U. S. Department of Agriculture, Forest Service

FOREST PRODUCTS LABORATORY

In cooperation with the University of Wisconsin

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# THE CONIFER LEAF OIL INDUSTRY

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Chemist in Forest Products

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The production of oils from the leaves or needles of various conifers is a small but fairly old industry in the United States. According to the best estimates obtainable the value of the oils produced annually from this source amounts to approximately $50,000. The leaves of only a few species of conifers are regularly distilled for their oils, since it is only for these oils that a steady demand has been created. The principal species employed are the black spruce (Picea mariana, Mill.), white spruce (Picea canadensis, Mill.), eastern hemlock (Tsuga canadensis, Linn.), red juniper (Juniperus virginiana, Linn.), and arborvitae (Thuja occidentalis, Linn.). The oils of white spruce, black spruce, and hemlock are very similar in composition. No attempt appears to be made to keep the leaves of the latter species separate, and for practical purposes a distinction between them seems unnecessary. The annual consumption of spruce and hemlock oil is estimated at 40,000 to 50,000 pounds. It is quoted at 45 to 60 cents a pound. The leaf oil of the red juniper is used largely in insecticides, the annual consumption being 15,000 to 20,000 pounds. The prices of the oils from the various native conifers are approximately the same as that given above. The oil of Pinus picea imported from Europe is sold at about $4 per pound but the annual demand is below 50 pounds.

## Yields of Oil from Various Species

The oil is found in longitudinal ducts running through the needles. The number and size of the oil ducts vary greatly with the different species, and on these factors the yield of oil is largely dependent. The number of oil ducts may vary from one to ten. Naturally the species containing numerous ducts of large size will give the largest

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yield of oil. This assumption has been verified in the various species examined. The longleaf pine needle contains five large oil ducts, the average yield of oil being 0.42 per cent, while the lodgepole pine needle contains two oil ducts, the average yield of oil being only 0.16 per cent. In all cases the yields are given in per cent of the weight of the green leaves.

The approximate yields and principal constituents of the various species are given in the table presented on the following page.

## Properties, Composition and Uses

As a general rule, the oils have a pleasant odor resembling the fragrance of coniferous forests. Occasionally the freshly distilled oils have a disagreeable odor that frequently improves with age.

The oils are composed mainly of terpenes, terpene alcohols and their esters, and sesquiterpenes. (See table.) Among the terpenes pinene and limonene are ordinarily present. The attractive odor of the oils is due mainly to borneol and its acetic ester. In general, the more highly prized oils contain large amounts of borneol, both free and combined. Spruce and hemlock oil contain 35 to 50 per cent of these constituents. The popular Siberian needle oil, of which 5,000 to 10,000 pounds are imported, contains from 29 to 39 per cent bornyl acetate. Among the sesquiterpenes cadinene occurs most commonly. Thujone is a characteristic constituent of thuja oil from Thuja plicata and Thuja occidentalis.

It is difficult to obtain direct information on the purposes for which the various oils are employed. A large amount of correspondence addressed to manufacturers purported to use these oils in their products afforded very little information. Information on the uses to which the oils are put was obtained mainly from dealers and distillers of the oil.

The spruce and hemlock oils are extensively employed as a perfume in greases and shoe blackenings. They are also used in considerable quantities in liniments and other medicinal preparations.

| Species | Yield of oil | Specific gravity [1] | Principal constituents |
|---|---|---|---|
| Red pine (P. resinosa) | 0.10 ? | | |
| Pitch pine (P. rigida) | .10 ? | | |
| White pine (P. strobus) | .10 | 0.9012 | α-pinene |
| *Longleaf pine (P. palustris) | .40 | .8829-0.8849 | Camphene, β-pinene, borneol, cadinene |
| *Cuban pine (P. heterophylla) | .27 | .8877-0.8894 | Camphene, β-pinene, borneol, cadinene |
| *Western yellow pine (P. ponderosa) | .08 | .8718-0.8849 | α-pinene, dipentene, borneol |
| *Sugar pine (P. lambertiana) | .09 | .8676-0.8738 | α-pinene, β-pinene, dipentene, borneol |
| *Digger pine (P. sabiniana) | .09 | .8517-0.8566 | α-pinene, limonene |
| *Lodgepole pine (P. contorta) | .23 | .8690 | Phellandrene, β-pinene |
| *Red fir (Abies magnifica) | .15 | .8665 | Phellandrene, β-pinene, borneol |
| *White fir (Abies concolor) | .13 | .8720-0.8777 | α-pinene, β-pinene, phellandrene, borneol |
| *Douglas fir (Pseudotsuga taxifolia) | .16 | .8727-0.8759 | α-pinene, β-pinene, limonene, borneol |
| Red spruce (Picea rubens) | .20 | .9539 at 16° | Borneol, bornyl acetate |
| Black spruce (Picea mariana) | .60 | .9274 at 19° | Bornyl acetate, terpenes |
| White spruce (Picea canadensis) | .10 | .9216 | Bornyl acetate, limonene (?) |
| Hemlock (Tsuga canadensis) | .40 | .9288 at 20° | α-pinene, bornyl acetate |
| Balsam fir (Abies balsamea, Mill.) | | .8881 at 20° | α-pinene, bornyl acetate |
| White cedar (Thuja occidentalis) | .50 | .915-0.930 | α-pinene, fenchone, thujone, borneol |
| Western red cedar (Thuja plicata) | 1.00 | .9305 at 25° | Thujone, pinene |
| *Incense cedar (Lib. decurrens) | .23 | .8655-0.8733 | α-pinene, limonene, borneol, libocedrene |
| Red juniper (Juniperus virginiana) | .20 | .887-0.900 | α-pinene, limonene, borneol, cadinene |
| Tamarack (Larix laricina) | .15 | .8816 | α-pinene, bornyl acetate |

[1] At 15° unless otherwise stated.

*Examined by author.

Cedar oil from Juniperus virginiana is employed mainly in insecticides. Thuja oil from Thuja occidentalis is used in insecticides and liniments. Various native and foreign oils are employed medicinally as inhalations for lung diseases, and as additions to baths and ointments in rheumatic afflictions. Various perfumes contain certain amounts of needle oils whose value consists in having a so-called "ozonizing" effect. The oil of Pinus montana mixed with chloroform is used in quantity as an embrocation. In Europe, especially, the finer needle oils are used extensively as perfumes in soaps.

## Distillers of Oil

The greater portion of the oil is distilled by small farmers in New England during the winter months when the farm work is slack. In 1912 a company in Seattle, Wash., was engaged in the distillation of the leaf oil of red cedar (Thuja plicata) on an extensive scale. The branches, 3/4 inch or less in diameter, were delivered in Seattle in bundles of 100 pounds at a contract price of $4.50 to $5.50 per ton, depending on their oil content. The material was packed in the stills and distilled with steam at a pressure of 40 to 90 pounds for 3 to 5 hours, the distillation being discontinued when the amount of oil coming over did not exceed 10 cubic centimeters in 5 minutes.

The average yield of oil was about 1 per cent of the weight of the green material. Young trees contained the largest amount of oil, and the leaves were richest in January, February, and March. The oil had a market value of 40 cents a pound, but this was scarcely sufficient to cover the cost of production. Most of the oil was employed in the manufacture of an insecticide called "Mothine." This was a dry powder containing about 35 per cent of cedar oil and 65 per cent of an absorbent made by nitrating the finely ground shells of peach pits.

Attempts have been made at various times to utilize pine needles for the production of fiber after the oil had been removed by distillation. The most ambitious attempts in this direction were made by C. M. and O. C. Terrell, of Grant's Pass, Oreg., who obtained patents Nos. 675,206 and 758,874, covering methods and apparatus. The plant, described by Brown,[2] utilized leaves systematically picked from young

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[2] Scien. Amer. 84, 344 (1901).

trees of the western yellow pine. The stills consisted of wooden tanks with steam connections, and had a daily capacity of 2,000 pounds from which 10 pounds of oil were obtained. After suitable treatment the spent needles produced a long, tough fiber that could be woven into fabrics or made into mattresses when mixed with hair.

## Forest Service Investigations

The large amount of lumber cut from coniferous woods renders available large quantities of needles and twigs that at the present time are not only a sheer waste but are frequently the cause of destructive forest fires. If a sufficient market could be created for the oils a great economic advance in management would be effected. At present, however, the demand and price for oils of this type do not warrant their manufacture on a large scale.

The leaves of a number of the most important western and southern conifers were distilled to determine the yield and chemical composition of the oil. Samples of these oils were sent to various manufacturers for practical tests. The most promising oils, judged from odor, were those of the longleaf pine and western yellow pine. Unfortunately in nearly all the oils and ester content was low and their odors did not surpass those of the already firmly established spruce and hemlock oils. There appears to be an increasing utilization of conifer leaf oils, and the creation of a demand for new oils, as well as the extension of markets for the common ones, may be anticipated. It is frequently difficult, however, to introduce a new oil on the market, even though it may have decided merit.

## Principles of Distillation

The oil is removed from the leaves by the familiar method of steam distillation, usually at atmospheric pressure. As the steam passes upward through the needles the oil volatilizes and the mixed vapors pass together into a cooling apparatus where condensation takes place. The condensation products soon separate into a layer of oil and water, owing to their immiscibility and difference in specific gravity.

## Factors Influencing Yield of Oil

Steam distillation under pressure is more rapid and produces more oil than distillation at ordinary pressure. When steam at atmospheric pressure is employed a greater yield is obtained if the needles are cut into small pieces. In this way the oil ducts are more exposed to the action of the steam and more material can be placed in the still. Experiments have shown that a still will hold 25 to 50 per cent more material when it is finely cut.

The largest yields are obtained from young trees. In New England cedar oil is distilled almost entirely from small trees growing in old pastures and abandoned fields. All trees growing in the open contain more oil than those in a normal forest stand. The season of the year also appears to have a considerable effect, the data available essentially agreeing in that the most oil is obtained during the winter and spring months. The leaves of the western red cedar (Thuja plicata) were richest in January, February, and March; the leaves of incense cedar were richer in February and November than during the intervening summer months.

## The Still and Its Operation

The experimental still used by the Forest Service is shown in Figure 1. The still proper was constructed in three parts. The cylindrical body of the still for holding the needles was 3 feet 6 inches in height by 2 feet 3 inches in diameter, and was made of 16 B.W.G. copper. The ends were flanged out and provided with iron rings 1-3/4 inches wide. The cover of the still and the top of the heating vessel were similarly flanged and provided with rings. The cover and base were fastened to the cylinder with malleable iron clamps, asbestos wire tape being used in the joints. In order to support the needles the inner base of the cylinder was provided with lugs upon which rested a removable frame covered with 20-mesh No. 25 B.W.G. brass wire. The exterior of the still was covered with several layers of asbestos in order to reduce radiation of heat and condensation of the vapors. The heating vessel containing the water was 3 feet in diameter and 2 feet 1 inch high, and was constructed of 11-gage copper. This vessel was provided with a 1/2-inch water gage and a funnel attachment to a hand lever stop for introducing water when necessary. The condenser consisted of 20 feet of 1-1/4-inch copper tubing wound in a coil of

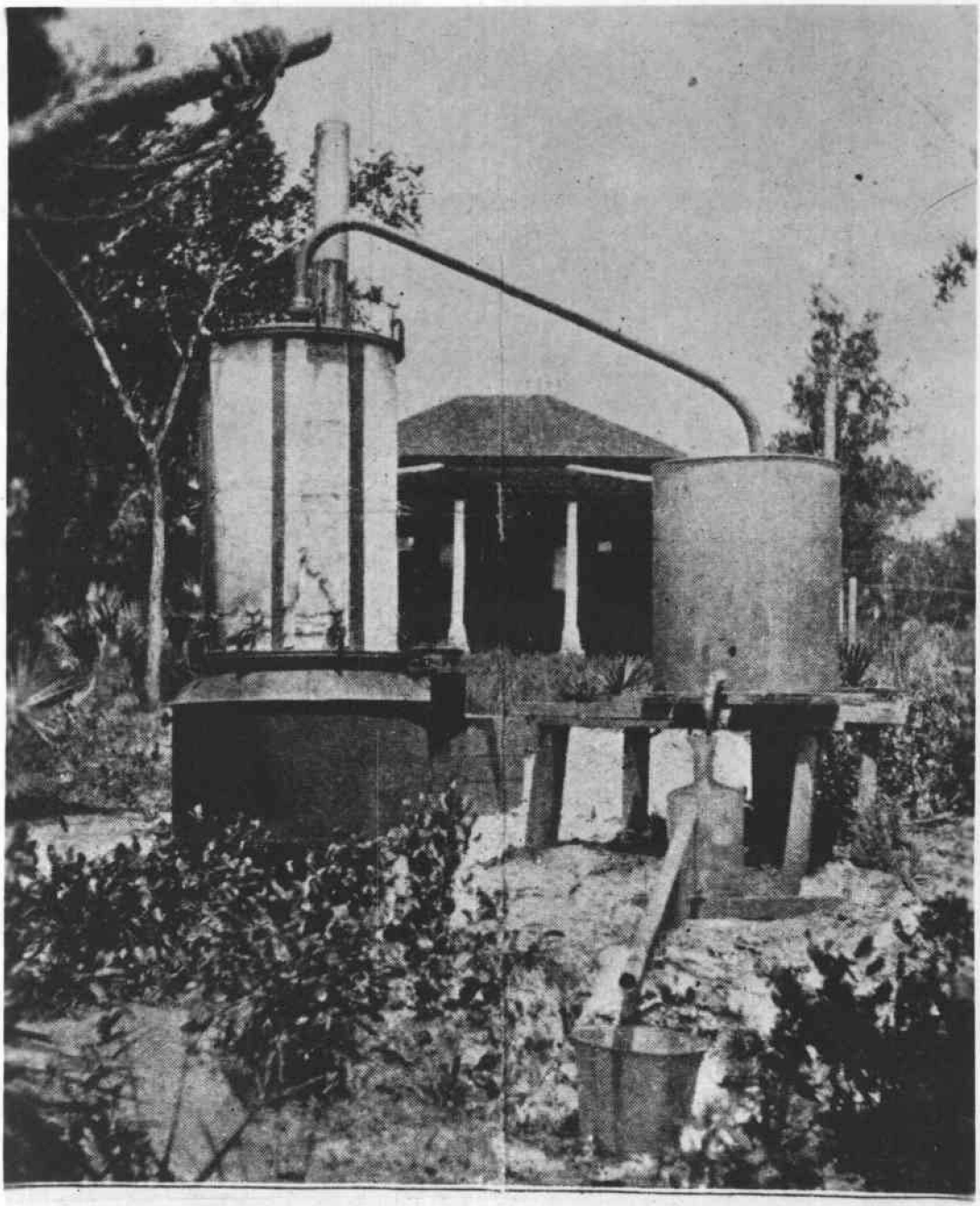

Fig. 1 - Still and Condenser

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1-1/2 feet internal diameter, placed in a galvanized iron tank 2 feet in diameter by 2-1/2 feet deep. The condenser was connected to the still with an 8-foot copper pipe, in two sections 2 inches in diameter. A 2-gallon aspirator bottle, having a brass syphon, served as a receiver.

The material to be distilled was first passed through a feed cutter, the needles and twigs being cut into lengths of 1/2 to 1 inch. When the chopped material was well packed into the still the charge varied from 350 to 500 pounds, depending upon the species. By filling the cylinder ahead of the rising column of steam, the needles are decidedly more compressible. The distillation was continued at the rate of 2-1/2 gallons per hour. At the end of 7 to 8 hours the quantity of oil fell to 5 to 6 cubic centimeters per hour, and the distillation was then considered complete. The oils are dried and filtered through cotton batting or fine muslin, and are then ready for market. Sometimes they are subsequently rectified by dealers.

The small distillers usually employ apparatus constructed partly of wood. The leaves are placed in rectangular or cylindrical wooden tanks while steam is introduced from a separate generator. The simple apparatus used by a New England distiller of cedar leaf oil will serve as a type of the stills frequently employed. His description is the following:

> A steam-tight box, 3 feet by 4 feet and 3-1/2 feet deep, with a boiler plate bottom is set on a rock furnace. Inside of this box is a grating 4 inches above the bottom to hold the cedar up to the top of the grating; a pipe from the top of the box 10 feet long carries off the steam. This steam pipe runs nearly its entire length through a trough of water kept cold by running water. The condensed steam drops into a glass jar covered with cloth for a strainer.