with a mixture in a ratio of 9:1 gave the first substance and an 8:2 mixture gave the second substance.

The first substance was recrystallized several times from petroleum ether. Its composition is $C_{30}H_{60}O_2$, mp 90-92°, acid nature, titrating with alkalis. Molecular weight 452, determined by the neutralization method and confirmed by mass spectrometry. The IR spectrum of the substance showed absorption bands characteristic for acyclic saturated organic acids of normal structure. The substance isolated has been characterized as melissic acid.

The second substance was purified by repassage through a column with silica gel, being eluted with benzene-ethyl acetate (9:1). Its composition is $C_{29}H_{50}O$, mol. wt. 414 (mass spectrum), mp 137-138°C (acetone), acetate 129-130°C. The substance gives positive reactions for steroids. On chromatographic investigation in a layer of silica gel in several solvent systems, it had the same R_f values as β -sitosterol, and when the substance obtained was mixed with β -sitosterol no depression of the melting point was observed. The IR spectrum of the substance could be superimposed completely on that of β -sitosterol.

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