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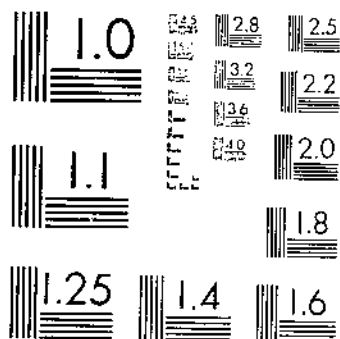
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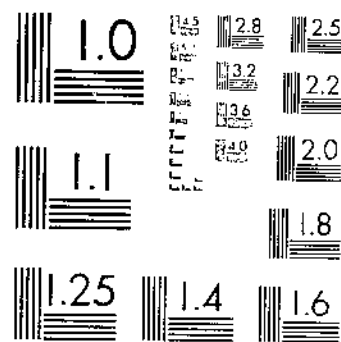
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METHODS OF EXTRACTING VOLATILE OILS FROM PLANT MATERIAL AND THE
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**UNITED STATES
DEPARTMENT OF AGRICULTURE
WASHINGTON, D. C.**

Methods of Extracting Volatile Oils From Plant Material and the Production of Such Oils in the United States

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INTRODUCTION

Volatile oils, or essential oils, as they are often called, are present in plants of many species. They may be removed from the plant material with a current of steam, frequently without any important change in composition. Although not all odorous plants owe their aroma to volatile oils, distinctly aromatic plants generally contain such oils.

Most volatile oils are complex mixtures of organic substances, and their value usually depends on the presence of one or more of these

constituents, which give them their characteristic properties. Some oils possess therapeutic properties and are used in medical practice. Others are useful in several ways, as, for example, oil of turpentine, which is a medicinal product, but is used mainly in the manufacture of paints. A great number of the commercially important volatile oils find their industrial application in providing an agreeable scent or flavor to perfumes, toilet articles, soaps, cosmetics, and confections.

Volatile oils are found in various parts of plants. For example, in the perfume roses the oil occurs only in the flowers; in the orange and lemon both the flowers and fruits contain oil but of different composition; in the mints the oil is present in the entire aerial portion; in sweet birch and cinnamon it is obtained from the bark; in valerian and calamus it is found only in the root. Sandalwood and cedarwood oils are examples of those obtained from the wood. The oil does not exist as such in some plants, but is developed through chemical reaction between certain constituents when the shredded or ground material is macerated with water. Wintergreen and bitter almond oils are examples.

METHODS OF EXTRACTION

In commercial practice volatile oils are removed from plant material by various methods, depending on the quantity of oil present, its commercial value, the stability of its aromatic constituents, and other factors. The tendency of some constituents to undergo changes when subjected to high temperatures calls for special methods of extraction whereby the final product is obtained without decomposition. Three methods are now used commercially: (1) Extraction by expression; (2) extraction by solution; (3) extraction by steam distillation.

EXTRACTION BY EXPRESSION

A small group of volatile oils, of which orange, lemon, and bergamot oils are the most important, may be obtained by expression. The oils, present in the cells of the rind of the fruit, are obtained by crushing or rupturing the cells and removing the oil by some suitable method. In southern Europe, where labor is plentiful, the oils were long obtained by absorption with small sponges. The fruit was cut in half, the peel was removed from the half section, turned inside out, thus rupturing most of the oil cells, and the oil was caught on and soaked up by a sponge held in the hand. Gradually machines that removed the oil from the peel with sponges were designed, but it was still largely a hand process. The oil obtained by hand or machine sponging is of high quality. Later, devices were introduced for handling the whole fruit mechanically for the production of the oil and the juice.

EXTRACTION BY SOLUTION

Extraction by solution involves the use of a substance that either dissolves or absorbs the aromatic constituents and the removal of the constituents from it by further treatment. Three modifications of this method are in use: (1) Extraction with cold solid fats; (2) extraction with hot liquid fats; (3) extraction with volatile solvents.

EXTRACTION WITH COLD SOLID FATS

The use of solid fats at ordinary temperatures for the extraction of perfume from flowers by absorption is known as the enfleurage process. It has been replaced to some extent by the more modern solvent-extraction method, but it is still used for the flowers of tuberose and jasmine. These flowers continue to produce valuable perfume for some time after they are picked and their oils are unfavorably affected by even moderate heat.

The enfleurage process requires so much hand labor that its use is necessarily limited to the production of flower oils of high market value. The equipment needed is very simple. It consists of many small wooden frames, each several inches high, about 16 inches wide, and slightly longer. A glass plate is fitted in the frame, and a layer of specially prepared fat is applied on both sides of the plate to absorb the odor from the flowers. This fat must be of the proper consistency, practically odorless, and of such composition that it will remain nonrancid for a long time under the conditions of use. A mixture of one part of purified tallow and two parts of lard has been widely used. When these frames are stacked one upon another, they provide many small, practically airtight compartments with a layer of fat on both the bottom and the top of the glass plate. The flowers, carefully cleaned and free from external moisture, are spread on the bottom layers of fat. The odorous constituents are absorbed by the fat on both top and bottom plate surfaces. After about 24 hours the flowers will have yielded most of their oil. They are carefully removed and a new charge of flowers is introduced. This procedure is continued throughout the harvesting period, after which the fat, saturated with the flower oils, is removed. This product is known as pomade; its value depends on the kind of flowers used and the degree of saturation.

Although such pomades may be used directly in the preparation of perfumes, the general practice is to extract the oils from the fat with high-proof alcohol in special containers in a series of manipulations that assure complete extraction of the oils and subsequent removal of any extracted fat by refrigeration. The "pomade extracts," which represent the true perfume of the flowers, may go into the trade as such or may be further processed by removal of the alcohol in a vacuum still at low temperature. The floral "absolutes" thus obtained may be purified by various means if the market value of the resulting product is high enough to justify the additional costs.

The flowers removed from the fat in the enfleurage process retain some odorous constituents that are not sufficiently volatile to be released from the flowers and absorbed by the fat. They can be dissolved with a suitable solvent to yield a useful product quite different from the pomade extracts and floral absolutes and of lower market value.

EXTRACTION WITH HOT LIQUID FATS

At one time widely used for extracting the oils from flowers other than tuberose and jasmine, extraction with hot liquid fats is now seldom employed commercially. It is cumbersome and the products obtained do not represent the true perfume of the flowers.

The flowers are immersed in a specially prepared fat. The mixture is heated to about 80° C. for about half an hour and then allowed to cool for an hour. It is finally reheated and then strained or filtered to remove the flowers. The proportion by weight of flowers to fat is about 1 to 4. New charges of flowers are introduced until the total weight of flowers immersed and macerated is about twice the weight of the fat solvent used. The perfume-saturated fat is sold as such, or an extract may be made from it with strong alcohol, as described on page 3.

EXTRACTION WITH VOLATILE SOLVENTS

The volatile-solvent method is used to extract oil from flowers only. Other parts of the plants would yield to the solvents large quantities of matter that could not be removed from the oil and that would detract from the delicate odor desired. This method involves the use of a process known as continuous-extraction. After passing through the material, the solvent is volatilized, condensed, and again passed through the material. The process is repeated until the entire charge is exhausted. At each passage of the solvent a portion of the volatile oil is dissolved and passes off with the solvent. The advantage of the method lies in the fact that only a relatively small quantity of the solvent has to be finally removed from the extracted oil. It requires special equipment, well designed and constructed of good materials.

Because of the delicate nature of the floral products extracted and the hazards involved in the use of highly flammable solvents, only skilled personnel can successfully operate such equipment.

Equipment of one type in commercial use consists of a cylindrical, upright extractor of copper or tinned sheet metal of about 300-gallon capacity, usually arranged in batteries of three, with the concentrator, condenser, and solvent storage tank. Each extractor holds about 350 pounds of flowers; approximately 50 gallons of solvent is required for extracting 100 pounds of the flowers. The extraction is carried out by subjecting each batch of flowers to at least three washings with the solvent. To begin the operation, the flowers in the first extractor are washed with a batch of solvent, which is then transferred directly to the concentrator. The flowers are given a second washing with a new batch of solvent, which is then pumped to the second extractor for the first wash and thence to the concentrator. A third batch of solvent used for the third washing of the flowers in the first extractor is used for the second washing in the second extractor and then as the first washing of a third batch of flowers in the third extractor, from which it goes to the concentrator. Similarly, a new batch of solvent is first used for the third washing in the second extractor, then for a second washing in the third extractor, on to a fourth batch of flowers as a first wash, and then to the concentrator. Thus the process continues with a minimum use of solvent. After the third washing has been drained off the adhering solvent is removed by blowing a current of steam through the flowers and into the condenser for recovery of the solvent. The flowers are then discarded.

The several washings are usually concentrated in the still or evaporator by blowing steam into a jacket beneath it until most of the

solvent is removed and recovered. The desired concentration is usually reached when the temperature in the evaporator is about 60° C. The extract is then transferred to a smaller vacuum still in which the rest of the solvent is completely removed under greatly reduced pressure. This operation requires skill and care so that all traces of the solvent are removed without damage to the delicate floral product.

The rotary extractor is also widely used. It consists of a heavily tinned iron cylindrical drum, revolving on a horizontal axle. Inside are four perforated, cylindrical, horizontal compartments, which are charged with the flowers through openings at the ends. Enough solvent is placed in the drum to fill it about halfway up to the axle level. As the drum is slowly rotated the flowers are dipped into the solvent, which passes into the compartments through the perforations. As the compartments rise above the solvent level the solvent drains back. This continues with each revolution of the drum. When the extraction is complete the extract is drained off and steam is passed into the drum to distill off the solvent adhering to the flowers. The extract is then concentrated in the same way as that obtained with equipment of the other type.

The rotary extractor has several advantages. The solvent is more effective in extracting the odorous constituents than in the stationary extractors and the yield is therefore greater; much less solvent is required; the solvent loss is lower; the equipment requires less floor space and is less expensive.

The concentrated extracts as obtained by either method, when cooled, are usually solids, owing to the plant waxes and other constituents present. They are known as flower concretes. As they contain all the odoriferous principals, they represent the true fragrance of the flowers.

The concretes may be used as such in the manufacture of perfumes, or they may be further processed by dissolving the odoriferous constituents in 95 percent alcohol, thus removing the insoluble waxes. The alcohol extract is concentrated in a vacuum still with great care to obtain the final product, known in the trade as flower-oil absolutes.

The nature and quality of the solvent used are of the greatest importance. Most satisfactory for the most expensive flowers is a high-grade petroleum ether, obtained by careful fractionation of gasoline. It volatilizes completely below 75° C., leaving no perceptible residue or odor. Benzene, highly purified so that it will completely volatilize at about 80°, may be used for extracting less valuable plant materials if the presence of some coloring matter extracted by it is not objectionable.

The foregoing discussion of solvent-extraction methods and equipment is intended only to provide a general understanding of the subject. The detailed information necessary to make use of such methods even on a small scale and to select the proper equipment must be obtained from experienced operators of the methods or from illustrated publications on the subject.¹

¹ GUENTHER, D. THE ESSENTIAL OILS (VOL. 1): HISTORY-ORIGIN IN PLANTS-PRODUCTION-ANALYSIS. 427 pp., illus. New York, 1948.

EXTRACTION BY STEAM DISTILLATION

The simplest and most economical method of removing volatile oils from plant material is by distillation with a current of steam. This method cannot be used for flowers having odors that are unfavorably affected by the action of steam. Most volatile oils, however, can be distilled by steam without serious decomposition. The chief advantages of the method are its simplicity, the comparatively brief time required for its operation, and the fact that large quantities of material can be handled at a small cost. It is the only method economically possible for the extraction of the great number of volatile oils of only nominal value, for which the more tedious processes would be impracticable.

The steam-distillation method is based on the facts that volatile oils are vaporized when the material containing them is subjected to a current of steam and that, when the mixture of oil and water vapors is condensed, the oil separates as a liquid in a layer that may be readily removed from the water. To accomplish this result it is necessary to supply (1) a tub or retort in which the plant material may be subjected to the action of steam obtained from any convenient source, (2) a suitable condenser for condensing the mixture of vapors, and (3) a receiver in which the condensed water and oil may be collected.

DESIGN AND CONSTRUCTION OF VOLATILE-OIL STILL²

The details of design and construction of volatile-oil stills vary somewhat according to the use to which the still is to be put. The capacity of the still, the availability of materials for construction, and the acquaintance of the builder with the latest approved details also determine the several modifications of the apparatus in commercial use.

The volatile-oil stills used by mint growers in the United States for the production of peppermint and spearmint oils are of the type required for any large-scale production of volatile oils from plants when the aerial portions or roots are used. Various modifications are possible with respect to capacity, source of steam, method of operation, and other features to fit the particular needs and circumstances. To determine the market possibilities of a volatile oil not already in commercial use a small still that will hold a few hundred pounds of material can be used to advantage. Such a still may have the same general design as a commercial still, but it will cost less.

A distilling unit of the design and capacity widely used by mint growers consists of a high-pressure boiler³ for generating steam, a tub,⁴ a condenser, and a receiver. The general arrangement of these parts may vary according to conditions, but the whole equipment should be so assembled that the outfit can be installed at the lowest

² All stills, regardless of their purpose, must be registered with the U. S. Bureau of Internal Revenue. Applications for the required blank forms should be made to the United States Collector of Internal Revenue of the district in which the still is to be operated.

³ In most States the laws require periodic inspection of steam boilers of the size and type used for distilling mint.

⁴ Several terms, as "tub," "vat," and "retort," are commonly used to designate that part of the distilling equipment in which the herb is packed.

cost and operated with the greatest saving of labor. Even the smallest distilling units generally include two tubs operated with one condenser, so that one tub can be charged while the other is in operation or is being discharged. For larger operations four or more tubs are usually operated in pairs, with a condenser for each pair. Sometimes a single large condenser is adequate for as many as four tubs.

TUBS

Because it takes less time to discharge and reload a tub than to complete the distillation, a more efficient arrangement in a multitub unit is to equip each tub with its own condenser. This makes it possible to keep the tubs in more nearly continuous operation and to eliminate some heavy iron pipes and valves that at times cause discoloration of the oil. Such a still is shown in figure 1.

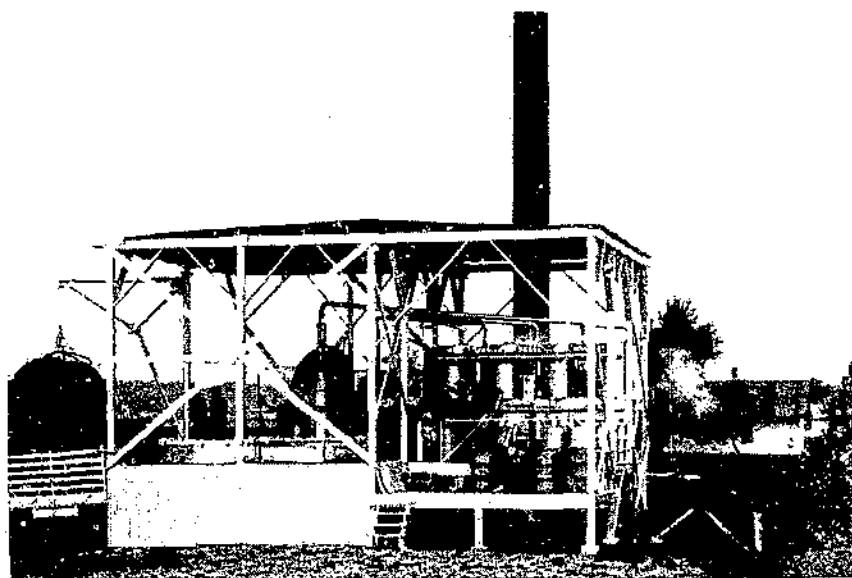


FIGURE 1.—Modern mint still, with a condenser for each tub. The herb is placed in a steaming tub (lower left), from which oil and water vapor pass to a condenser (upper right). The oil and water flow in liquid form to a receiver (oil separator), from which the water is drawn off as necessary and the oil is made to pass into a drum (lower right).

The tubs are set down part way in a platform, which serves as a working floor, the tubs projecting usually about 2 feet above this floor. A lifting windlass on a crane or track is mounted in such a position that it can be used for loading and discharging each tub.

The tubs in use vary somewhat in size. They are 6 to 9 feet deep and 6 to 7 feet in diameter. Some are made slightly larger at the top than at the bottom to facilitate removal of the spent herb. Most of them are made of 16-gage galvanized steel (fig. 2). The steamtight gasket consists of a flat strip of composition material riveted to the rim of the tub or to the under edge of the cover, which is fastened down with adjustable eccentric clamps.

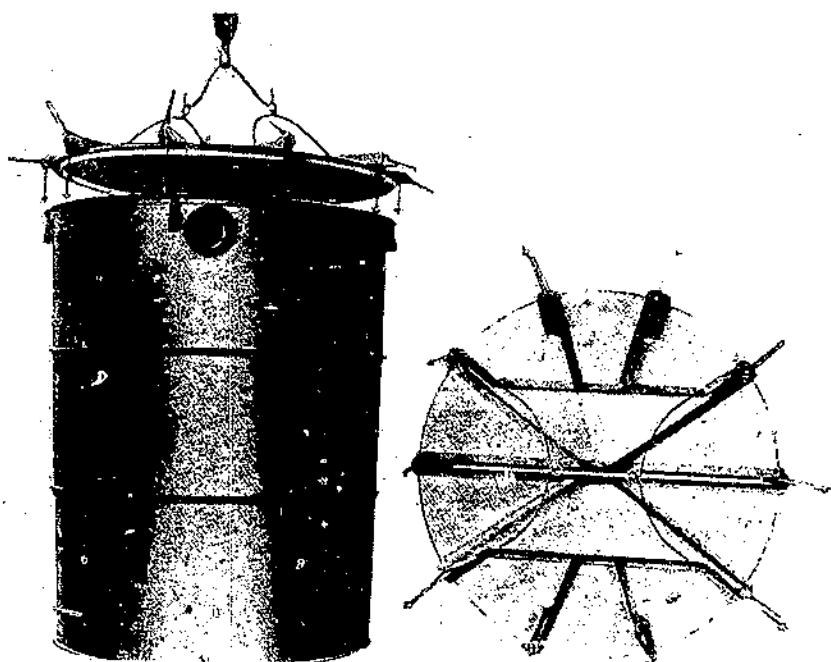


FIGURE 2.—Galvanized-steel tub.

The steam is admitted from a $1\frac{1}{2}$ -inch pipe just above the bottom of the tub. An even distribution of steam through the charge is effected by means of a T with open ends. The outlet for the steam and oil vapors is through a pipe from the side just below the cover. This pipe has a diameter several times larger than that of the inlet pipe, to prevent pressure from building up in the tub. It usually extends upward and then passes overhead to the condenser (fig. 1).

CONDENSERS

The worm-type drip condenser has been in use for many years in one form or another. One type consists of six or eight horizontal sheet-metal pipes joined at the ends by elbows to form a continuous series (fig. 3). The first two lengths of pipe from the top are 7 or 8 inches in diameter; the rest are reduced successively in size, the last one, from which the condensed oil and water flow, being 2 or $2\frac{1}{2}$ inches in diameter.

Condensation is obtained by water flowing over the pipe from a perforated trough mounted directly above the condenser. This water drains into a lead-off trough at the bottom. On some condensers a lead-off trough is mounted below the third pipe, with a second perforated trough immediately below to furnish a fresh supply of cold water to the remaining pipes. The water flows to the troughs by gravity from a reservoir overhead. A steady and ample supply of water is drawn by pumps either from wells or from small streams in the vicinity of the still. The hot water that drips from the condenser is frequently used in the boiler, thus saving fuel. At some convenient

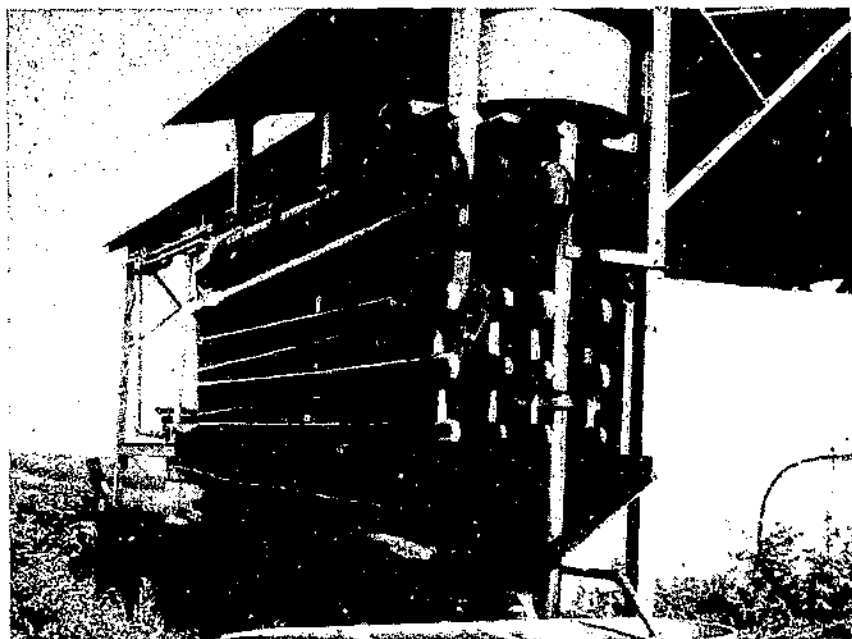


FIGURE 3.—Four worm-type drip condensers.

point in the upper part of the condenser is an air vent that can be opened when the steam is shut off. The air entering through it will prevent the condenser from collapsing, which otherwise is likely to occur because air cannot enter fast enough through the small end of the condenser.

The type of condenser described came into extensive use mainly because it could be constructed at a small cost, but it is soon destroyed by rust. Built of copper pipe lined with tin, it will last many years.

On mint farms the drip-type worm condenser has recently been replaced largely by a tank type, which consists of a worm arrangement similar to that used with the drip type, but having the worm enclosed in a large tank. Water enters the tank continuously, so that the pipes are bathed constantly in cool water. For large operations tubular condensers are sometimes used. Such a condenser consists of an upright galvanized-steel shell in which are mounted numerous upright galvanized-iron pipes of small diameter, somewhat like the flues in a boiler (fig. 4). As the vapors pass down through the pipes they are condensed by the cold water that circulates around the pipes; the condensed water and oil then flow from a narrow outlet into the receiver. Though rather expensive, such a condenser lasts a long time, requires little space, and is very efficient.

OIL RECEIVERS

The receivers in which the oil is collected are of simple design and construction. They are cylindrical, made of galvanized iron, and vary in capacity from a few to 50 gallons, depending on the capacity of the still. Most oils are lighter than water. When they are distilled

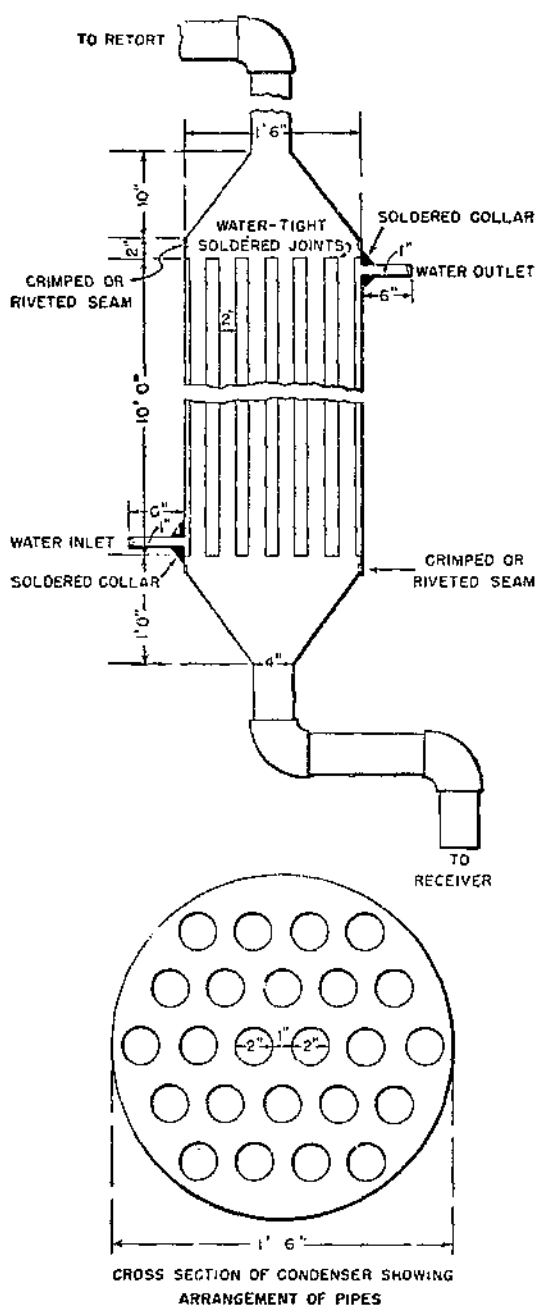


FIGURE 4.—Details of a tubular condenser.

the water is drawn off the bottom by means of a pipe that extends up along the side of the receiver to within a few inches of the top, where it is fitted with an elbow and a short extension pipe. As distillation proceeds the surface of the oil in the receiver is kept at the desired level by raising or lowering the end of the extension pipe from which the water drains. At a point near the top of the receiver is an outlet through which the oil can be drawn off (fig. 5).

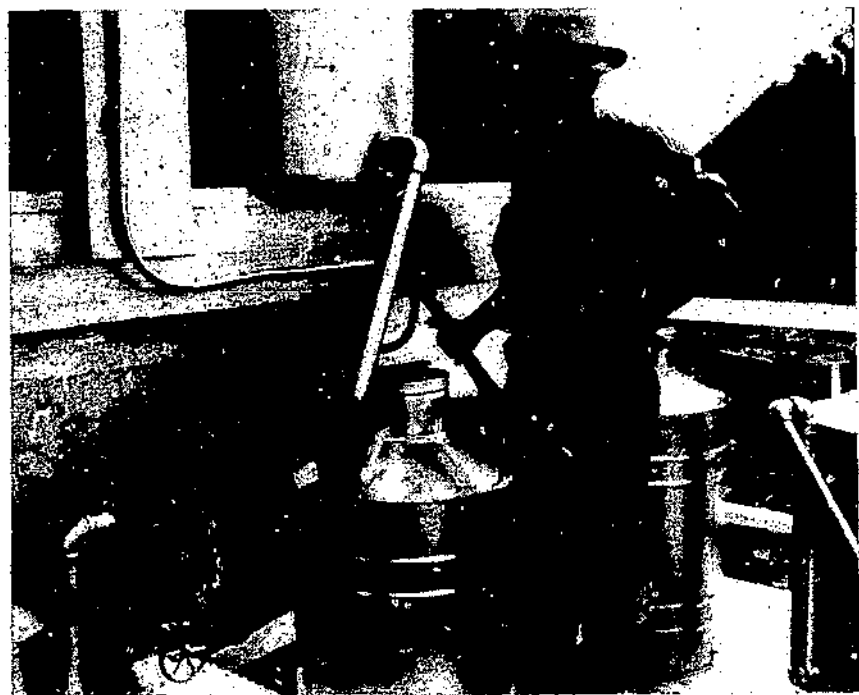


FIGURE 5.—Removing oil from the receiver. The operator has brought the oil level up to the discharge spout by raising the extension pipe of the water outlet.

A few oils, such as sassafras and wintergreen, are heavier than water and will separate in a layer on the bottom of the receiver. When distilling such oils, the gooseneck drainpipe is closed and a funnel of suitable size is set in the receiver with its stem extending into the water to about two-thirds of the distance to the bottom. As the mixture of water and oil runs into the funnel, the oil globules continue down through the stem and settle on the bottom of the receiver. From here the oil may be drawn off through a stopcock. The clear water is drained off through the outlet at the top used for drawing off oils lighter than water. This outlet should be of sufficient size to permit the water to drain off as fast as it flows from the condenser into the receiver.

Several devices are used to prevent churning of the contents of the receiver by the flow from the condenser, which might cause some of the oil to be drawn off with the water. In the case of oils lighter than water one method is to direct the flow into a funnel-topped pipe

that extends about half the distance to the bottom of the receiver, where it is fitted with a short elbow that directs the oil toward the surface. Another method is to admit the flow from the condenser into the receiver through a pipe at a point about two-thirds the way down, a baffle plate immediately below the inlet directing the separated drops of oil toward the surface.

OTHER TYPES OF EQUIPMENT

Some of the volatile oils produced in the United States on a small scale, especially those obtained from wild plants, are distilled with relatively crude equipment, often hand-made in part on the farm. The tubs are sometimes made of wooden staves, and the vapors are condensed by being passed through a long length of iron pipe laid along the ground or a series of such pipes in a wooden trough supplied with water from a small dammed-up stream. Heavy metal tubs are frequently used, in which case water is introduced with the plant material and steam is generated by means of a fire underneath. This eliminates the need for a boiler, thus reducing the cost of the equipment, although the time required to exhaust the charge is much greater.

Where the plants distilled grow wild it is usually necessary to move the stills from place to place as the plant supply is exhausted. Such itinerant stills are usually of the simplest types. They are probably less efficient than the commercial stills used for widely cultivated volatile-oil crops, but appear to serve best the purpose for which they are designed.

OPERATION OF VOLATILE-OIL STILL

PREPARATION OF MATERIAL FOR DISTILLATION

Material to be distilled, no matter of what nature, should be in a condition that insures the removal of the oil in the shortest time, due consideration being given to the cost of bringing it into that condition. Bulky material that cannot be readily packed and hence occupies much space should first be ground or chopped, in which condition it can be loaded into the tub more advantageously and the removal of the oil from it hastened. Green and succulent material may frequently be prepared by passing it through a feed cutter; heavy woody material must be chopped either by hand or with hogging machines. In the distillation of pine stumps and sassafras roots, as carried on in the Southeastern States, use is made of these machines. Certain seeds, such as anise, caraway, and coriander, are usually crushed, thus greatly facilitating the removal of the oil. Herbaceous material frequently is dried, either partially or completely, before distillation to obviate the handling of the unnecessary weight due to the large water content of the material. Such partial or complete drying also reduces the time required to exhaust the charge, as well as the volume of steam needed. An example of the practice is the partial drying of mint herb where large acreages are harvested. The wilted herb is referred to as mint hay; in that condition it is convenient to handle but is not dry enough for the leaves to break off and get lost in handling. Herbaceous material that is completely dried usually can be distilled without loss

of oil, provided none of the principal oil-bearing portions are lost while being handled in that condition. Some volatile oils, however, suffer serious deterioration if the herbs containing them are allowed to dry before the oil is distilled.

Some plants from which volatile oils may be distilled do not contain the oil as such, but contain certain basal substances, which, under proper conditions, react with one another to form the oil. The oil may then be removed by the usual process. Examples of such oils are wintergreen, sweet birch, bitter almond, and mustard. The chemical reaction that results in the formation of these oils is readily induced by simply macerating the material or soaking the ground or chopped material with water for a suitable period, after which distillation may proceed. The material is usually macerated in the tub of the still, so that no transfer of the mixture is necessary.

The bulk of the oil usually distills over in the early period of the operation. Small quantities continue to collect for some time, depending on the manipulation of the steam current and the nature of the plant material. In most cases the distillation is complete in less than 2 hours; and in large-scale operations in less than an hour. To determine whether all the oil has been distilled over, when the operator is not experienced in the distilling of any particular material, a small quantity of the distillate from the condenser may be collected in a glass cylinder or a large glass test tube. If any small globules of oil separate, the distillation has not been completed. Whether or not it is advisable to continue the process depends on several considerations. If the oil is high-priced, it is well to continue the distillation further; but if it is of only moderate value and an abundance of plant material is available, it is more economical to shorten the process and thus increase the quantity of material that may be handled in a given time.

DISPOSAL OF SPENT MATERIAL

After the charge has been exhausted, the top of the still may be taken off immediately and the spent material removed in any convenient manner. The practice of loading and discharging the still by means of either a derrick or a block and tackle greatly reduces the labor and saves much time. The spent material is often returned to the field as fertilizer; sometimes it may be used for fuel. In the commercial distillation of peppermint the spent herb is dried and used for stock fodder. Economies of this nature reduce production costs.

CARE AND HANDLING OF OIL

The oil as removed from the receiver contains water, and a layer of emulsion consisting of oil and water is usually present also. It is advisable to let the oil stand in this condition in a suitable container long enough to permit the emulsion to break. Sometimes this takes place rapidly; again, it proceeds very slowly. Much depends on the character of the oil and the amount of churning that took place in the receiver during distillation. If a separatory funnel is available, the last portions of water and the emulsion may be drawn off from the oil and allowed to stand until the emulsion has broken, when the separated portions of the oil can be removed. If on standing the emulsion does not separate readily, transferring it to a funnel

containing a folded filter paper may bring it about. On passing through the paper the emulsion is broken up and the oil and water collect in separate layers. The apparatus required for collecting, separating, and filtering the oils when small quantities are being prepared is shown in figure 6.

The presence of moisture in the oils makes them cloudy and detracts from their appearance. Moisture, if not removed, will hasten chemical changes in the oils, especially in the presence of air. This may appreciably affect their value. It is desirable therefore that the oils be dried as soon as possible by filtering through filter paper in an ordinary funnel. Filter paper for this purpose may usually be



FIGURE 6.—Apparatus for collecting, separating, and filtering volatile oils, for use with small experimental stills.

purchased in drug stores. Large quantities are usually filtered through chamois.

Filtering gives the oil a clear, bright appearance, which is a distinct asset when it is placed on the market. It does not, however, remove all traces of moisture. If the oil is to be kept any length of time and is of such a character that its value is affected by slight changes in composition, it must first be treated with a substance that will absorb this moisture. Dry sodium sulfate has a strong affinity for water, and the addition of small quantities, with occasional shaking, is sufficient to remove all final traces of moisture. The oil may then be filtered and placed in bottles or cans with only slight danger of any change in the composition for some time. It is advisable to fill the containers completely in order to exclude the air. The best bottles for the purpose are made of amber-colored glass; those of plain glass should be kept in a dark cabinet to prevent deterioration of the oil through the action of light.

In steam distillation traces of other volatile substances are sometimes distilled over with the oil, imparting an undesirable odor, which must be removed before the oil can be used. This removal is best accomplished by submitting the oil to a second distillation in an apparatus of the same type but smaller. By this procedure a small portion of heavy, highly colored oil is usually left behind in the still, while the portion distilled over is greatly improved in odor and color. An oil thus treated usually commands a higher price than the crude oil. Oils may also be improved in quality by redistilling them by direct heat under greatly reduced atmospheric pressure in specially designed apparatus. By this method certain undesirable fractions are eliminated. Oils that have been subjected to a process of redistillation are known as rectified oils.

Small distillers do not generally rectify their oil. Large producers who wish to meet a special demand for a superior product or who are themselves users of the oils, however, frequently rectify theirs. Oils that must conform to legal standards or that are used for the manufacture of products standardized by long practice to a definite flavor or odor must be handled with special care by the producer if a steady market is to be found.

COMMERCIAL PRODUCTION OF VOLATILE OILS IN THE UNITED STATES

Of the large number of volatile oils consumed in the industries, only a few are produced commercially in the United States. For various reasons the domestic production on the whole has not increased in variety, nor does it appear probable that there will be any appreciable increase in the near future. New sources of valuable oils may be developed to augment the total production, but this no doubt will be offset by the declining production of others, especially those obtained from natural sources that gradually become exhausted. The relatively high cost of labor in the United States has restricted this industry to oils that can be produced by a minimum expenditure of labor. In fact, the relatively high cost of labor acts as a serious bar to the production of a great number of valuable oils from plants that could be grown under our conditions of soil and climate.

The principal commercial oils produced in this country⁵ may conveniently be placed in three groups: (1) Those obtained from wild plants; (2) those obtained from plants grown commercially for the purpose; and (3) those obtained as byproducts in other industries.

OILS OBTAINED FROM WILD PLANTS

The wild plants from which oils are produced include trees, shrubs, or herbs growing without cultivation over wide areas and in sufficient abundance to make their collection and the distillation of their odorous parts economically possible. Most of them are produced over scattered areas, the production centers being shifted frequently as the supply of material becomes exhausted and new fields are entered. The methods of distillation are frequently crude, and, as much of the production has been by many small operators, the distillation points are often widely scattered and introduction of the more modern methods has been retarded.

The largest production of any one volatile oil in the United States is that of oil of turpentine, which is obtained by the distillation of turpentine from the oleoresin of several species of *Pinus* that grow over the southeastern Coastal Plain and in the Gulf Coast States. This industry, including also the production of rosin and the distillation of pine oil from pine stumps, generally referred to as the naval-stores industry, is distinct from the volatile-oil industry as a whole. Since the naval-stores industry is described in detail in other literature, it will receive no further attention here.⁶

CEDARLEAF

Cedarleaf oil is obtained from the leaves and small branches of the northern white-cedar (*Thuja occidentalis* L.). It is produced in northern New York, Vermont, and, to a less extent, in New Hampshire and Maine by farmers when they are not occupied by routine farm work. With few exceptions the distillations are conducted with rather crude equipment, mostly constructed from materials locally available and at small cost. Most stills are made of spruce planking, tongued and grooved. The seams are calked with clay or other suitable material. Some producers use steel tanks. Steam is admitted into the tub directly from an outside source, usually an old sawmill or hoisting engine. The stills are set up near springs or streams that provide ample water for the condenser through gravity flow or by means of pumps.

⁵ It is not the purpose in this bulletin to discuss oils distilled in the United States from imported raw materials, but only those obtained from materials produced here.

⁶ The Department of Agriculture has issued the following bulletins on this subject, which are no longer available but can perhaps be consulted in libraries:

SCHROCK, A. W., and BETTS, H. S. THE NAVAL STORES INDUSTRY. U. S. Dept. Agr. Dept. Bul. 229, 58 pp., illus. 1915.

VEITCH, F. P., and DONK, M. G. WOOD TURPENTINE, ITS PRODUCTION, REFINING, PROPERTIES, AND USES. U. S. Dept. Agr., Bur. Chem. Bul. 144, 76 pp., illus. 1911.

VEITCH, F. P., and GROTLISCH, V. E. TURPENTINE: ITS SOURCES, PROPERTIES, USES, TRANSPORTATION, AND MARKETING, WITH RECOMMENDED SPECIFICATIONS. U. S. Dept. Agr. Dept. Bul. 898, 53 pp., illus. 1920.

The most desirable material for distillation consists of the leaves and small branches removed from trees about 5 feet high. It is cut up and packed tightly in the stills. Such material is reported to yield from 1 to 1.5 percent of oil.

The equipment is moved from time to time, sometimes several times within a season. This is necessary to avoid long-distance hauling of the brush. The small trees are brought to the still, where the usable material is trimmed off. The heavy wood furnishes fuel for the boiler. The spent material after drying is also used for fuel. It takes about 5 years for new growth to reach the desired size.

Procuring the material for the stills involves hard labor, often under severe weather conditions. Considering the labor involved, the returns are relatively small, but they provide additional income without undue interference with the usual farm operations.

The oil is used for general scenting purposes. It is commonly brought by the producers to local storekeepers or to collectors in a central location, where it is cleaned by removal of dirt and water before entering the market. Some producers, however, sell directly to large dealers in essential oils.

ERIGERON

Erigeron (*Erigeron canadensis* L.), generally called horseweed, is a very common weed throughout the farming sections of the northern half of the United States. It develops rapidly in old meadows and abandoned grainfields and in a few years will grow so thick and tall that it has all the appearances of a cultivated crop. On old abandoned mint fields, or on fields where unfavorable spring weather has prevented the planting of corn and other crops, horseweed promptly takes possession. It contains a volatile oil that is used commercially to a limited extent, largely in the preparation of pharmaceutical products. Mint growers frequently cut the weed and distill it as a side line to their other oil business, especially when a well-developed field is available. The crop is cut with a grain binder; when the stand is good, from 25 to 30 pounds of oil an acre is obtained. The quantity of oil produced varies greatly from year to year. The consumption has declined to an estimated 2,000 pounds annually.

SASSAFRAS

The common sassafras (*Sassafras albidum* (Nutt.) Nees), widespread and abundant on wastelands in the eastern third of the country, is the source of sassafras oil. The oil is widely used as a flavor in carbonated beverages and dentifrices and for its medicinal properties in some pharmaceutical preparations. It is produced mainly in Kentucky, Tennessee, North Carolina, Virginia, and southern Indiana and Ohio. All parts of the sassafras tree contain the oil in varying quantities, but only the roots and stumps are used. Some of the oil is distilled in small, rather primitive stills that are probably moved from one place to another as new supplies of the needed material must be found. There are several larger operators with more modern stills.

The raw materials for them is obtained either from farmers, who procure it from their land and deliver it to the still, or through the

distiller's collection crews, who are provided with the necessary equipment. The trees are felled and the stumps pulled out of the ground. The stumps are cleaned of adhering dirt and hauled to the distillery, where they are run through a hogging machine and reduced to small chips. The chips are stored above the still, into which they are loaded as needed. The bark of the root contains from 5 to 9 percent oil, whereas the wood contains less than 1 percent. When the sap is flowing in the spring or when the ground is frozen, much of the bark will strip from the roots and remain in the ground when the stumps are pulled. It is the practice, therefore, to engage in this operation in late summer or fall. Distillation is conducted in the usual way. Steam is admitted from a boiler and blown through the chips in the still. The operation is completed in about 4 hours. The yield of oil varies from 1.5 to 2 percent, depending on the proportion of wood and bark in the charge.

SWEET BIRCH AND WINTERGREEN

Two native plants—sweet birch and wintergreen—yield volatile oils of identical flavor. These oils consist of about 99 percent methyl salicylate, to which their therapeutic properties are due. Methyl salicylate can be made cheaply synthetically, and, as it has the same medicinal properties as the natural oils, the United States Pharmacopoeia permits its use for medicinal purposes, provided it is labeled accordingly. However, the oils have certain flavor characteristics not possessed by methyl salicylate. Therefore they are in demand as flavoring agents in carbonated beverages, chewing gum, and dentifrices. For their therapeutic value they may be used in liniments and ointments, but for that purpose they probably are not superior to methyl salicylate. Sweet birch and wintergreen oils do not exist in the plants as such; they are formed when plant enzymes act on certain glycosides present in the plants. To bring about this reaction the plant material is chopped or crushed and then macerated in warm water in the still, generally overnight, before the usual distillation is started.

The sweet birch, or black birch (*Betula lenta* L.), is rather widely distributed from New England to Tennessee and Florida. Production of the oil is a very limited industry, chiefly in north-central and eastern Pennsylvania, in the Connecticut Valley, and in the southern Appalachian region of Tennessee, North Carolina, and South Carolina. The trade recognizes two grades of the oil—northern oil produced mainly in Pennsylvania and farther north, and southern oil produced farther south. A superior fragrance, claimed for the northern oil, is reflected in the market values of the two grades. No botanical differences can be discerned between the trees in the two regions, and the physical and chemical characteristics of the oils from the two regions do not provide a basis for this distinction by the trade.

Production of birch oil in five north-central Pennsylvania counties is largely a winter activity of farmers, who thus utilize their otherwise slack period to add to their income. The task of cutting, hauling, and trimming the branches is less laborious in winter because it is not hampered by the leaves that contain no oil. It is impractical and uneconomical to haul the material more than a few miles; hence,

many stills are moved from place to place. Several years are required for the new growth on the cut trees to reach the desired size.

The stills, which have a capacity of about 200 cubic feet and hold from 1 to 1½ tons, are constructed of heavy planks made as leakproof as possible. The bottom is faced with heavy sheet copper so that a fire may be maintained directly under it. The stills are firmly packed full, with the small material at the bottom.

If the maceration period is at night, distillation is conducted through most of the following day. The yield approximates 0.5 percent of oil. The oils are heavier than water and therefore settle to the bottom of the receivers, which must be designed accordingly. The water that flows from the receivers during the early period of the distillation holds considerable oil in suspension; this is generally returned to the still with the next charge.

Production of the so-called southern oil is no doubt accomplished in much the same manner. The distilling equipment is of simple and inexpensive design, and stills are moved from place to place to be accessible to the material needed.

Wintergreen (*Gaultheria procumbens* L.) is a small, low-growing, perennial evergreen herb usually found in cool, damp places in woods, most abundantly in the mountains of the Eastern States. Production of the oil is centered in Carbon and Luzerne Counties in Pennsylvania. Collection of the herb is slow and difficult. The plants are usually partly covered with fallen leaves that must be raked off, after which the leaves and small stems are pulled off by hand and placed in sacks. Much of the work is under low-growing trees. The distillation is usually done in summer, when the plant contains the most oil and children in the family can help collect it.

The oil is obtained by maceration and distillation with equipment like that used for birch oil, only smaller. Most of the stills are of simple, home construction, but several are of more advanced design. The time required to exhaust a charge is reported to range up to 12 hours. The yield of oil depends on the season of the year, the proportion of leaves and stems, and the completeness of the chemical reaction during the maceration. The average is about 0.5 percent. The annual production of the oil has decreased steadily for years; at present only a few people are engaged in it.

WITCH-HAZEL

The witch-hazel shrub (*Hamamelis virginiana* L.) contains a volatile oil that has long been considered the therapeutic agent in witch-hazel extract used in external medicine. The oil, however, is not produced as such. The product obtained in Connecticut from the witch-hazel shrub is the aqueous distillate resulting from steam distillation of the young branches. No oil separates during the distillation. To each 1,000 cc. of distillate obtained 150 cc. of alcohol is added. It is an official product of the National Formulary VIII in which it is described under the names "Hamamelis water," "witch-hazel water," and "distilled witch-hazel extract."

There are no farmer producers operating crude equipment. The industry in south-central Connecticut is limited to the operations of a few producers with highly developed modern copper stills, using selected

raw materials. It is reported that generally 50 gallons of filtered aqueous extract is obtained from 1,000 pounds of brush. To this the alcohol is added, and the product is then stored in oak barrels that are paraffined on the inside. There are no statistics on the quantity of the extract produced.

OILS OBTAINED FROM CULTIVATED PLANTS

The production of volatile oils, other than turpentine, from wild plants is of comparatively small magnitude in the United States, as compared with the production from plants cultivated for the purpose, although the number of varieties of cultivated volatile-oil plants is small. By far the most important of the volatile-oil plants grown are peppermint and spearmint. Peppermint is grown mainly in Indiana and Michigan in the Midwest and in Oregon and Washington on the Pacific coast. Spearmint is an important crop on many peppermint farms in the Midwest. In 1950 the total acreage of peppermint and spearmint was reported to be 61,800 acres, of which 42,100 acres were in the Midwest and 19,700 acres on the Pacific coast. In the same year 1,622,000 pounds of peppermint oil and 478,000 pounds of spearmint oil were produced. Detailed information on mint farming is available.⁷

DILL

Dill oil, obtained from the herb *Anethum graveolens* L., has come into use as a substitute for the herb in the flavoring of pickles and other food products. It was first produced in the North Central States about 1930 in response to the demands of pickle and kraut manufacturers. Ohio, Indiana, and Michigan were the principal centers of production, which later shifted to Oregon and Idaho. For some time the center of production was in the vicinity of Berne, Ind., where up to 500 acres of dill was grown annually and six or seven stills were in operation. At that time the growers received \$4 to \$5 a pound under contract. A decline in price resulted in a gradual reduction in acreage until only about 5 percent of the former acreage was being grown there several years ago. Information on the acreage in the Northwest is not generally available, but at present the principal production of the crop is apparently in the Willamette Valley in Oregon.

In Indiana dill is sown, early in spring, directly in the field with a beet or bean drill, in rows about 22 inches apart. The crop is ready to harvest in 90 to 105 days. It is harvested with a grain binder, usually about the middle of July, when the earliest seed has ripened. The herb is allowed to cure in the field for a day or two and then distilled with the equipment used in distilling mint. It takes from 2½ to 3 hours to exhaust the charge. The yield ranges from 15 to 50 pounds an acre. At times a second crop is obtained the same year, but the yield is small and usually unprofitable. There are several varieties of dill, some of which are not suitable for the purpose because they yield less oil or oil of poor quality.

The herb used for flavoring consists of the leaf, stem, and seed, the seed in various stages of maturity. To be a satisfactory substitute for the herb dill oil must have the same flavor. As the leaf oil and seed

⁷ SIEVERS, A. F., and STEVENSON, E. C. MINT FARMING. U. S. Dept. Agr. Farmers' Bul. 1988, 30 pp., illus. 1948.

oil are quite different, it is essential that the oil be distilled from the herb harvested at the stage at which it is used for pickling.

JAPANESE MINT

A variety of mint (*Mentha arvensis* var. *piperasens* Malinvaud) that for years was grown almost exclusively in Japan—hence called Japanese mint—is the only commercial source of natural menthol in normal times. During World War II this mint was grown extensively in Brazil. It remains to be seen which of the two countries will become the principal supplier of menthol.

This variety of mint is closely related to the peppermint grown in the United States, but it produces an oil with up to 80 percent of menthol, which makes it a much better source of menthol than the American peppermint oil with only 50 percent. The species was grown successfully in California about 25 years ago, when the high price of menthol made the crop profitable. During World War II, when menthol was once more of high market value, it was again introduced into that State in the general vicinity of Shafter. However, interest in the crop could not be sustained when greatly increased Brazilian production of menthol and the end of the war clearly pointed to an early decline in the price of the product.

The Japanese mint is less hardy than American mint and is therefore not well adapted to the mint-growing sections of the North Central States. Experimental plantings in many parts of the country have shown that this menthol content of the oil is generally highest when the crop is grown in the Northern States and in California. Japanese mint grows vigorously under irrigation in California and produces two harvests a year. The yields of oil there average 60 to 70 pounds an acre and are larger than elsewhere. As the oil contains about 80 percent of menthol, the crop is undoubtedly best adapted to that State. The higher returns more than offset the higher cost of production.

The crop is grown and distilled like American mint. Extraction of the menthol from the oil is accomplished by refrigeration, which causes the menthol to crystallize. The oil is separated from crystals with a centrifuge and again refrigerated, the process being repeated several times. The dementholized oil is poor in flavor and not generally acceptable for the purposes for which American peppermint oil is used. The Food, Drug, and Cosmetic Act requires that goods flavored with Japanese mint oil, or the dementholized oil, be labeled "flavored with corn mint." It is not practical for the average grower of this mint to undertake extraction of the menthol. The general practice has been for growers to sell the oil under contract to the principal consumers of menthol or to dealers in oils who have the facilities for economic separation of the menthol.

LEMON GRASS

The commercial oil of lemon grass is obtained from one or more species of *Cymbopogon*. Only one attempt has been made to grow this tropical plant in the United States. About 1940 the United States Sugar Corp. began its cultivation on Everglades land in southern Florida. Lemon grass was believed to have commercial possibilities because the grass from which the oil had been removed could be

combined with low-grade molasses from the sugar factory to make a stock feed. The growing, harvesting, and distillation were conducted in the most economical way, and in the course of several years the plantings increased to 1,000 acres annually. However, greatly fluctuating market prices of the oil and steadily mounting labor costs forced the abandonment of the undertaking in 1947.

Lemon grass oil is widely used for scenting soaps and cosmetics, and is the principal source of several products important in the manufacture of perfumes.

TANSY

Tansy (*Tanacetum vulgare* L.) is a minor crop on some mint farms. Buyers of tansy oil report that the average annual production is not more than 1,000 pounds. This amount is obtained from about 100 acres in southwestern Michigan and northern Indiana. In 1946 about 60 percent of the acreage was on a single large mint and truck farm in Michigan.

Tansy is not a very popular crop. The market demand for the oil is small because it has only a limited use in medicine and the price fluctuates greatly, having ranged from \$2 to \$9 a pound. The principal reason the crop is grown at all is that once established it will continue productive for years.

The crop is started by setting the field with young plants 5 to 6 inches high, pulled from established fields and set in rows 3 feet apart with transplanting machines. The usual cultivation and some weeding are necessary the first season, but the plants spread rapidly and broadcast over the field in a year. Thereafter, because of the dense growth, weeds are largely suppressed. The crop is usually harvested with a grain binder when in bloom. If labor is available the bundles are placed in shocks; otherwise they are left on the ground to cure. Some growers cut the crop and cure it in the swath like mint. Because of its heavier stems, it has to cure somewhat longer than mint before being distilled.

WORMSEED

The volatile oil of American wormseed (*Chenopodium ambrosioides* var. *anthelminticum* (L.) Gray) has been produced from this cultivated plant without interruption for more than a hundred years in Carroll County and adjoining areas in central Maryland. Why this small special industry has never shifted to other regions is not known. The plant is adapted to a rather wide area, and experiments have shown that it can be grown elsewhere. The oil has only one commercial use. It is an efficient vermifuge, because of its principal constituent, ascaridole. For a long time it has been a therapeutic agent for the control of certain intestinal parasites. For many years, however, it has to some degree been replaced by carbon tetrachloride. As always with crops yielding products of limited use, overproduction has been frequent, especially following years when the price of the oil was attractive. The market value of the oil has fluctuated greatly but not entirely because of supply and demand. In some seasons, for reasons not always understood, the oil has been low in ascaridole, with a corresponding reduction in value.

Although wormseed is grown in a limited area, statistics on acreage and production are not always available. According to the Agricultural Census of 1940, the production in 1939 was 38,281 pounds from

927 acres by 240 growers. Later figures are not available. The seed is sown early in spring in outdoor seedbeds. When the seedlings are 4 to 5 inches tall they are set in the field with tomato planters in rows so spaced that the usual farm cultivation implements can be used. The cultivated strain of the plant is shorter and bushier and produces more seed than the wild form. As the seed covering contains a high percentage of oil, the cultivated plant yields more oil to the acre. In early fall the crop is harvested with a mower with a buncher attachment, as used for seed clover, and allowed to dry partially before it is distilled. The yield of oil ranges from 10 to 40 pounds an acre.

The quality of wormseed oil depends on its ascaridole content, which is easily affected by several factors. If the crop is harvested when too immature the ascaridole content of the oil is low. The temperature of the condenser water and the rate of distillation must be carefully controlled. Under certain conditions ascaridole is soluble in water, and some producers redistill the distillate water, thus recovering much of this important constituent.

WORMWOOD

Wormwood oil is obtained by distillation of the perennial herb *Artemisia absinthium* L. Its production in recent years has been confined to southwestern Michigan, where the crop is grown on about 500 acres of muck soil in St. Joseph and Cass Counties, and to western Oregon, where 27 acres was grown in 1939, according to the United States Census. The crop is grown mainly by producers of mint oil. The plants are started in seedbeds in spring for fall transplanting to the field, or in fall for spring or summer transplanting. The soil in the seedbed must be well prepared and in fine tilth, and the seed scattered evenly on the surface and left uncovered. Most growers prefer to transplant the seedlings when they are 5 or 6 inches high, but smaller or much larger plants can be used. The seedlings are loosened with a fork, lifted from the ground, gathered, and tied into large handfuls. When the plants are rather large the tops are cut off on a chopping block with a sharp knife or hatchet. The bundles are then dipped in water and placed close together in a crate, in which they are taken to the field. The planting is done with celery- or mint-transplanting machines. At present farm-wage scales it costs \$35 to \$40 to grow the seedlings for 1 acre and set them in the field. Cultivation sufficient to control weeds is required, and hand weeding is generally necessary if the field is to remain productive for several years. The more successful growers use fertilizers, commonly a fall application of 3-12-12 and about 200 pounds of ammonium nitrate to the acre in the spring.

Wormwood is harvested in the early- to full-bloom stage. A grain binder pulled by a tractor with a power take-off is used. The wormwood stems are coarse and tough; hence, the extra power is required. The bundles are put in small shocks and allowed to cure for several days, after which the oil is distilled in mint-distilling equipment. The yield varies greatly, ranging from 7 or 8 pounds an acre from poor fields to as high as 40 pounds in exceptional cases. On the average, 20 or 25 pounds may be expected if the crop is handled well and the season is favorable. If the weeds are controlled well a wormwood field

remains productive for several years. The crop does not appear to be subject to serious diseases or insect pests.

Practically all growers of wormwood produce the oil under contract, extending over 5 to 10 years. The oil has only one important use—as a therapeutic component of a liniment for man and animals. The manufacturers of the liniment provide the contracts for the growers. As there is no outlet for the oil for other purposes, production in excess of the quantity contracted for is inadvisable. There is no opportunity for much increase in wormwood acreage where it is now grown or for its introduction elsewhere in the United States.

OILS OBTAINED AS BYPRODUCTS

CEDARWOOD

The oil of the red heartwood of eastern redcedar (*Juniperus virginiana* L.) has long been valued as a moth repellent, which is the basis of the use of the wood in the construction of clothes chests and closets. The oil is used for impregnating garment bags, is a component of cleansing and polishing liquids, furniture polish, and sweeping compounds, and is used in the scenting of soap.

Cedarwood oil is produced entirely as a byproduct in the manufacture of cedar lumber, cedar chests, pencil slates, and other articles. Utilization of redcedar sawdust and other wastes for this purpose has at one time or another been a definite part of the operations of half a dozen or more manufacturers of cedarwood products in southwestern Virginia, western North Carolina, Kentucky, Tennessee, and Florida. Eastern redcedar stumps and roots and old fence rails of the same wood are also frequent sources of the oil. Production of the oil has been sporadic, as its price has fluctuated over a wide range, owing in part to overproduction.

To distill the oil with steam in conventional distilling equipment it is necessary to reduce the waste-wood stumps or other large material to very small particles. First the material is passed through a hogging machine, in which it is chopped into thin pieces up to 10 inches in length. A hammer mill reduces the pieces to a coarse powder, which is then loaded into a still that holds from 3,000 to 5,000 pounds. Distillation is conducted as with other oils, but 8 hours is usually required to exhaust the charge. The water that flows from the receiver is generally conducted into a second receiver, where a small additional quantity of oil is obtained that is redistilled, resulting in a water-white oil known in the trade as double-distilled. The yield of oil depends on the proportion of red heartwood and sapwood used. The heartwood may yield up to 4 percent of oil; sapwood generally yields less than 1 percent.

Since 1935 there have been several attempts to produce cedarwood oil in Texas. Eastern redcedar is not abundant in Texas, but two other species, *Juniperus ashei* Buchholz and *J. monosperma* (Engelm.) Sarg., which grow over several million acres, were intended to be utilized for the purpose. These species are removed in land-clearing operations and cut into posts or burned. It was believed feasible to produce the oil commercially in connection with such operations, and several years ago several hundred thousand pounds of oil was produced annually. However, the oils from the two species differ from the oil of *J. virginiana* in composition and are therefore not adapted in all

cases to the same commercial uses. This appears to have made it difficult to find an adequate market outlet for such oils at a profitable price. Moreover, the production costs are reported to be greater than that of the Eastern cedarwood oil obtained as a byproduct in the red-cedar industry. It seems unlikely that any Texas cedarwood oils will again enter the trade in substantial quantities unless conditions more favorable for their production develop.

LEMON AND ORANGE

Of the several products obtained from citrus fruits in the United States the volatile oils from the rinds are particularly important. Lemon, orange, and grapefruit oils are produced in California, Florida, and Texas, mainly in connection with the freezing and canning of the juices from the fruit. Florida also produces tangerine and lime oils in limited quantities. Cull fruit is a source of the oils, particularly lemon oil. The fruit is passed through crushing rolls, whereby the juice is squeezed out and the oil cells in the rind are ruptured. Part of the oil thus liberated is found in the juice; the rest remains in the crushed rind and pulp. The juice is passed through a centrifugal separator that separates out the oil. This oil is of fine quality and needs only to be filtered to meet the highest market requirements. The rind and pulp are ground and distilled with steam. The oil thus obtained is somewhat inferior in quality. The residual material is used as a cattle feed. Both lemon and orange oils are used extensively as flavoring agents. The production of citrus oils by the method described has become well established in the utilization of citrus fruits in the citrus-growing States.

APRICOT AND BITTER ALMOND

The volatile oil obtained from the kernels of the apricot (*Prunus armeniaca* L.) is for all practical purposes identical with that obtained from the kernels of the bitter almond (*P. amygdalus* var. *amara* (DC.) Foeke), as both contain the glycoside amygdalin from which the oil is produced by chemical reaction under certain conditions. Much of the oil used commercially is obtained from apricot kernels. In California large quantities of pits are left over annually from the canning of apricots, and the kernels from these pits from time to time have been used for the production of the volatile oil. Bitter almonds are not produced in large quantities in this country, but the imported nuts may perhaps be used to some extent for the production of the oil. Peach, prune, and cherry kernels also contain amygdalin and can be used for the purpose, but their commercial utilization has not been undertaken in this country.

For the production of volatile oil the kernels are ground or crushed and then macerated with warm water for 1 hour, which induces a chemical reaction whereby the volatile oil is formed. The mass is then subjected to steam distillation. If it is desired to utilize the fixed or fatty oil, this can first be largely removed from the ground kernels by means of a hydraulic press or oil expeller. The yield of volatile oil varies greatly, according to the care exercised in the maceration. The temperature of the mass and the time allowed for maceration are exceedingly important. The oils consist mainly of benzaldehyde and hydrocyanic acid. Their chief commercial uses are

in the perfumery industry and for flavoring confectionery. As hydrocyanic acid is a powerful poison, it must be removed if the oil is to be used in food products.

HOP

A volatile oil is obtained by steam distillation from lupulin, a resinous brown powder on the strobiles of hops. It has the typical aromatic and flavoring qualities of hops. When hops are harvested by machinery some of the lupulin and some broken strobiles accumulate in the picking machines. This is the material used at times to a limited extent as a source of the small quantities of oil produced. It is usually sold by producers to manufacturers of and dealers in brewers' supplies.

FACTORS TO BE CONSIDERED IN GROWING VOLATILE-OIL PLANTS

In considering the advisability of cultivating plants for the production of volatile oils, due thought should be given to the cultural requirements of the plants, the most practical method of extracting the oil, and the marketing problem, with special reference to the trade requirements and the prices obtainable under normal conditions. The commercial growing of such plants frequently involves greater hazards than the growing of the more common crops, especially in connection with marketing the products obtained. Oils that are extensively produced in foreign countries are especially subject to occasional overproduction, thus creating periods of market depression. On the other hand, political or economic disturbances frequently reduce foreign production to such an extent that a period of high prices results, with its attendant benefit to the domestic producer.

Factors that determine the economic possibilities of a volatile-oil crop are soil and climate, acre yields, cost of production, competition with other crops, and price and marketability of the oil.

The character of the soil must in large measure determine the feasibility of growing commercially any volatile-oil plant in any region. Such plants are as exacting in their soil requirements as are the common staple crops, and any attempt at their cultivation without due consideration of this fact is certain to end in failure. Of even more importance perhaps is the influence of the climate—rainfall, temperature, and length of the growing season. The development of the oil in the plant is affected in a most pronounced way by such factors, and divergence from the most favorable conditions generally results in lowering the yield and quality of the oil.

If the crop under consideration is not already being grown in the United States or if its cultivation is to be undertaken in a region with growing conditions different from those where it is established, the grower generally lacks information on the suitability of the new region for its culture. It is then unwise to undertake extensive operations without preliminary experiments on a small scale to obtain cultural data. Such experiments will require a year or more, but may be the means of eventually saving time and expense and may in fact prevent the loss of the investment.

Assuming that the crop is reasonably well adapted to the region where it is to be grown, the acre yield will be determined by the tonnage of material produced and the percentage yield of oil from

such material. Conditions that favor a large yield of material do not always result in a high-percentage yield of oil. In fact, the opposite is frequently the case. This is especially true if the entire aerial portion of the plant is used. In herbs such as the mints, for example, the leaves are the principal oil-bearing part, and any condition that causes a rank growth, such as excessive rainfall or an overabundance of certain fertilizers, will result in a relatively high percentage of stems. Such an herb yields less oil than a shorter herb with an abundance of leaf growth. The greater cost of handling the heavy tonnage of herbs yielding a low percentage of oil further reduces the net monetary returns. On the other hand, if conditions are unfavorable, so that a normal growth of the herb is not obtained, the acre yield of oil will also be below normal.

The length of the growing season frequently determines whether a maximum yield of material can be obtained. Late-spring or early-fall frosts, even though they occur only in occasional years, are a great hazard. In most plants the maximum quantity of oil is present during the flowering stage; therefore, the growing period should be long enough to permit this stage to be reached.

The method of cultivation also has an important bearing on the acre yield. Thorough cultivation is desirable whenever possible, and the removal of weeds, especially those that are aromatic, is necessary to prevent their unfavorable effect on the quality of the oil. Plants that are grown in rows may, under certain conditions, be made to give greater yields by planting the rows only far enough apart to permit early cultivation, so that by midseason the growth will shade the ground. This conserves soil moisture, reduces weed growth, and prevents a rank woody development of the plant.

The cost of growing volatile-oil crops varies greatly with the methods of propagation, the amount of cultivation required, and the details of the harvesting methods. If the plant can be grown from seed sown directly in the field, the propagation cost is relatively low. If seed must be sown in seedbeds and the seedlings later transplanted to the field, a greater expense is involved, even if the transplanting is done by machinery. Propagation by roots also involves considerable labor and, consequently, additional cost. Annuals must be planted each year; the initial propagation cost of a perennial covers the entire period of the plant's life. Elimination of hand labor wherever possible is of the utmost importance in reducing production costs to a minimum. The harvesting of the herb is usually very simple and can be done with labor-saving machinery. The cost of harvesting flowers by hand is the principal economic obstacle in this country to the successful production of perfume plants like the rose and the jasmine.

The cost of constructing a still, although calling for a considerable outlay at the start, is minor if the enterprise is conducted on a sufficiently large scale and over a period of years. A first-class distilling outfit is good for many years if properly cared for. It should be emphasized, however, that the purchase or construction of such equipment is not warranted unless the acreage devoted to the plants is large enough. If several kinds of plants can be grown, the harvest periods of which come successively during the season, the distribution of labor incident to the continued operation of the still over a longer period tends to reduce the labor costs.

Whether or not the cultivation of volatile-oil plants is an economic possibility in any locality depends partly on the value of the land to be used for the purpose. This, in turn, is determined by the value of other crops which the land can produce. In other words, the volatile-oil plants must yield net returns at least equal to those of other crops that are being grown or that may be grown in the region.

If the oil produced is of acceptable quality and is in reasonable demand, there is no special difficulty in marketing it. The small producer generally finds it necessary to dispose of his product to dealers who specialize in supplying the trade with scenting and flavoring agents. The larger producer, however, frequently finds it possible and advantageous to sell directly to the consumer. This is sometimes done by contract. Where a number of producers are operating in the same region, the large producers sometimes buy from the small ones. Both dealers and consumers generally have buyers in the regions where oil production has acquired some magnitude, and if the market is active they either contract in advance or buy from the producers as soon as the distilling season is over. Prices are subject to wide fluctuations for various reasons. Foreign production is an important determining factor, and the balance of supply and demand is easily disturbed by seasonal conditions. Concentration of the entire production of an oil in one restricted area may result in periodic underproduction or overproduction, because unfavorable conditions cannot be offset by better-than-average yields elsewhere, as is the case with many staple crops grown over wide areas. The grower of volatile-oil plants must therefore be prepared for some years in which his returns will barely pay the cost of production. Such years are usually followed by a return of more favorable market conditions, because low prices tend to reduce the acreage the following year. This is especially true if the crop is an annual, such as wormseed, which makes it possible to grow other crops for a year or more and then return to the oil-plant crop whenever conditions appear favorable. With perennial plants, the initial propagation costs of which are high, this is usually impractical.

Volatile oils are sold largely on quality, and low-grade oils—except perhaps in years of extreme shortage—can be sold only below the market price, if at all. The United States Pharmacopoeia requires certain standards of purity for oils that are used for medicinal purposes, and these requirements must be met. The physical properties by which the standard of purity is generally determined are specific gravity, angle of rotation, and solubility in alcohol. Oils that contain important constituents capable of definite measurement by analysis must contain not less of such constituents than the minimum required by the Pharmacopoeia. A number of factors—unusual seasonal conditions, unsuitable soil, or improper manipulation of the still—may be responsible for below-standard oil. Through long experience the trade becomes accustomed to certain characteristics of oils; even if no legal standards are required, any oil having a perceptibly different odor, flavor, or color from that to which the trade is accustomed will either be rejected or command a lower price.

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