

*Collection of Drug in the Spring of 1925.*—The first lot was dug April 17th on Mendota Drive near the Black Hawk Country Club grounds on the north slope of the hill. The soil was a sandy loam and there had been no recent rains. The plants were hardly above ground. The moisture determinations by the xylene method yielded 65.0 per cent for the roots and 64.5 per cent for the rhizomes.

A second lot was collected May 2nd near the Illinois Central right of way west of the Cemetery. The plants were from six to twelve inches high. Recent rains had left the soil rather moist. Moisture determinations yielded 74.0 per cent for the roots and 74.1 per cent for the rhizomes.

A third lot was collected May 19th from the same locality. Moisture determinations yielded 73.2 per cent for the roots and 73.4 per cent for the rhizomes.

P. S. The sucrose, reported above, when recrystallized by A. H. Uhl, melted at 181–182°;  $[\alpha]_D = +59^\circ$ ; after hydrolysis  $[\alpha]_D = -11^\circ$ . For sucrose  $[\alpha]_D = 66.5^\circ$ ; for inverted sucrose  $[\alpha]_D = -32^\circ$ . Possibly the irregularities in melting point and angle of rotation are attributable to the presence of inorganic substances as indicated by the ash content. The amount of material was not sufficient to admit of purification by repeated crystallization.

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### THE VOLATILE OIL OF LEDUM GRÆNLANDICUM.\*

BY E. V. LYNN, ARNOLD LEHMAN AND RUSSELL CAIN.

The plants of the genus *Ledum* are small, evergreen, ericaceous shrubs, growing in swamps, bogs and other wet places in the northern part of Europe, Asia and America. The species, *grænlandicum*, is a native of North America, where it is called Labrador Tea and used, especially by the natives as a pectoral and tonic. It differs from others of the same genus by the rusty and hairy character of the lower side of the leaf. The whole plant, and particularly the leaf, has an aromatic odor and taste which is very pleasing. The agreeable odor of *Ledum grænlandicum* suggested the possibility of valuable volatile constituents and an attempt was made to separate them.

The plant itself, commonly known as Labrador, Continental, or James Tea, presents a shrub-like growth, the stems being very stout and branching, occasionally reaching a height of five or six feet. The shrub is an evergreen, bearing snowy white flowers in a convex cluster. The leaves are alternate and sessile, ranging in length from one to three inches. They are oval shaped, narrow and entire, with upper surface smooth and the lower composed of densely matted, red-brown hairs.

As far as could be learned, no investigational work has ever been reported on this plant, although *Ledum palustre* has been examined repeatedly (see references). The latter gives by steam distillation from 0.3 to 2.0 per cent of a volatile oil, the yield apparently depending upon age of plants, season, habitat, portions used, etc. The oil is a greenish or reddish viscid liquid of a penetrating narcotic odor and a pungent, unpleasant and persistent taste, and consists of a stearoptene and a liquid portion in approximately equal proportions. The latter has a boiling point of 180° to 250° C.,  $d_{16} 0.93$  to 0.96. It frequently does not separate

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any further solid by lowering the temperature, but the dissolved stearoptene may be completely removed by vacuum distillation. The liquid was found to contain a ketone,  $C_{12}H_{24}O$ , which can be removed by sodium bisulphite and a sesquiterpene,  $C_{15}H_{24}$ .

The solid, which has been called ledum camphor, is a tricyclic sesquiterpene alcohol with the formula  $C_{15}H_{26}O$ . It crystallizes in prisms, melting point  $104-5^{\circ}C.$ ,  $(\alpha)_D$  in 10 per cent alcohol solution  $+7.98^{\circ}$ , boiling point  $282-3^{\circ}C.$ , soluble at  $17.5^{\circ}$  to the extent of 10.4 per cent. It sublimes readily in long needles but is so easily oxidized, even by the oxygen of the air, that great care must be exercised in distilling it at ordinary pressures. Dehydrating agents readily convert it into the basal sesquiterpene, ledene, which may or may not be identical with the one occurring in the oil. This artificial hydrocarbon boils at  $255^{\circ}C.$  and has a specific gravity of 0.9237 at  $19^{\circ}C.$ , 0.9349 at  $0^{\circ}C.$  Sundvik has found that the camphor is a powerful poison, acting principally upon the central nervous system.

A first distillation of a small quantity of the leaves from *L. grœnlandicum* giving sufficient oil to warrant further investigation, a larger quantity was distilled with steam at  $275^{\circ}C.$  The whole plant above ground, 43.6 Kg., yielded 6.4 cc. of oil without cohobation, or a return of only 0.013 per cent. Since this was very low, even if calculated on the basis of dried material, and much smaller than would have been expected from the preliminary distillation, it was concluded that little oil was present in the stems, which constituted a large portion of the total material.

In a second distillation the fresh leaves were used with a minimum amount of stems, the yield being 12.5 cc. from 40 Kg., using the same conditions as before. The aqueous distillate, 39 liters of which had been collected, was cohobated to give 3 cc. of oil, bringing the total to 15.5 cc. a return of 0.035 per cent. Based upon dry material, this would be about 0.1 per cent which is much smaller than the minimum from *L. palustre*.

The oil from both stems and leaves was dark amber in color while the later distillate was emerald green. Both possessed a characteristic odor resembling that of the plant and also somewhat similar to that of citral. The mixed oils had a specific gravity of 0.8998 at  $21.2^{\circ}C.$  and an index of refraction of 1.4917. Fractional distillation gave the following results:

166-175°	2.0 cc.	very slight color	245-255°	0.8 cc.	
175-185°	1.5 cc.		255-265°	1.4 cc.	amber color
185-195°	4.0 cc.		265-275°	1.6 cc.	
195-205°	1.3 cc.		275-285°	1.0 cc.	
205-215°	0.5 cc.		285-295°	0.6 cc.	
215-225°	0.9 cc.		295-305°	0.1 cc.	blue color
225-235°	0.7 cc.		305-310°	0.1 cc.	
235-245°	0.3 cc.		Residue		dark brown or black

The mixed fractions up to  $255^{\circ}$  gave a specific gravity at  $24.4^{\circ}$  of 0.8626, solubility in water 3.4 per cent, phenols by sodium hydroxide 2.8 per cent, aldehydes by sodium bisulphite 1.7 per cent,  $(\alpha)_D +1.21^{\circ}$ . The mixed fractions 255-295° gave specific gravity at  $24.4^{\circ}$  0.8922, solubility in water 5.0 per cent, phenols 3.5 per cent, aldehydes 0.71 per cent. All fractions were cooled in a freezing mixture but no solid material could be separated.

The amount of material at hand precluded any closer investigation but the

results obtained indicate several conclusions. There is very little if any ledum camphor or other stearoptene present in this species, although the season of the year, March, in which it was collected may have an influence. Fractionation points to limited quantity of phenols and aldehydes and little or no dicyclic terpenes, while strongly suggesting the presence of considerable monocyclic. Sesquiterpenes are undoubtedly contained in the oil, as is also the blue hydrocarbon azulene, which has been previously found in milfoil and wild ginger oils.

We are awaiting the opportunity to gather larger quantities of material in order to make a more extended investigation.

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## EXTRACTS OF ACONITUM COLUMBIANUM.

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The principal aconite species of the rocky mountain region is *Aconitum columbianum*. While it is widely distributed, its peculiar habitat localizes the plant to shady moist areas.

The plant was investigated by the author primarily for its poisonous properties. Interest in the study suggested a comparison of its therapeutic value with that of U. S. P. drug, the imported species.

Samples<sup>1</sup> were collected at two distinct periods of growth, the first when the plants were well started in their initial growth, *i. e.*, pre-flowering stage, and the second when in full bloom. The tubers (mother and daughter) were separated from the above ground portions in each collection. The method used in the chemical assay was the U. S. P. IX, official for aconite. The following is a summary of the results:

Tubers (flowering)	—0.839%
Tubers (young plants)	—0.774%
Above ground (flowering)	—0.350%
Above ground (young plants)	—0.758%

<sup>1</sup> Identified by Professor Aven Nelson, Botanist, University of Wyoming.