Improvements in The Field Distillation of Peppermint Oil

By
A. D. HUGHES
Professor of Mechanical Engineering

Bulletin No. 31
August 1952

A Cooperative Research Project of the Engineering Experiment Station and the Agricultural Experiment Station.

Engineering Experiment Station
Oregon State College
Corvallis

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TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

ILLUSTRATIONS

LIST OF TABLES

FOREWORD and ACKNOWLEDGMENTS

The experimental work reported in this bulletin was performed by Professor A. D. Hughes as a joint project of the Engineering Experiment Station and the Agricultural Experiment Station at Oregon State College, Corvallis, Oregon. The project was under the supervision of S. H. Graf, Director of the Engineering Experiment Station, and R. S. Besse, Associate Director of the Agricultural Experiment Station.

This work was made possible largely through a financial grant by the William J. Wrigley, Jr Company of Chicago. The assistance of the A. M. Todd Company of Kalamazoo, Michigan, and the I. P. Callison Company of Chehalis, Washington, in furnishing peppermint oil samples and in testing oil samples is greatly appreciated. The Cobb Manufacturing Company of Jefferson, Oregon, has assisted the project in the construction of the pilot plant and other experimental equipment.

The cooperation of many of the peppermint oil growers, members of the Oregon Essential Oil Growers League, has been excellent in permitting the use of their plants and peppermint hay for experimental purposes. Without their help, much of the project could not have been completed. Thanks are especially due M. C. Helms, Delmer Davidson, K. R. Allen, C. U. Snyder, O. D. Stephenson, Earl Chartrey, J. G. Cowles, and Dale Turnidge.

Assistance of staff members of Oregon State College also is appreciated; included are G. W. Gleeson, Dean of the School of Engineering, Milosh Popovich, Chairman of the Department of Mechanical Engineering, J. B. Rodgers, Head of the Department of Agricultural Engineering, D. E. Bullis, Chemist of the Agricultural Experiment Station, and W. C. Baker, formerly Associate Professor, and W. E. Phillips, Instructor, both of the Department of Mechanical Engineering.

The help of several engineering students is also acknowledged for their assistance in conducting many of the laboratory and field tests, and in calculating, tabulating, and plotting the results included in this Bulletin. These assistants included B. A. Peavy, D. T. Phillips, R. W. Reid, M. K. Miller, W. L. Moor, D. F. Hayes, E. O. Merklin, R. E. Powne, R. A. Engdahl, and P. D. Beck.

Many others too numerous to mention have contributed to the project by helpful suggestions and constructive criticism, all of which are gratefully acknowledged.

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I. INTRODUCTION

1. **Objectives of the project.** Peppermint oil has been distilled by a rather hit-and-miss method for over 60 years in the United States alone, and even the present methods not only are very wasteful of fuel oil, cooling water, equipment, and manpower, but considerable quantities of the valuable oil are "degraded" or completely lost because of improper equipment or procedures.

The author had occasion three years ago to observe several of the peppermint growers' field distillation units in action. From an engineering standpoint many possible improvements in the process were apparent.

Many of the operators do not fully understand what is going on within the distillation vat or tub, the condenser, and the separating (or receiving) can, and as a consequence have in some instances developed practices which unknowingly wasted or degraded their oil.

With the object of developing improved methods for the field distillation of peppermint oil, a cooperative research project was established in 1949 at Oregon State College under the joint direction of the Engineering Experiment Station and the Agricultural Experiment Station. A yearly grant from the William Wrigley, Jr Company of Chicago through the Agricultural Research Foundation at Oregon State College has made the project possible.

While some phases of the project are still under investigation, it was thought worth while to issue a joint bulletin at this time in order to inform the peppermint growers and still operators of improvements discovered to date. Any further developments will be covered by supplemental bulletins at a later date. In other words, this bulletin covers the progress made to date during the past three years since the start of the project.

"Improvements" as designated in the title is considered to include any changes in still equipment or operating procedure which will result in saving peppermint oil which would otherwise be lost during the distillation process. It is also construed to include changes in equipment or procedures which will reduce the net cost of distilling the peppermint oil by reductions in fuel oil, cooling water, overhead, and manpower.

No attempt will be made in this bulletin to make recommendations regarding the cutting, curing, or loading of the hay out in the field nor the disposition of the spent hay after it has been processed. Those subjects could well be covered by separate projects in themselves if sponsors could be found to finance the investigations. However, some of the distillation equipment and procedures are affected by the handling methods used in the field, and to that extent may be mentioned.

2. **Production and uses of peppermint oil.** The production of peppermint oil is a small but important agricultural industry of the Pacific Northwest, and the states of Oregon and Washington produced almost 60 per cent of the total crop of 1,622,000 pounds for the United States in 1950. The remainder came largely from the states of Michigan and Indiana. About two-thirds of this amount is used in this country annually in chewing gum, dentifrices, food flavoring, and for medicinal purposes, and the remainder exported. While the value of the oil varies with market conditions, in 1950 it ranged from $5 to $7 per pound (approximately a pint) giving the total crop a value of nearly $10 million. The value of the final products in which peppermint oil is used is estimated at $300 million per year. The quantity and value of the total crop in 1951 did not vary appreciably from that listed for 1950.

3. **Characteristics of peppermint oil.** Peppermint oil (*mentha piperita*) is a very complex clear organic liquid, containing 50 to 60 per cent menthol, and having a specific gravity of approximately 0.90. Being a natural product, its properties vary widely with the soil and conditions under which the plant is grown and the time of cutting, and to some extent are affected by the process of distillation. Typical values of the physical and chemical properties of Oregon peppermint oils have been published by Tornow and Fischer (1).* Average values for three Oregon oils are given in Table 1, taken from this paper, and compared with US Pharmacopoeia (USP) XIII peppermint oil.

4. **Field distillation process.** Peppermint oil is obtained by the steam distillation of peppermint hay after a short curing period in the field. The steam and mint oil vapors are then condensed at atmospheric pressure and delivered to a separating (or receiving) can. The oil being lighter than water and largely insoluble floats to

* Numbers in parentheses indicate references in the bibliography on page 50.

Table 1. TYPICAL PHYSICAL AND CHEMICAL PROPERTIES OF OREGON OILS.

Type of oil	Locality	Ester %	Total menthol %	Optical rotation 100 mm/25°	Specific gravity 25°/25°	Refractive index 20°
C	Lower Columbia	5.60	53.90	—23.5	0.9018	1.4629
E	Upper Willamette	7.57	59.21	—24.7	0.9020	1.4621
S	Central Willamette	7.36	57.05	—23.03	0.9032	1.4633
USP	....	>5	>50	—18 to —32	0.896 to 0.908	1.4590 to 1.4650

Information above from published values by Tornow and Fischer (1).

the top of the container while the water is drained continuously off the bottom. The oil is floated off the top periodically, and placed in 55-gallon drums for shipment to a processing plant. Here it may be analyzed, centrifuged, blended with other grades or classes of oils to a desired analysis. In some cases, it is vacuum-distilled to remove certain undesirable fractions.

II. FIELD DISTILLATION UNITS

1. Typical plants in Oregon.

a. PLANT WITH STATIONARY TUB, OPEN DRIP-TYPE CONDENSER. For the benefit of those who are not familiar with the types of equipment normally used in the field distillation of peppermint oil, flow diagrams of typical plants found in Oregon are shown in Figures 1 and 2.

Figure 1 illustrates a plant using stationary round tubs in which the hay is loaded and removed by some sort of crane or conveyor. These tubs usually have a large flat or conical lid which may be swung in place after the hay has been loaded. In most cases where stationary tubs are used, the hay is packed in by having workers walk or jump on it after each layer of hay has been dumped. Also in many cases the steam is turned on as soon as the first layer or two have been added, which tends to wilt the lower portion of the load and allow more hay to be packed on top.

This type of tub is commonly connected to an open drip-type condenser coil which may be of galvanized iron, aluminum, monel, or stainless steel. This coil may be from 4 to 8 inches in diameter where the vapors enter at the top and usually makes several loops in a vertical plane before discharging into the separating (or receiving) can. Cold water is allowed to drip from troughs above the top coil and sometimes from an additional trough lower down, thus condensing the steam and mint oil by a combination of conduction and evaporative cooling.

In many plants with the open-type coils, very little use is made of the cooling water after it has passed over the coil. Others arrange to utilize at least part of the waste water for boiler feed water.

Figure 1 also indicates a type of separating can which is used in some of the plants, consisting of a galvanized iron cylinder about 15 inches in diameter and 6 to 8 feet long, usually partly buried in the ground. The condensed steam settles to the bottom of this separating can and is drawn off the bottom through a pipe connection. The peppermint oil which rises to the surface is usually drawn off periodically by simply raising the level of the water in the separating can.

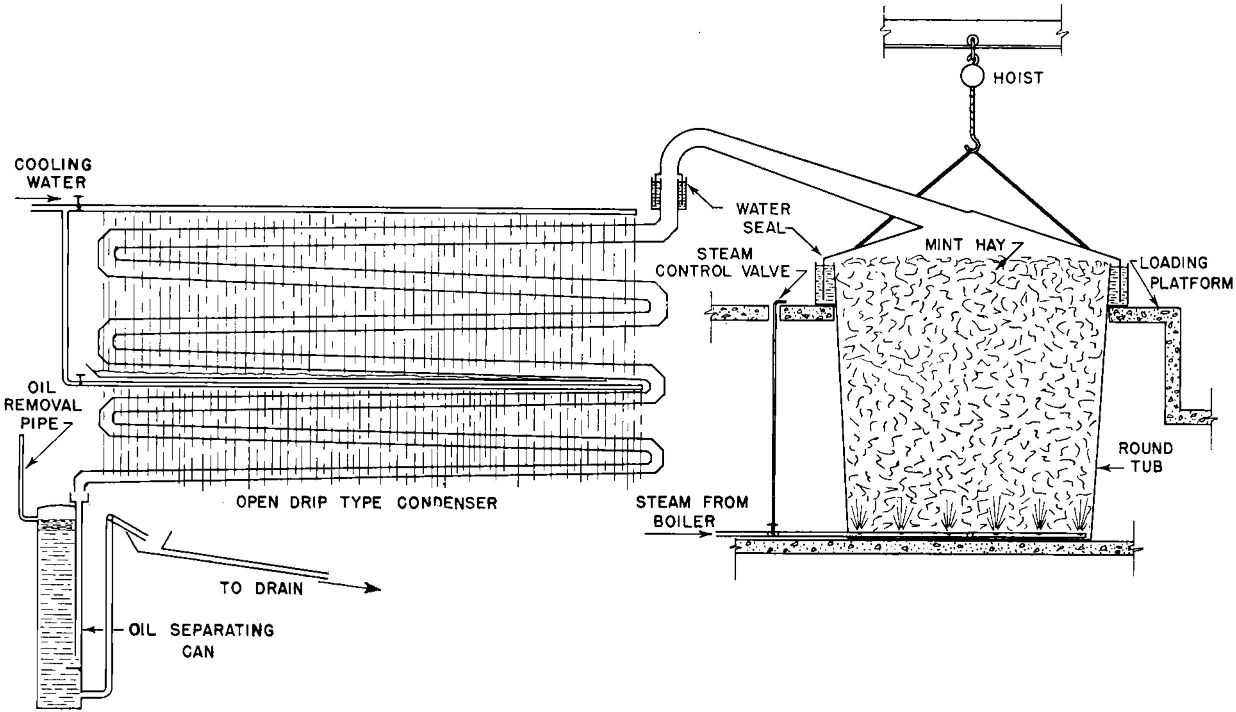

Figure 1. Field distillation unit with stationary tub and open drip-type condenser.

b. PLANT WITH PORTABLE TUB, SUBMERGED CONDENSER. Figure 2 shows another common type of distillation unit in which the hay is loaded out in the field into rectangular galvanized iron tubs mounted on flat-bed dump trucks. These tubs are loaded in the field with long hay by using a regular hay loader, or may be loaded with chopped hay blown in from a chopper. The truck is usually driven into the stall alongside the condenser and the still top lowered into place by means of a crane. After clamping this top all the way around with quick-acting toggle clamps and connecting the discharge pipe to the condenser, the steam is turned on. This enters the load through pipes fastened to the bottom of the tub and connected to the steam line by means of a flexible hose.

This type of plant quite often uses what is known as a "submerged" condenser in which the steam and mint oil vapors from the tub are collected in a header from which lead out a number of tubes (usually three passes) which are submerged in a rectangular tank of water. The condensate and mint oil are collected in the lower header and usually drain directly into the funnel of the separating can.

Another common type of separating can is shown in Figure 2. This is usually made of galvanized sheet iron and is about 30 inches in diameter and 4 feet high. The steam condensate and mint oil mixture come down a tube to about the middle of the separating can where it strikes a horizontal baffle. This is supposed to disperse the oil in approximately a horizontal direction, permitting it to rise to the top to the collecting chamber, a short cylindrical section on top of the separating can.

The steam condensate settles to the bottom of the can where it is drawn off and either discharged to a drain or sent to a redistillation unit.

The combination of equipment indicated in Figures 1 and 2 is merely to indicate the types in common use; there are many plants using different combinations of similar equipment. Many plants also use portable round tubs mounted on tractor-drawn trailers.

Perhaps at this point it might be well to mention that these plants in the main have always been manually operated, and with very few instruments other than the pressure gage on the boiler, which is required by law. Since they operate at most only a few weeks a year, during the short harvest season only, the mint growers are reluctant to spend money on controls or better equipment unless they can be assured that it will pay off in a very short time.

2. **Redistillation unit.** Peppermint still operators have for many years known that the separating cans indicated above did not

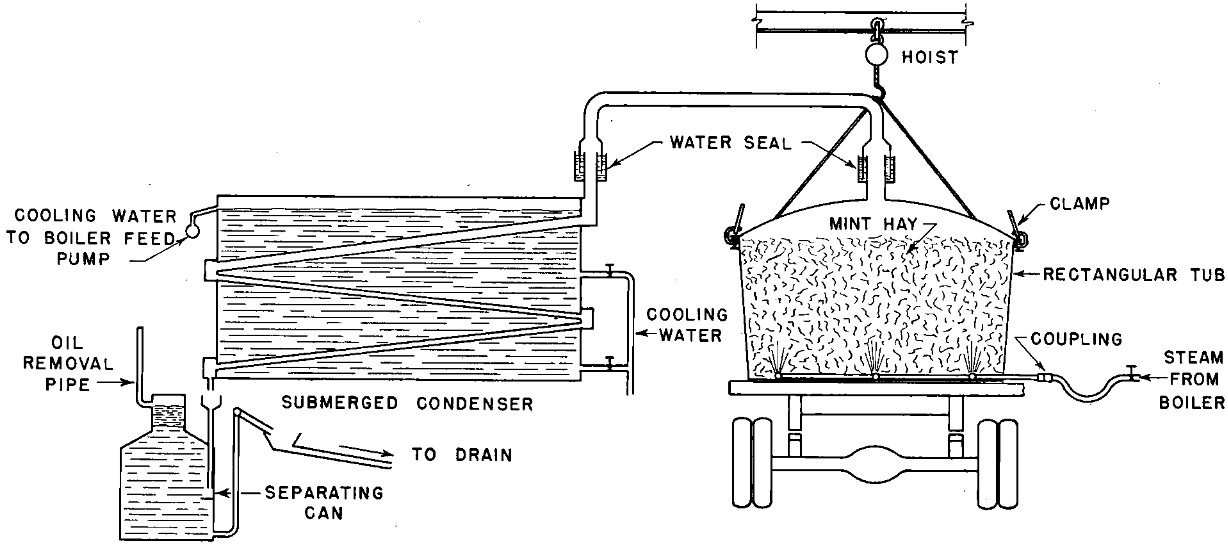

Figure 2. Field distillation unit with rectangular portable tub and submerged condenser.

separate all of the oil from the condensed steam, so a secondary process was developed in an attempt to extract valuable oil from the separator can outflow water, which oil would otherwise be thrown away. The redistillation or recovery process takes place in what is commonly called a "redistill." The overflow water from one or more separating cans is directed into the redistill, a cylindrical container about 36 inches in diameter and 30 inches high, where it is heated to the boiling point at atmospheric pressure, usually by a submerged high pressure steam coil. This vaporizes any small amount of peppermint oil which may have been left in the water, and this vapor together with the steam that also evaporates from the liquid surface is carried to another condenser. The condensed water and redistilled (or recovery) oil from this water-cooled secondary condenser are collected in another small separating can where the redistilled oil floats to the top and the water is drawn off the bottom and discarded. In the redistillation unit not all of the water passing through is evaporated, and it seems only necessary to bring it to a good rolling boil in order to release the oil contained in it.

The oil obtained by this recovery or redistillation process is different from the prime oil obtained by the first or prime distillation process from the hay. It has a slightly greater specific gravity, and has a stronger or acrid odor. While this secondary or redistilled oil does not have the quality or flavor of prime oil, it does bring a price of from half to two-thirds that of prime oil, which makes it worth recovering. It has been the practice for years in custom distilleries for the still operator to take this secondary oil as a part of his payment for the use of the still. In many cases, the returns from this lower grade oil have paid the fuel bill for the season so it is no little item. However, as the redistillation unit itself requires almost as much steam as a full-sized distillation tub, the economics of the situation are not entirely solved as yet.

3. **Mint still survey.** It was felt desirable to obtain some information from the mint growers themselves concerning the type and capacity of field distillation units then in operation. A questionnaire was sent out in late 1949 to all of the mint growers in Oregon and 32 replies were received from those who were plant operators. This represents about one-third of the estimated number of plants in Oregon at the time.

Of the 32 plants, it is interesting to note that 16 used stationary tubs and 16 used portable tubs, each plant on the average cooking two tubs at a time when in operation. According to the survey, 25 of the 32 plants used long hay and 7 used chopped hay. Twelve of the plants used open-drip type condenser coils and 20 used the submerged type condenser.

As far as boiler capacity is concerned, taking the installed boiler capacity and dividing it by the average number of tubs cooking at a time, it appears that about 48 boiler horsepower of installed capacity is used per operating tub. This does not mean that the boilers are used to their entire rated capacity, but since many of the plants are short of steam, no doubt this is the case in these instances.

The plants included in the survey operated anywhere from 2 to 56 equivalent 12-hour days during the 1949 season with the average of the 32 plants standing at 22 days of operation. During this season, the 32 plants averaged 7,400 lb of prime oil each with the plant capacity varying from 300 to 30,000 lb. With this wide range of capacity, a break-down of the size of the plants based on the season's output indicates the following:

Season's output of prime mint oil	*Number of plants in survey*
Up to 5,000 lb	16 plants
5,000 to 10,000 lb	10 plants
10,000 to 20,000 lb	2 plants
Over 20,000 lb	4 plants
Total	32 plants

This would indicate that most of the plants are of smaller size, probably operated by individual growers for their own and possibly neighboring fields.

As a measure of the performances of these 32 plants, the operators were asked to give also the amount of fuel oil used during the season. Then taking the total pounds of mint oil produced for the season divided by the gallons of fuel oil burned gives a ratio which is a measure of the performance of the plant. On this basis the pounds of mint oil produced varied from 0.50 to 1.50 per gallon fuel oil, the average being approximately 1 lb. It is obvious that the more mint oil produced per gallon of fuel oil the more efficient the plant.

4. **Tests on field distillation units.** It was thought best to run a few tests on some actual full-sized field distillation units to see just how they had been run for these many years, with the idea that possibly some obvious improvements would show up during such tests. Several still operators gave splendid cooperation in allowing their plants to be instrumented and tested. Some data for a typical

plant of the type shown in Figure 2 will be given as an example; this plant used portable truck tubs and submerged condensers. An over-all view of the plant is shown in Figure 3.

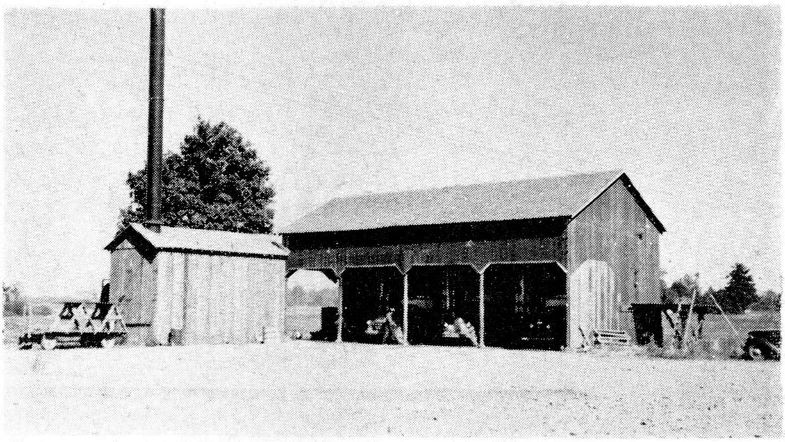

Figure 3. Helms plant near Jefferson, Oregon.

a. HELMS PLANT, NEAR JEFFERSON, OREGON. The first test made on the Mike Helms plant near Jefferson, Oregon, was simply to determine the usual method of operation. The regular operator maintained the normal manual control of the plant for the entire test. Instruments had been previously installed to measure the temperature of the cooling water in and out of the condenser, the condensate temperature leaving the condenser, the steam pressure in the lines entering the bottom of the tub, and provisions were made to measure the rate of condensed steam and mint oil leaving the condenser. These latter items were obtained by measuring the time for a given quantity of water and oil to collect in a cylindrical graduate. All of the foregoing readings were taken at five-minute intervals, measured from the time the steam was first turned on.

At this point it might be well to explain that still operators commonly measure the time of processing or "cooking" from the time the steam "breaks through" the load of hay which is usually some 10 to 15 minutes after the steam is turned on. However, for these tests it was felt desirable to measure the time from the first opening of the steam valve, since obviously some of the hay was being cooked as of that moment.

The first test made on the Helms plant was with a load of chopped hay which was considered quite green; in other words, it had not had time to cure properly in the field. A graphical log of this test run is shown in Figure 4. Note that the operator opened the valve to the tub and maintained a pressure of about 60 psig until the "break through" time. The boiler pressure during this time varied from 120 down to about 105 psig. In this plant the still top

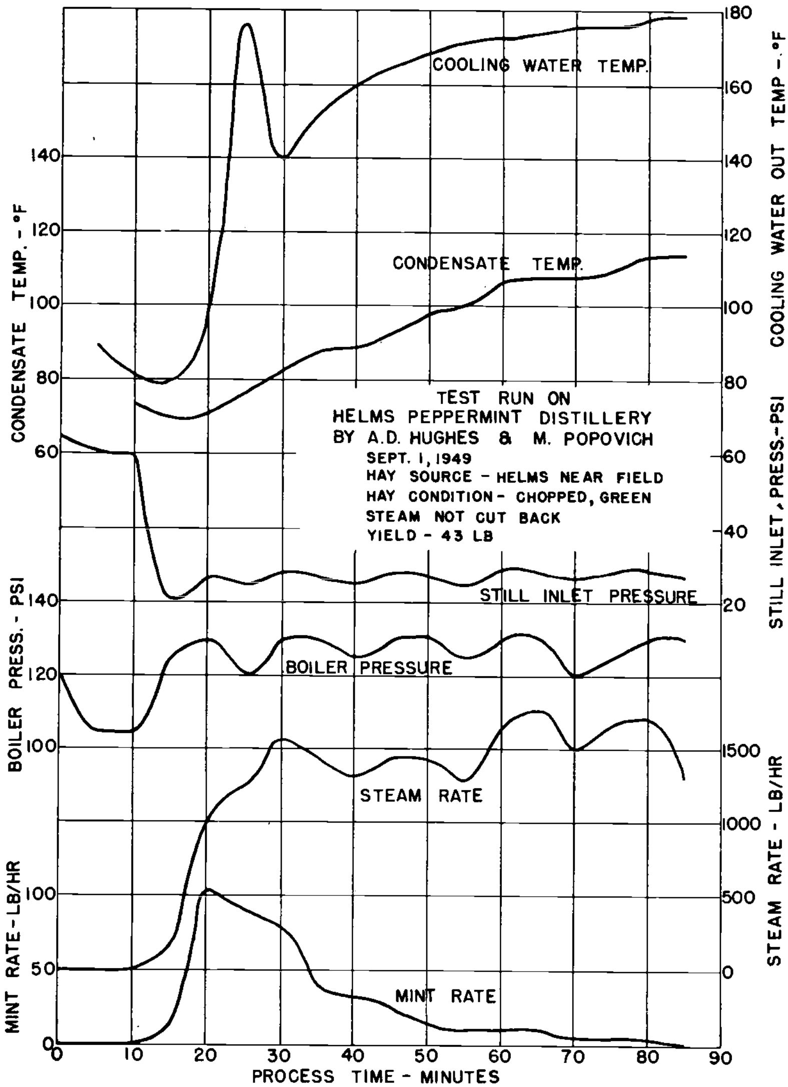

Figure 4. Log of normal run—Helms plant.

cover contained a ¾-inch pipe tapped opening from which the plug was removed. This permitted direct venting of the large quantity of air trapped in the tub with the hay and the "break through" time was determined by the moment steam started coming through this vent. At that time the operator quickly put the plug back in and dropped the steam pressure to the still to about 25 psig. Incidentally, the still nozzle pressure is not ordinarily known to the operator; he usually operates by the number of full or partial turns of the valve which he considers to give the best operating conditions. It was a revelation to him actually to see the steam pressure to the still measured by means of a pressure gage.

In this first test the cooling water out temperature started in at about 90 F and dropped to slightly below 80 F before sufficient quantity of steam came over to cause the temperature to rise again. After the "break through" time it will be noted that the steam rate and mint rate increased very rapidly; the steam rate going to slightly over 1,500 lb per hour and the mint rate reaching approximately 110 lb per hour. Note that the mint flow peaked rather rapidly and then fell off with no adjustment on the steam rate. At a time 25 minutes after the steam valve had been opened, the cooling water temperature reached 175 F and the operator opened up the cooling water valve which caused the temperature to drop suddenly to 140 F. The condensate temperature gradually increased from about 70 F up to 113 F at the end of the run. In this case the total run time was about 85 minutes from the time the steam was turned on or about 75 minutes from the "break through" time. This was the standard rate at which this type of green hay was processed. This load yielded 43 lb of oil.

Incidentally, it is a simple matter for a still operator to obtain the steam rate and mint rate for himself. The time required in seconds to collect 1 gallon of steam condensate (water) divided into 30,000 will give the pounds per hour of steam being used. For example, if it takes 25 seconds to collect 1 gallon of condensate leaving the condenser, 30,000 divided by 25 equals 1,200 pounds per hour of steam used. This divided by 30 gives the equivalent steam consumption in boiler horsepower; in this case 40 boiler horsepower for the usual steam pressures and temperatures in mint stills.

The mint rate is found more easily by using a graduate having cubic centimeter or milliliter markings. Using one of these, the number of cubic centimeters collected divided by the number of seconds it took to collect the sample and multiplied by 7.1 will give the mint oil rate at that moment in pounds per hour.

It was noted on this test on the Helms unit that the steam rate was fairly constant after the peak mint rate had been reached. From this test it appeared obvious that once this peak rate had been reached there was no necessity of continuing the steam flow at such a high rate since there was no material increase in the temperature of the vapor and the smaller amounts of mint coming over could not justify the large amount of steam being used. On this basis, the test shown in Figure 5 was made in which the steam pressure to the tub was "cut back" to reduce the amount of steam used after the peak mint rate had been passed. Thus the still inlet pressure is shown dropping in 5-lb decrements from the original 20 psig, down to a minimum of 2 psig. In other words, an attempt was made to have the quantity of steam follow the quantity of mint oil coming from the tub so that as the mint rate dropped off the steam should drop off also. At the end of 80 minutes after the steam was turned on, the still inlet pressure was raised to 5 lb to see whether any appreciable quantity of mint oil would come over with the increased steam rate. There was a slight increase in the amount of mint oil coming over, which indicated that the pressure had been dropped too low for that particular run. While these two runs on the Helms plant were not based on identical types of hay, it was interesting to note that the yield was the same for both loads, yet the total steam consumption for the "cut back" process was about 25 per cent lower than by the first method.

In the Helms plant the cooling water from the condensers flowed by gravity to a large tank outside the plant, from which the boiler feed water was taken by the boiler feed pump. In this instance, however, it appeared that less than one-third of the hot water available from the condensers was being used in the boiler, the rest being wasted.

b. DAVIDSON-KIEPER PLANT, NEAR TALBOT, OREGON. The Delmer Davidson-Herman Kieper plant near Talbot was the second one to be tested in the 1949 season. This is a relatively new plant, having operated only one season previously, and it consists of six 8-ft diameter and 8-ft deep stationary tubs, connected three at a time to three submerged condensers. In other words, the tubs were of the type shown in Figure 1, but the condensers and separating cans were as shown in Figure 2. The mint hay was loaded in the field on flatbed trucks which were backed into the loading platform. The hay was then lifted from the trucks and dumped into the tubs by means of an electric hoist and hay fork. Two tubs are alternately connected to the same condenser; one tub can be cooking while the other is being loaded or unloaded, resulting in practically constant operation of the condensers. This plant usually runs 24 hours a day during the harvest season. Steam is furnished by a 125-hp Kewanee boiler with an automatic oil burner.

Here again the first test was made simply recording the essential data as the regular still operator followed his usual procedure for the type of hay involved, as indicated in Figure 6. Note that the still inlet pressure was relatively low, never getting above 5 lb and

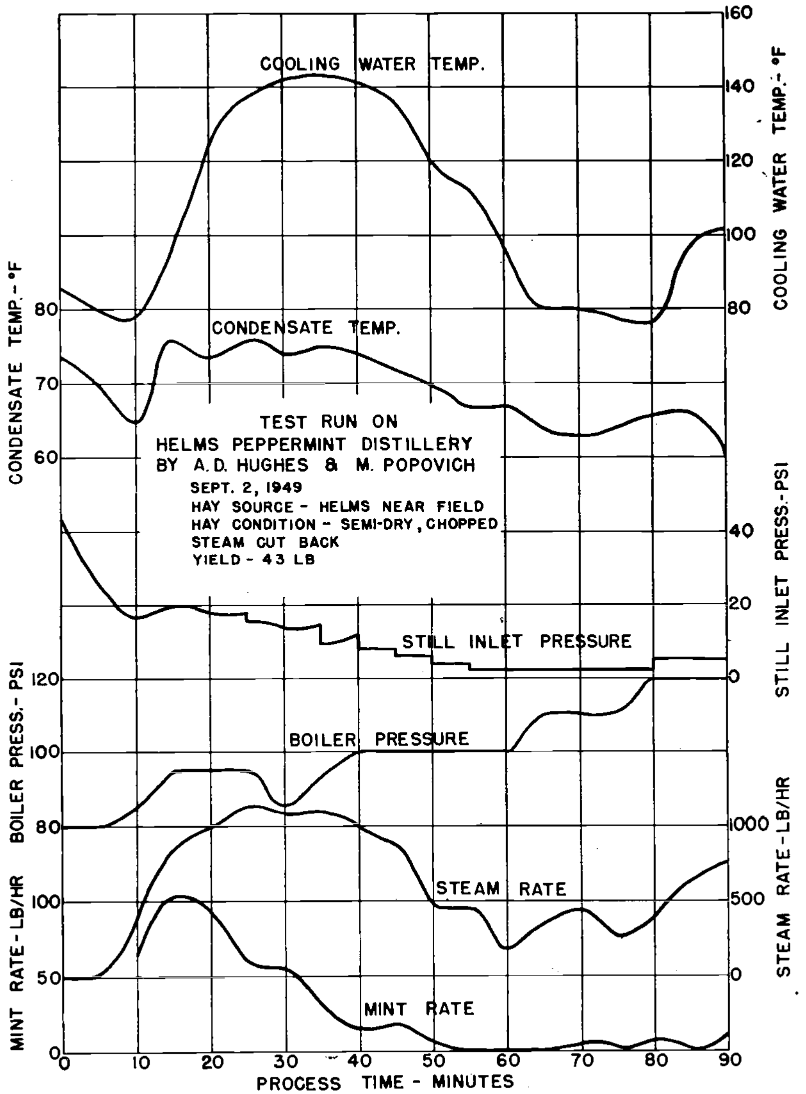

Figure 5. Log of test run with steam cut back—Helms plant.

most of the time running between 1 and 2 lb. The boiler pressure stayed relatively close to 95 lb gage during the entire run.

Although this hay was considered dry, the operator had some difficulty in getting the load to "break through" until about 25 minutes had elapsed. No provisions were made in this still for venting the air present with the hay in the tub and the blowing of this air at the outlet of the condenser was quite noticeable. No doubt this slowed up the process as the steam could not get to the condensers until the air had been forced through. More steam pressure might have been used, but with the large floating lids used in this plant, only a slight increase in the steam pressure would cause the lids to float or rise in the water seal and allow steam to escape to the atmosphere, which was not desirable.

During this test the operator made some changes in the rate of cooling water flow and managed to keep the temperature within the range of 175 F to 205 F; the condensate temperature, however, varied from 62 F up to 168 F.

In the test on the Davidson-Kieper plant a remote-reading thermometer was installed in the line between the tub and the condensers to measure the vapor temperature. The variation in this temperature also is shown in Figure 6 and it is interesting to note that the still top temperature, as this was called, reached a maximum of about 214 F at the same time the mint rate reached its maximum of about 67 lb per hour. This would indicate that a maximum amount of mint oil is brought over with the steam just at the time the load is heated up to about 212 F, the temperature of steam at normal atmospheric pressure. Note that in this test the steam rate was fairly constant from the 30-minute time on as there was no change in the steam valve by the operator.

Based on the values obtained in the first test on the Davidson-Kieper plant, the writers were permitted, through the cooperation with Mr. Davidson, to experiment with different procedures. In the second test, shown in Figure 7, the cooling water rate was not changed but the still inlet pressure was cut back to 2.5 lb gage pressure 30 minutes after the steam was turned on. This was in an attempt to get the steam rate to follow the mint rate. The reduction of steam pressure did not affect the temperature of vapor leaving the mint hay, yet the mint rate curve took the same typical shape. It was estimated that in this case the cutting back of the steam rate saved approximately 20 per cent on steam.

It is difficult to compare these two runs in regard to the yield of mint oil since in the first case the hay was dry, but in the second, dry and quite weedy.

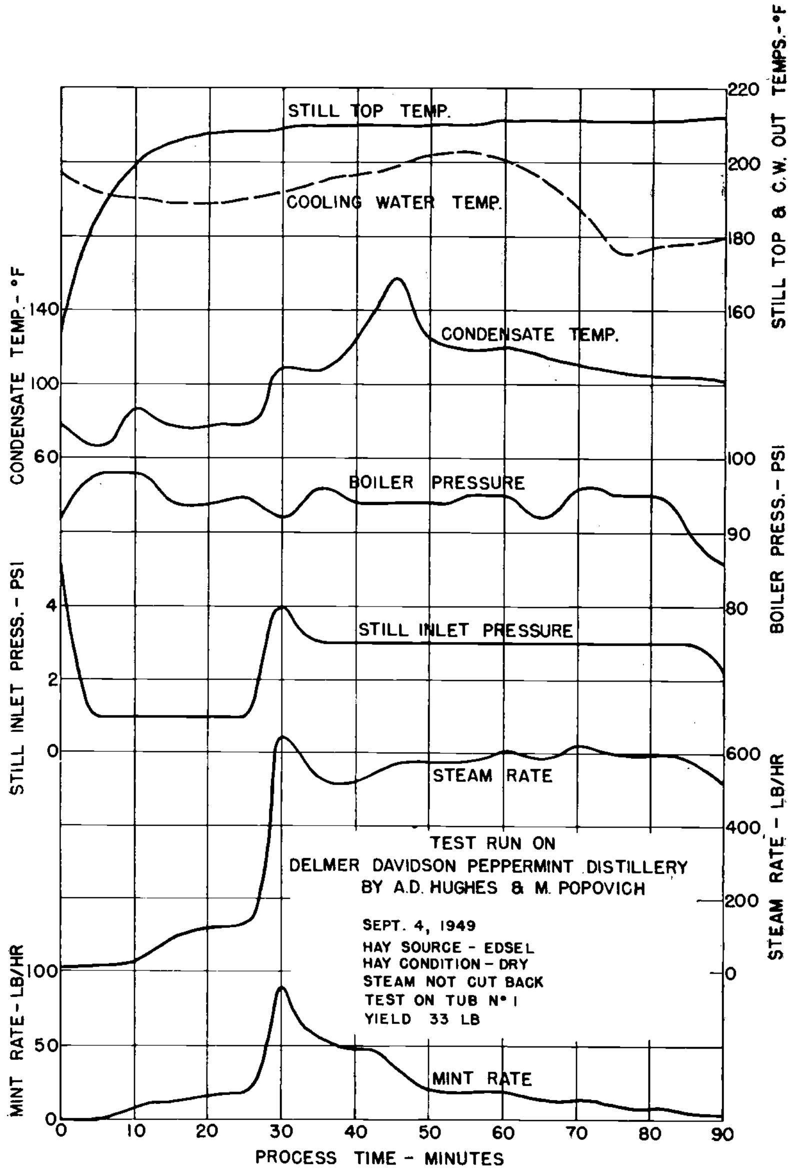

Figure 6. Log of normal run—Davidson-Kieper plant.

c. EARL CHARTREY PLANT, CLATSKANIE. The open-air plant of Earl Chartrey is shown in Figure 8 and included a 100-hp vertical boiler with a manually controlled steam atomizing burner. Rectangular portable tubs mounted on flat-bed dump trucks were loaded with

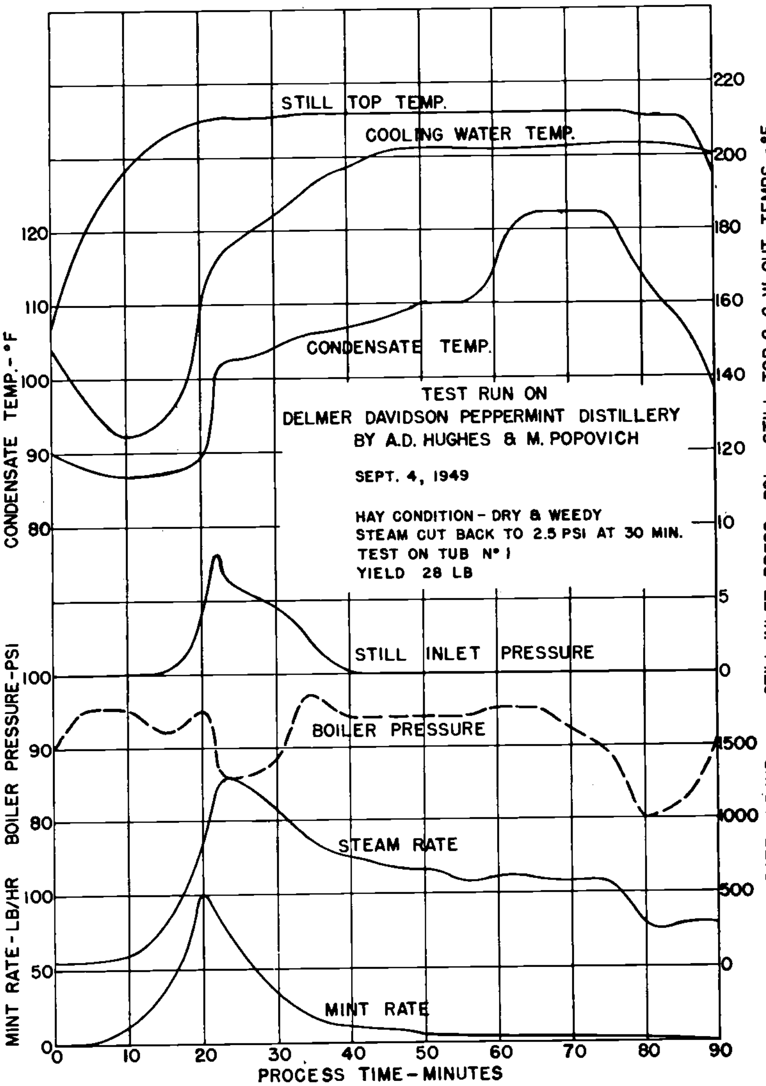

Figure 7. Log of test run with steam cut back— Davidson-Kieper plant.

long hay in the field with hay loaders and these portable tubs were backed into the two stalls during the processing period. One tub was connected to a submerged condenser and the other connected to an open drip-type condenser. For these tests instruments were placed on the portable tub which was connected to the submerged condenser, since this setup permitted measurement of the desired quantities more readily.

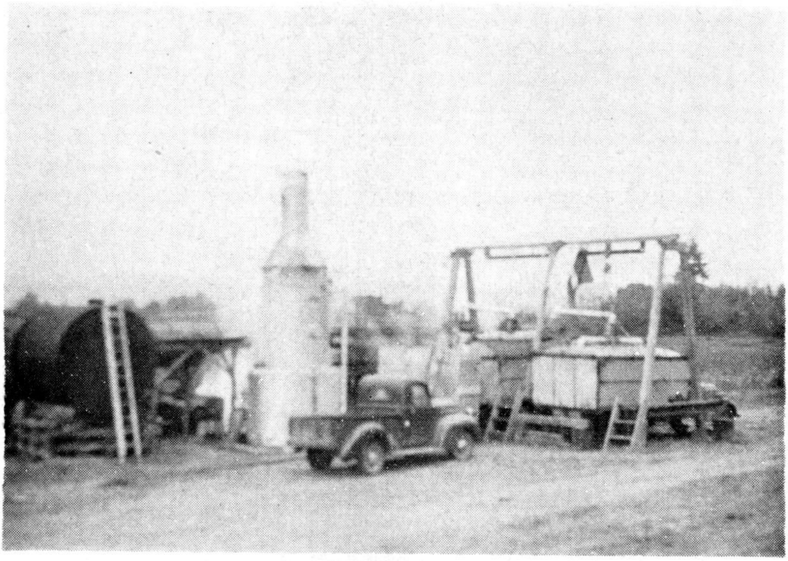

Figure 8. Plant of Earl Chartrey, Clatskanie.

The results of the first test made on this plant are shown by the curves in Figure 9, and show no unusual characteristics which have not already been discussed. However, in this case the hay was quite wet and green; it had been cut only the day before, cloudy and drizzly weather having prevented proper curing. The curves indicate that it took almost 25 minutes for the steam to break through the load although the still inlet pressure was between 40 and 50 lb gage. The observers were able to follow the movement of the steam up through the load on the outside of the tub by placing their hands on the bare sides. It was obvious by this method of observation that the hay had not been loaded uniformly and the corners of the rectangular tub showed evidence of heating up first. There was a chilly wind blowing at the time also, which no doubt added considerably to the amount of steam required to heat up the hay in the uninsulated tub.

Again in this first run, the operator processed the hay in the same method he had been accustomed to, and the observers merely took data at the various temperatures and pressures during the pro-

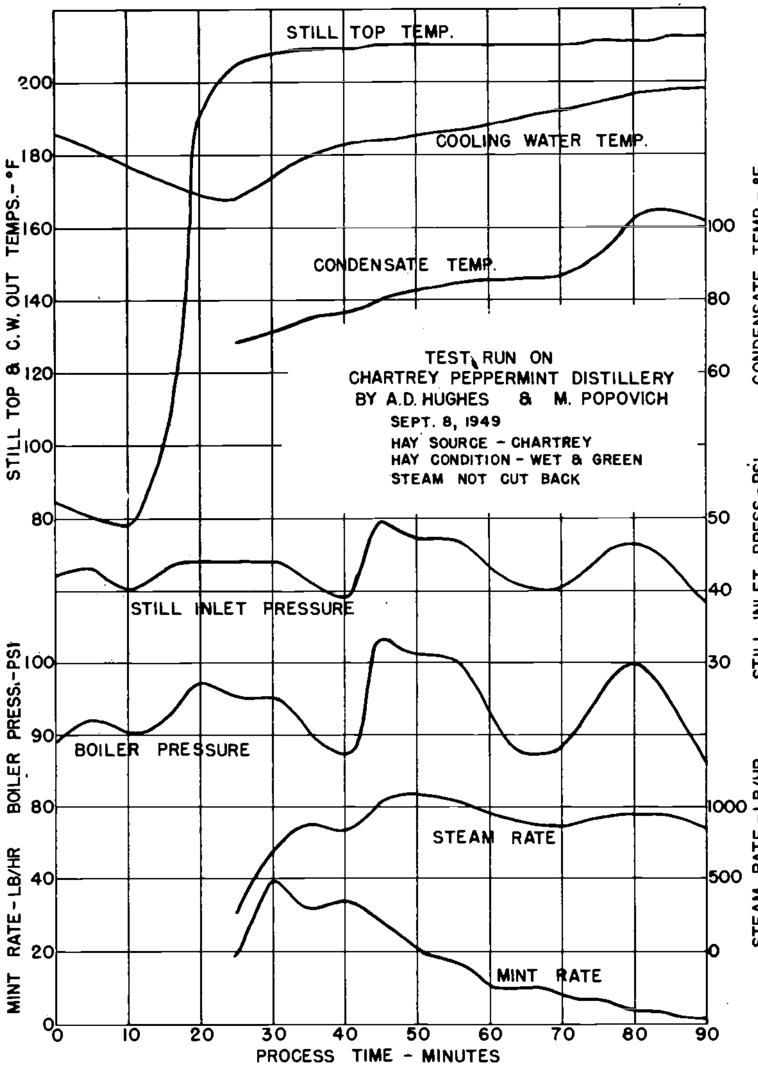

Figure 9. Log of normal run—Chartrey plant.

cess, as shown in Figure 9. Note that the boiler pressure varied considerably during the first test run, but this was to be expected under manual control of the burner. The valve controlling the steam to the tub was not adjusted after it was opened at the beginning of the run and for this reason the still inlet pressure varied directly with the boiler pressure as shown on the curve in Figure 9. For this reason also, the steam rate was practically constant after the majority of the mint oil had passed over to the condenser. Again in this instance, the still top temperature, or the temperature of the steam and mint oil vapor passing over to the condenser, reached a maximum temperature of about 212 F shortly after the peak of the mint rate had been passed. The cooling water temperature varied from about 168 F up to 198 F during the run and this was somewhat responsible for the variation in the condensate temperature which ranged from 68 F to 105 F.

From the above data it is believed that the steam rate could have been considerably reduced during the latter part of the run without any sacrifice in the amount of mint oil produced and if the cooling water rate had been regulated automatically, much less cooling water would have been required. Also, the condensate temperature could have been kept within the desired range of 100 F to 110 F. It is also believed that the time required to reach the "break through" period could have been materially reduced if the bare rectangular tub had been insulated from the cold, drizzly weather conditions outside. In this plant it was noticed that neither the boiler nor the steam lines were insulated, effectively reducing the capacity of the boiler to produce steam.

Based on the first test run at this plant, the writer was permitted through the cooperation of the operator to change the operating conditions slightly, with the intention of reducing the quantity of steam required for distilling the oil. Figure 10 shows the results of this second test on the same type of hay and, so far as is known, the same quantity of hay. The steam pressure to the still was reduced gradually after the still top temperature reached a maximum value of 212 F. The still inlet pressure was about 45 lb gage at a time 50 minutes after the start of the run and this was reduced about 10 lb each 5 minutes after that time. From the curves in Figure 10, it can be seen that the steam rate then tended to follow the mint rate and there was no noticeable difference in the quantity of mint oil produced. In this installation it was not possible to measure the quantity of oil produced from each load as the oil was drawn off only after several loads were processed.

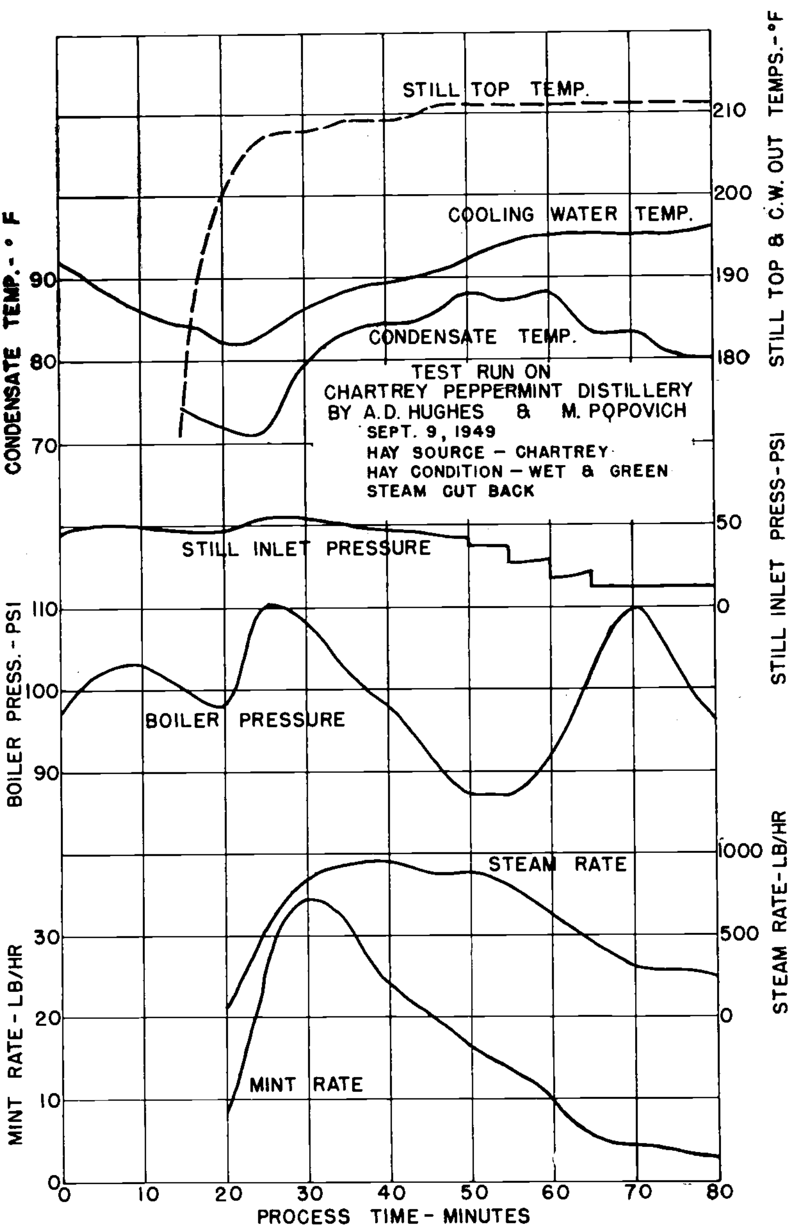

Figure 10. Log of test run with steam cut back—Chartrey plant.

In this second run the condensate temperature varied from 72 F to 88 F which indicated that too much cooling water was being used and the condensate temperature was too low for maximum rate of separation of the oil and condensate.

This was the first set of runs made on wet hay and, so far as could be determined, it appeared that wet mint hay simply required more steam and thus more process time than dry hay to remove the oil. No particular differences were noted in the appearance of the oil or in its quality.

d. HEAT WASTAGE. In one particular plant it was noted that large quantities of hot water were being wasted, which was largely made up of the cooling water from the condensers. In this particular plant this hot water was collected in a large tank and part of it used for boiler feed water. However, a measurement was made of the overflow from this tank which was at the rate of 42.5 gallons per minute, the water in this case being at 118 F. This is the equivalent of 37.5 boiler horsepower being thrown away down the drain. If this heat could be saved by some economical means, about 125 gallons of fuel oil during a 12-hour day could be saved.

There is also a large waste of heat in the condensate leaving the separating cans, which might be recovered. This water, however, is contaminated with mint oil and cannot be used as feed water in generating steam in the boiler. Some form of heat exchanger might be employed to conserve this otherwise wasted heat and thus save more fuel, or in effect, increase the present capacity of the boiler.

It is obvious in inspecting several of the boilers used in field distillation units that sufficient care is not taken to keep the boilers clean and in good operating condition. In many cases the boilers are overloaded, causing the burners to smoke. The resultant soot is deposited on the tube surfaces where it acts as an effective insulating blanket. There is a large portion of the heat available in the fuel oil which is wasted up the stack under these conditions. A definite program of flue cleaning should be established for each plant during any possible shut-down periods of the season.

Many of the fire-tube boilers are probably well scaled up on the water side of the tubes, but it is doubtful whether it would be economical to add any chemical to the feed water to attempt to remove the scale for two reasons: first, any such chemical might affect the quality of the mint oil produced, since the steam comes in direct contact with the oil; second, the expense of such treatment, even if effective, would probably not be justified during the short period of operation of a single season.

III. PILOT PLANT DESIGN AND CONSTRUCTION

1. **Need for a pilot plant.** On the basis of the tests on the Helms plant and several others, it was obvious that many desirable tests could not be adequately or safely performed in the regular field distillation unit. Harvest season is always a rush affair, some stills running continuously 24 hours a day, and the regular production could not be held up to make any changes or to add instruments or special equipment. Also there was no control over the type of hay brought in; one load might be dry but with heavy stems and fewer leaves, the next quite green with many leaves, and all tubs might not be filled or packed in to the same degree. Also, during experimentation it might be possible to lose the oil from a load of hay, and with this valued at from $150 to $300, the still operator would rather have the experimenting done elsewhere. Many problems in the distillation of peppermint oil require close control of the operating procedure and conditions, as well as control of the hay originally placed in the tub.

Typical problems mentioned by growers, some of a controversial nature, are indicated below:

1. Which type of hay gives the best yield, long or chopped hay?
2. How can wet or green hay best be distilled?
3. What steam pressures or temperatures are best? What steam rates?
4. How long should the mint hay be cooked?
5. What type of condenser should be used?
6. What can be done to the separating can to prevent loss of oil in the overflow?
7. What temperature should the condensate and mint oil be for best separation?
8. Would insulating of tubs possibly cause "burning" of the peppermint oil?
9. How can fuel and labor costs of distillation be reduced economically?
10. Could a continuous still be developed which would avoid many of the troubles of the batch process now used?

2. **Design basis.** In an attempt to find the answers to these and many other possible problems arising in the field distillation of peppermint oil, it was decided to design and construct a small portable pilot distillation plant. It should be flexible enough to demonstrate directly to a grower, when set up alongside his own plant, the effect of various temperatures, pressures, or distillation times on the

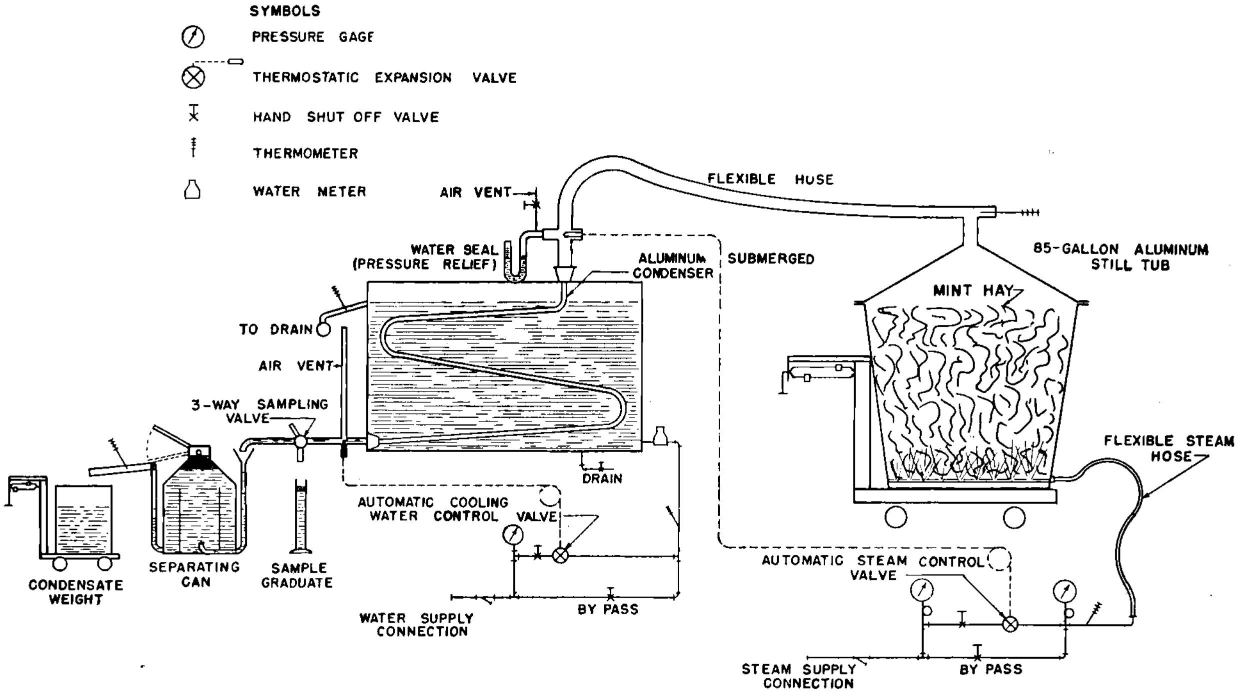

Figure 11. Schematic flow diagram of pilot plant.

amount and quality of mint oil produced. After considerable investigation, it was decided to build and mount on a two-wheel trailer an all-aluminum distillation unit having a tub capacity of about one-thirtieth of the full-sized field tubs, or approximately 12 cubic feet. The tub is 30 inches in diameter at the top and 30 inches high, and holds enough mint hay to produce about one pound (roughly a pint) of oil, which is enough to measure accurately, yet not so much as to risk the spoilage of any great quantity of hay or oil should some trial runs turn out badly. A schematic flow diagram of the pilot plant is shown in Figure 11.

An all-aluminum submerged condenser was designed to be similar to the existing larger ones, but made to match the small tub and provide for the possible use of various instruments, controls, and baffle arrangements. The discharge of condensate and mint oil from the condenser was arranged to go to a conventional-type separating can scaled down to one-thirtieth of full size, with provisions to take samples at any time.

Since the portable pilot plant operates only in the laboratory or out near a field distillation unit, where steam and cold water are readily available, provisions were made through the use of flexible hoses to furnish these items directly to a control panel where the operator can control and measure everything going into or out of the pilot plant. The tub was mounted on platform scales so that any change in weight during the process could be noted.

3. **Laboratory tests.** In connection with the design of the pilot plant, it was felt desirable to obtain as much information as possible concerning some of the physical and thermal properties of peppermint oil. Typical published analyses include solubility in alcohol, specific gravity, optical rotation, refractive index, esters, menthol, and menthone, all of which appear to have little to do with the distillation process or the design of distillation equipment.

After searching for some time through published literature, it was apparent that very little had been done to determine some of the physical and thermal properties of peppermint oil which should be considered in the design and operation of a field distillation unit. Of interest in this connection would be viscosity, specific heat, solubility in water, demulsibility in water, heat of vaporization, vapor pressure, distillation curves, oxidation, and corrosiveness in contact with certain metals. Samples of both "prime" and "redistilled" or "secondary" oil were obtained for these tests.

For those who are interested in the way in which these laboratory tests were conducted, the procedure and results will be found in the Appendix of this bulletin.

4. **Method of construction.** Considering the corrosiveness of peppermint oil, particularly in condensers, it was decided to make the pilot plant an all-aluminum unit. While stainless steel would have been somewhat better in corrosion resistance, it is more difficult to fabricate in the shapes desired. Also, at the time of the pilot plant construction, it was more expensive and harder to obtain than aluminum.

Through the courtesy of the Cobb Manufacturing Company of Jefferson, Oregon, the various parts of the 85-gallon tub and submerged condenser were welded using a shielded arc process with very satisfactory results.

To facilitate loading and unloading of the hay samples, the tub was mounted in trunnions so that it could be lowered to the ground for unloading and loading, then lifted back on the scales during the cooking period.

A flexible hose was used to connect the top of the tub with the condenser to avoid the use of water seals, which are rather bulky and messy. It was also planned to try cooking the hay under vacuum and under pressures, either of which would "blow" a water seal.

In order to be able to obtain a sample of the mint oil and steam condensate leaving the condenser at any time during the test runs, a three-way sampling valve was installed in the line between the condenser and the separating can.

Air vents were provided to remove air from the tub and condenser, as indicated in Figure 11. The one above the condenser has a valve in it which should be closed by hand when the "break through" starts during a run, but the air vent on the lower condenser header can be left open to the atmosphere at all times.

5. **Selection and operation of instruments and controls.** The purpose of the pilot plant had to be considered in selecting the instruments and controls for use with it. In principle, the plant is not any different from many of the larger field distillation plants, but it was decided to have it completely instrumented so that all pressures, temperatures, weights, and rates of flow could be determined accurately during each test as desired. In addition, the plant was piped with alternate automatic control valves, so that both manual and automatic operation could be demonstrated. As can be seen in the schematic flow diagram shown in Figure 11, an automatic condenser cooling water control valve was installed with a manual by-pass valve around it to permit manual operation when desired. A view of the actual unit with its controls is shown in Figure 12.

It was felt that some automatic method of "cutting back" the steam pressure after the load had completely heated up was desir-

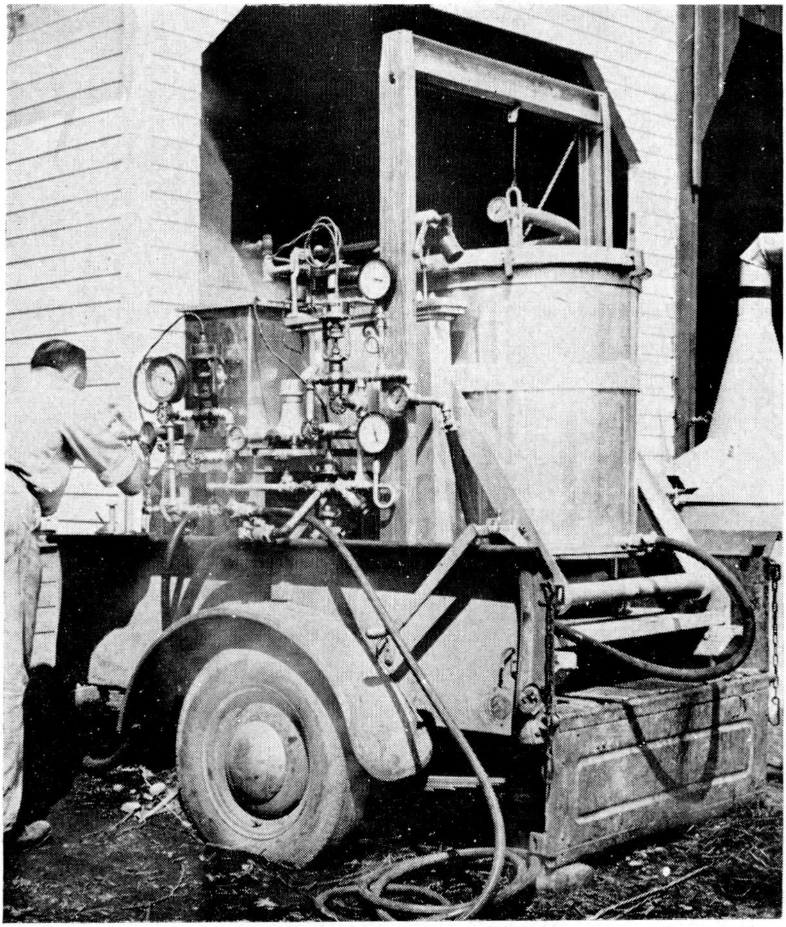

Figure 12. Pilot plant in operation at Dale Turnidge farm, Jefferson.

able, and for that purpose another automatic valve was installed in the steam line to the tub with the control bulb located in the vapor line between the tub and condenser. More test work has yet to be done on this control valve to determine the proper setting of the valve and location of the control bulb.

6. **Tests on baled hay.** Because of the unavailability of certain parts and controls desired for the operation of the pilot plant, it was not ready for operation before the 1950 harvest season was over.

However, through the fine cooperation of Mike Helms of Jefferson, Oregon, arrangements were made to let the cut mint hay in a portion of one fairly uniform field become very dry before baling it. The baling process with the ordinary pick-up baler doubtless caused some shatter loss, but 43½ bales were formed and stored at Oregon State College until such time as the pilot plant was ready for operation and the personnel were available to run the tests on the baled hay.

The baled hay was weighed in at 2,820.5 lb on September 11, 1950, or an average of 65 lb per bale. The storage room was heated to normal temperatures, and it was almost three months before the tests with the pilot plant were begun. In all, 38 different test runs were made using the baled hay during a period of about five weeks, each bale being weighed just before it was placed in the tub. When it had all been weighed in, the total was 2,654.8 lb, a loss of 3.8 lb or 5.8 per cent average weight loss per bale. Most of this was undoubtedly water vapor, since the total oil content was in most cases less than 1 lb per bale. However, at the same time one could not help believing there was some loss of peppermint oil, too, as the air in the storage room was always quite pungent.

Incidentally, baling of peppermint hay does not appear practicable on a commercial basis, because of the extra cost of baling, the shatter loss due to handling of very dry hay, and the evaporative losses which would occur during storage. Even though great precautions were taken, spots in the middle of several bales became quite moldy; these portions were set aside and distilled separately after the regular runs were made to avoid contamination of the rest of the oil. All of the oil obtained from the stored baled hay was quite dark, evidence that some oxidation took place also during the storage period.

While the tests on the baled hay were in no sense attempts to duplicate field conditions, they did provide an excellent opportunity to try out the pilot plant, train personnel to operate the plant, and calculate its performance and possibilities. Many variables such as steam inlet pressure, cut-back pressure, time of cut-back, cooking time, condensate temperature, cooling water temperature, and location of control instruments were effectively experimented with. And with the hay of almost uniform dryness, that usually important variable was practically eliminated. It can be said that the use of the baled hay saved the necessity of waiting almost a year before the next crop would become available.

A standard tub load was set at 75 lb of the dry hay, made up of thin sections from about 1¼ bales, carefully trampled in to avoid channeling of the steam through the load. Being very dry, the "break-through" time was quite short—from 4 to 6 minutes—and the cooking time could have been cut down considerably, but most of the runs were 60 to 75 minutes long to be sure that all the oil was removed from the hay. Here again, the cooking time was measured from the time the steam was first turned on.

The yield varied from 0.896 to 1.215 lb of oil per tub of 75 lb of dry hay, with a total of 37.01 lb of oil obtained from the 43½ bales of hay. In several cases where the yield appeared low, the hay was dumped out, stirred up, and cooked again. Each time, however, the added yield due to recooking was disappointing, and would hardly justify the additional steam, labor, and time required. The loads giving lower yields invariably were those having a higher percentage of stems and less of leaves, although the initial weights were all the same.

Results of the tests on the baled hay indicated that a considerable saving (up to 25 per cent) in the quantity of steam required to cook the hay, and a saving of at least half the cooling water normally required can be accomplished by dropping the steam pressure after the load has become thoroughly heated. Automatic control of the cooling water was used throughout the tests, holding the condensate between 100 and 120 F, thus providing rapid and complete separation of the oil from the water in the separating can. The outflow of condensed water from each run was carefully weighed to determine the quantity of steam used, and also visual inspection and tasting indicated very little carry-over in this outflow water. In fact all of this outflow was stored in a large closed can for six months to see whether any appreciable quantity of oil would rise to the surface in that period of time. After that period the water still gave off quite a strong odor, but only a very thin film of oil had risen to the surface. This small quantity of visible carry-over is evidence that keeping the mixture warm while the separation is taking place materially reduces the loss of prime oil which would otherwise go down the drain, or be caught as lower grade oil in the redistill.

IV. FIELD TESTS WITH PILOT PLANT

1. **Selection of problems for investigation.** Under Section III-1, typical problems suggested by growers, still operators, and others have been listed. It was difficult to select the ones to work on, since there were so many and the season is so short. Some problems, also, did not lend themselves to solution by use of the pilot plant, being more appropriately done on full-scale installations.

However, attempts were made to determine answers to the following problems: long vs chopped hay, effect of time of drying, and effect of partial loads of hay.

2. **Long vs chopped hay.** Tests on long vs chopped hay were made through the cooperation of Dale Turnidge near Jefferson, who furnished the hay samples, and the steam and water required to operate the pilot plant.

Two sets of test runs were made; in each case, hay from two adjoining windrows in the field was used. The long hay from one windrow was placed directly in the pilot plant tub and distilled; the hay in the adjoining windrow was put through the regular chopper into a large tub, then transferred to the pilot plant tub and distilled immediately. The samples of long hay weighed 86 and 154 lb, while the chopped hay samples weighed 179 and 270 lb respectively. The first set of samples indicated 5.4 per cent more oil per ton of long hay, and the second set indicated 4.7 per cent more oil per ton of long hay. From these tests, it would appear that, based on the original weight of the hay, long hay produces about 5 per cent more oil than chopped hay under the same test conditions.

These figures check quite closely with the experience of growers who had made similar tests of their own on full-sized loads in the field. More test samples should be run before the evidence could be considered conclusively in favor of long hay. However, there is another side to this controversy which each grower or still operator must consider. On the basis of experience with both the smaller pilot plant and with larger field distillation units, from 50 to 100 per cent more chopped hay can be packed into the same size tub. Since the cooking time does not seem to vary appreciably for either long or chopped hay, and the long hay requires considerably more labor to handle it, the reduction in overhead costs per pound of oil produced may well indicate that chopping produces oil more cheaply. In other words, the answer to this question depends on several factors outside of the distillation process itself. Another factor to be considered is the ease of spreading the spent chopped hay back on the fields after distillation; the long hay "slugs" are very difficult to break up and spread out.

3. **Effect of time of drying (or curing).** In connection with the determination of the best curing time, it was decided to take two sets of samples of long and chopped hay and subject them to different drying (or curing) times out in the field before distillation. It was thought that this might show the "best" curing time to produce the greatest yield of oil.

This series of tests was made on the hay from two parallel windrows of equal length in one of Dale Turnidge's fields. The long hay from one row, weighing 1,627 lb, was weighed out in 8 and a fraction pilot plant tubfuls, each full tub of hay weighing 175 lb. These samples, already having cured one day, were then spread out on waterproof paper sheets in a nearby open field for further drying.

The hay from the other windrow was put through a Gael chopper and blown into a standard round trailer tub from which the smaller tub of the pilot plant was filled. The chopped hay, with a total weight of 1,680 lb, filled 6 and a fraction tubfuls at 260 lb each, and then these samples were placed on waterproof paper sheets for further drying. It was noted, however, that during the process of weighing out the samples of chopped hay, it was "heating" and turned black quite rapidly on exposure to the air.

In weighing out these samples every attempt was made to trample them in the tub with uniform density, and from the weights indicated in the foregoing, the chopped hay samples each weighed approximately 50 per cent more than the long hay samples of equal volume. Incidentally, these samples were weighed out on the afternoon of August 15, 1951, one of the hottest days of the year, and the days following were also quite dry and hot.

The results of this test are shown in Table 2, showing that at least one long sample and one chopped sample were distilled each day for 6 days, and since there were more long hay samples, these were dried up to 9 days.

It can be seen that both the long and chopped hay lost moisture at about the same rate, both losing about 50 per cent of their original weight in the samples which were distilled after drying six days. However, the long hay appeared to lose very little oil content during this curing period except for the last sample which had lain out in extremely hot and dry weather for 9 days. After 4 or 5 days the hay was abnormally dry and the leaves would easily have shattered off with any rough handling. Care was taken with all these samples to pick up any shattered leaves which were caught on the waterproof papers. This, of course, would not be the case in field operation where the hay would be picked up by a long hay loader or a chopper, and any loss due to shatter might be appreciable after 3 days of curing in warm weather.

The total oil yield for this series of chopped hay samples was much less than for the long hay, but as mentioned before, the chopped hay passed through a "heat" before it could be spread out on the waterproof paper sheets. This process no doubt caused the loss of some of the oil, and would not ordinarily occur in the regular field distillation because the chopped hay is distilled right after cutting.

The bruising of the hay during the chopping process evidently releases some of the oil in vapor form, as it usually smells of oil much stronger than long hay. However, as the chopped hay is

Table 2. EFFECT OF CURING TIME ON LONG AND CHOPPED HAY
(All samples collected August 15, 1951 at Dale Turnidge farm near Jefferson)

Sample No.	Wt when picked up lb	Date distilled	Days from pickup	Wt when distilled lb	% loss in wt	Oil yield lb	Yield per ton orig hay lb
Chopped hay							
1	260	Aug 16	1	232	10.8	1.21	9.35
2	260	Aug 17	2	205	21.2	1.15	8.82
3	260	Aug 18	3	186	28.5	1.19	9.20
4	260	Aug 19	4	177	32.6	1.21	9.34
5	260	Aug 20	5	149.5	42.5	1.05	8.06
6	260	Aug 21	6	135.0	52.0	0.96	7.40
7	120*	Aug 24	9	49.0	59.0	0.538	9.00
Totals	1,680	....	....	....	......	7.308	8.72 Avg
Long hay							
8	175	Aug 16	1	154	12.0	1.105	12.60
9	175	Aug 17	2	133	24.0	1.040	11.86
10	175	Aug 18	3	133	24.0	1.030	11.76
11	175	Aug 19	4	111.5	36.4	1.135	12.96
12	175	Aug 19	4	108.5	38.0	0.968	11.00
13	175	Aug 20	5	89.5	48.8	1.108	12.64
14	175	Aug 21	6	91.0	48.0	1.140	13.02
15	175	Aug 23	8	89.0	49.0	1.084	12.38
16	175	Aug 24	9	76.5	56.3	0.858	9.82
17	52†	Aug 24	9	24.0	54.0	0.263	10.10
Totals	1,627	....	....	....	......	9.730	11.94 Avg

* Partial load, combined with No. 17 long hay.
† Partial load, combined with No. 7 chopped hay.

handled in the field, there is not any great opportunity for this loss to occur except in the top layer of hay in the tub where it is exposed to the atmosphere. Even then the exposure is usually only for a few minutes while the hay is being transported from the field to the still.

4. **Partial loads of hay.** It was thought to be of interest to see whether the depth of the hay in the tub would affect the yield per ton of hay. First, the pilot plant tub was filled to a depth of only 6 inches with long hay which was rather soggy, not having had proper curing. Table 3 indicates the yields obtained from three different depths of hay taken from the same section of a field and all cooked the same length of time one right after the other in the same morning.

Table 3. EFFECT OF DEPTH OF LOAD.

Depth of hay in tub, in.	Wt of green hay in tub, lb	Oil yield, lb	Oil yield per ton hay, lb
6	40.5	0.112	5.49
15	120 0	0.477	7.98
30	250.0	1.107	8.85

The results obtained from these three test runs are rather surprising, and more difficult to explain. Since all the runs with different depths of hay obviously could not be made at the same time, the last sample with the 30-inch or full depth may have had some more time to dry, but it hardly seems possible that this might cause the yield per ton of green hay 6 inches deep to be only 62 per cent as much as for a full tub load.

Another interesting sidelight on the partial load experiment was that it did not take appreciably longer for the oil to be removed from a full load than for the partial loads. Possibly since the hay was rather green and soggy, the thickness of the bed did not have as much effect on the oil produced as did the condition of the hay itself. Again this brings out the point that peppermint hay is a widely variable product within itself, and these variations in the hay can more than offset the more easily controlled test procedures. It is felt, however, that the size of the load has little to do with the cooking time, since under practically all of the conditions of test runs on the pilot plant with the one-thirtieth of full size tub it took just as long to remove the oil from the hay as in the full-size field tub. The author's opinion regarding this is that the oil which comes over more slowly is that from the heavier stems of the peppermint plant, and it just takes considerable time for the oil to pass through the greater depth of plant material than is present in the leaves.

5. **The pilot plant as a redistillation unit.** In the observation of different separating cans and redistill units in operation, it became apparent that some method was needed to determine how effective these devices actually were. Sometimes under conditions which seemed to be the best possible, droplets of oil could be seen on the surface of the separator can overflow water. At other times when conditions were very unfavorable (very wet or green hay, for example, or cold condenser) there might or might not be visible oil on the surface of the outflow water.

Since it was difficult to instrument one of the grower's redistills and to control the type of inflow to it, an attempt was made to use the portable pilot plant tub itself as a redistill unit. As the tub had an 85-gallon capacity, it could handle the complete separator can overflow for a normal run, and this large body of water could then be brought to a rolling boil by bleeding steam directly into it through the regular steam connection.

In all, 10 different runs of outflow water were made in this manner and the secondary oil removed varied from 1.44 to 2.0 cc per gallon of the water. In these tests, different types of separating cans and test conditions did not seem to affect the results conclusively one way or the other. One test also was made on the outflow water from a redistill unit which appeared to be operating satisfactorily, and this water contained only 0.16 cc per gallon. However, the outflow water from the redistill separator can showed approximately 4.0 cc of oil per gallon.

The process of using the pilot plant tub as a redistill unit did not work out very satisfactorily, and further work on this problem is discussed later.

V. STUDY OF THE MINT OIL SEPARATION PROCESS

1. **Effect of velocity and temperature.** Based on the many observations made out in the field, and particularly in connection with the pilot plant experimentation, it became evident that further study should be given the separation process itself. There were times when carry-over of mint oil in the separator overflow was high without apparent reason, and during times of heavy flow when one might expect it, there was many times little or none.

If a sample of mixed mint oil and steam condensate is taken from a condenser and allowed to stand, a large part of the oil comes directly to the surface. However, in watching the graduate with a bright light behind it, the smaller droplets will be seen moving

FIG. 10
FIG. 11
TABLE 2
FIG. 17

about in more or less random but generally upward motion. It takes about half an hour for a visible clear area to develop at the bottom of the graduate. Eventually it is supposed that the mint oil would work its way to the top though there has been some indication that separation would never take place completely under these conditions.

From the rate-of-separation curve for mint oil (see Figure 18), it appears that the warmer the oil and water mixture, the more rapid the separation rate. For example, at 60 F, the curve indicates that it would take 6½ minutes for a definite line of separation to occur, while if the mixture is at 120 F, it only takes 1⅓ minutes, or one-fifth as long.

Referring to Figure 4, it can be noted that at the beginning of a run, when the flow of both mint oil and condensed steam is heavy, the temperature of the mixture was usually found to be much lower than toward the end of the run. This means that at the end of a run the separator can is full of warmer water. Since it does not usually take more than 15 or 20 minutes at the most from the time the steam is turned off a load that is cooked until a new one starts "breaking through" again, there is little time for the 100 or so gallons of water in the separator can to cool off. In many stills where the operation is completely manual, the cooling water is allowed to run through the condenser while the loads are being changed, resulting in a cold condenser. Thus the first condensate to come over is quite cold, and since the major portion of the mint oil comes over in the first 15 or 20 minutes after "break through," much of the oil has been dumped into the warm separator while the oil itself is 20 to 30 degrees colder than the water already in the separator. While this temperature difference is not enough to make the cold mint oil heavier than hot water, it seemed possible that the colder water coming over with the oil might settle to the bottom with sufficient velocity to carry some mint oil with it.

The average downward velocities in the common types of separating cans vary from about 3 to 12 feet per hour. The first figure applies to the 30-inch (100-gallon) cans when handling 1,000 lb of steam per hour, while the latter applies to the smaller 15-inch cans some operators are still using. While the upward separating velocity of mint oil at various temperatures has not as yet been determined, it did seem that dumping the cold condensate mixed with mint oil into a can of warmer water might easily cause local downward velocities far greater than the upward velocity of the finer droplets of mint oil. This would cause some carry-over of mint oil to the drain, especially during the first part of a run where the flow was the greatest.

2. **Laboratory tests with the glass separating can.** It was apparent that one could not observe what was going on during the separation process in existing separating cans. Mr. R. E. Powne, a graduate student in mechanical engineering at Oregon State College, took the separation process as the subject for his thesis in partial fulfillment of the Master of Science degree (5).

To observe the separation process more easily, he constructed a small separating can with plate glass sides so that the complete process could be seen and photographed. The model has the same proportions as a standard separator can, but is only 8 inches wide, 12 inches high, and 3 inches between the glass sides. The inlet, outlet, and oil drawing tube were placed in normal relative positions, as indicated in Figure 13.

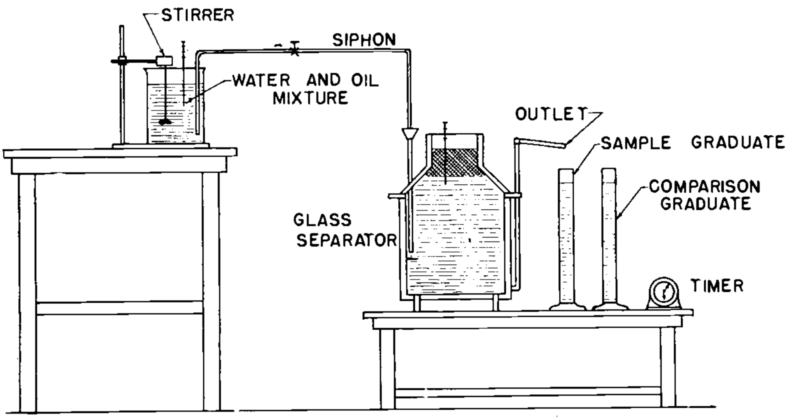

Figure 13. Test set-up using glass separator.

Peppermint oil is normally a clear, practically colorless liquid, and in order to follow the path of the oil it was necessary to obtain a dye that would photograph easily and be soluble in the oil but not in water. A dye called Sudan III, recommended by Mr. D. E. Bullis, chemist in the Agricultural Experiment Station at Oregon State College, fulfilled these requirements, coloring the oil a bright red. Thus wherever red coloring was found, it meant that mint oil was present. The red dye showed as black in the black and white pictures.

In order to feed in a uniform mixture of mint oil and water, a small electric stirrer was set up to keep it stirred. In checking over normal oil/water concentrations for a number of field stills, a mixture of 5 per cent oil in water was selected to be siphoned over into the glass separator at a rate which would give average downward water velocities in the range of 2 to 3 feet per hour.

It is difficult to describe in words, or even with still pictures, the startling results obtained by using this glass separator with varying temperatures of incoming mixtures of mint oil and water, both above and below the temperature of the water already in the separator. For that reason, it was decided to record the movement of the oil with 16-mm colored motion pictures.

For the benefit of those who may not have the opportunity to see the film, a brief word summary is included. When the mixture of mint oil and water is at a temperature equal to or above that of the water already in the separator, separation occurs quite readily, and most of the oil appears to rise quickly to the top. In a normal run on a manually controlled field distillation plant, this is the condition that occurs during the last portion of a run long after the "break through" time when the condenser is warmed.

However, when the mixture of mint oil and water enters the separator at a temperature of 10, 20, or even 30 degrees colder than the water in the separator, an entirely different phenomenon occurs. Incidentally, this condition is quite common during the first few minutes after the "break through" period and it is about this time that the maximum rate of mint flow occurs, as indicated in Figure 4. Under these conditions, the cooler entering mixture tends to settle directly to the bottom of the separator, and does this so rapidly that some of the mint oil is carried right out the drain. In the moving pictures, the incoming mixture appears as a waterfall bouncing off the splash plate below the inlet pipe and heading for the outlet pipe without disturbing the layers of water above. Naturally under these conditions considerable quantities of valuable oil are lost in the separator overflow. A still black-and-white view of this phenomenon is shown in Figure 14.

It was obvious after watching this phenomenon in the glass separator that something should be done to prevent this direct bypassing of cold water and mint oil without allowing time for complete separation. Several schemes were tried such as changing the location, size, and direction of the inlet pipe, in hope of directing the oil and water mixture toward the top of the separator can and improving the separation. However, the only solution which appeared to be effective was to place a tight baffle between the inlet pipe and the drain outlet, as shown in Figure 15. This trapped the colder mixture and forced it to go up and over the baffle before it could reach the drain, and gave the oil considerably more time to rise to the top; it also eliminated the high-velocity down currents. In addi-

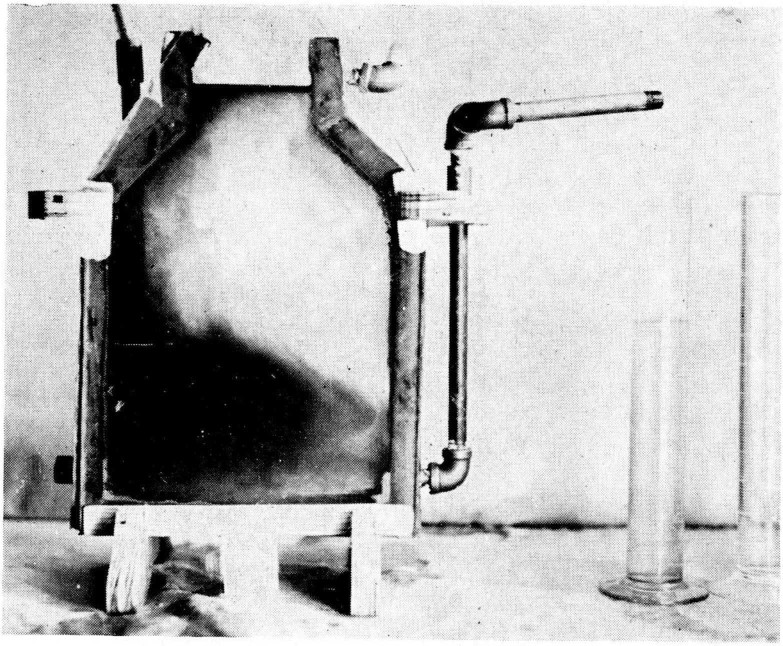

Figure 14. View through glass separating can.

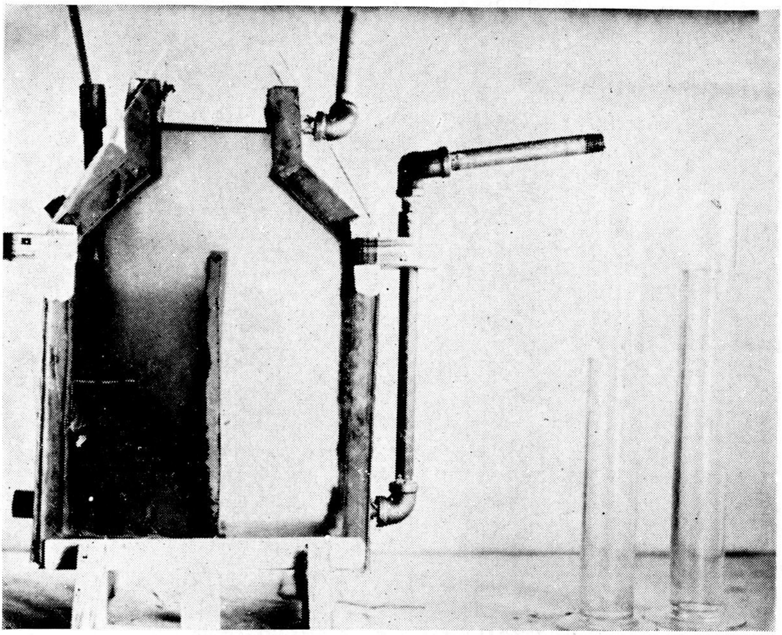

Figure 15. Effect of baffle in glass separating can.

tion, the use of such a baffle would not affect the normal (or expected) operation of the separator when the incoming mixture was warmer than the water already in the separator.

3. **Basis for design of baffled separator.** In the model glass separator, the baffle was simply a flat sheet of material dividing the separator into two sections, since the cross-section of the separator was rectangular. However, on a full-size field installation, the baffle should take a cylindrical shape, as indicated in Figure 16, with the mint oil and water mixture entering at the bottom center. It will be noted that the outside diameter of this baffled separator is increased about 40 per cent over that usually used in the normal 100-gallon separating can. This increase is for two important reasons: (1) The diameter of the inner can was selected so as to hold the quantity of cold mixture that might enter the can during the first 15 or 20 minutes of the run, thus preventing "short-circuiting" to the drain during that time. By that time in the run the condensate

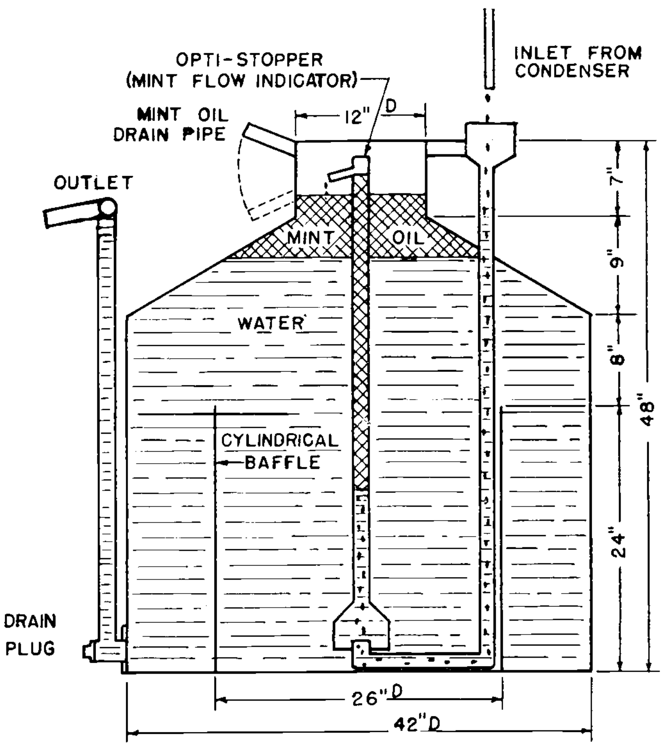

Figure 16. Sectional view of baffled separator with opti-stopper.

has usually warmed up so that separation can take place as it should. (2) Since the inner can blocks out the center portion of the whole separator can, and the flow must be upward in this section, the outer diameter had to be increased to prevent downward velocities high enough in the outer section to carry oil out the drain by entrainment.

Two separator cans of this design were constructed and used this past season, with quite satisfactory results. However, some difficulty arose in attempting to measure the effectiveness with which the improved baffled separator removed the oil from the water. This phase of the project is being investigated further. Incidentally, existing separating cans can be equipped with a baffle to prevent most of the carry-over but in a smaller can the baffle might not be quite so effective.

4. **Operation of the opti-stopper.** Many times still operators have indicated (seriously and otherwise) that some device should be invented to tell the operator when a load of hay has had all the oil removed from it; in other words, something to tell them the optimum (or best) time to stop cooking. As mentioned before, mint hay is a variable product, and dry hay may have all the oil removed from it in a few minutes, whereas green or wet hay may not be done in an hour and a half or two hours. To be safe, the still operator naturally follows a standard cooking schedule which may not be economical of steam, manpower, or equipment for all runs alike.

In connection with the development of the baffled separator, a device was worked out by the author and Mr. W. E. Phillips, an assistant on the peppermint oil project. Since it indicates the optimum or best time to stop cooking, it was nicknamed the "opti-stopper" for want of a better name.

The device (Figure 16) is very simple, and from tests made so far, seems to work very satisfactorily. The opti-stopper consists simply of an open tube with a funnel fastened to its lower end and a small spout mounted near its upper end. It is as simple in principle of operation as it is in construction. The oil and water mixture is fed in at the bottom below the inverted funnel. The oil naturally wants to rise and displace the water in the tube. Since the oil has a specific gravity of about 0.9, every 10 inches (vertical) of water outside the tube will support a column of mint oil 11 inches high inside the tube. Putting it another way, for every inch it is desired to have the mint oil rise above the top surface in the separating can, the tube must extend 10 inches down into the water below. In the usual separating can it is possible to have the oil pour out the little spout 2 or 3 inches above the top surface of the oil, and at the same time indicate the rate of mint oil flow coming from the load of hay. In other words, when the flow of mint oil stops, it is time to quit cooking, as there is no oil coming over.

The opti-stopper will in no way interfere with the normal operation of the separating can, and it is not intended that all of the oil will appear at the spout; it is merely an indicating device which will show the rate at which the mint oil is flowing. It will require a quantity of oil to "prime" it the first time it is put in operation, but this quantity of oil simply stays in the tube, and the whole unit can easily be removed at any time without affecting the normal separation process.

VI. RECOMMENDATIONS AND CONCLUSIONS

1. **Recommendations.** As a result of the many laboratory and field tests made of the distillation and separation of peppermint oil under this research project, certain recommendations are in order which will improve mint still operation in several respects. Not all of these recommendations will apply to any particular plant, but once the plant operators realize the principles involved, they can apply some of the ideas to their own plants. Nor do all of the improvements suggested involve extensive changes or the purchase of expensive new equipment; many of the recommendations indicated below are simply changes in procedure. Also in many plants some of these suggestions have already been followed with satisfaction by more progressive operators.

a. Install a pressure gage (100-lb capacity for most plants) with steam loop (or siphon) on the high-pressure steam line between the steam control valve and the tub, so that the operator will know the steam pressure going to the tub, rather than having to depend on "number of turns" of a valve handle. It should be pointed out that although boiler pressure is 100 to 125 lb per sq in., pressure at the tub is usually considerably lower because of friction in the lines and valves.

b. An air vent of at least $\frac{3}{4}$ in. size should be installed in a convenient location in the still top or on the pipe connecting the still top to submerged condensers. This will permit the venting of the air initially in the tub of hay so that it will not have to force its way through the condenser and block the passage of steam. Of course, this vent should be closed as soon as steam or mint vapor shows up, but bleeding the air may save 5 to 15 minutes on the "break through" time for each load. Such a vent does not seem to be necessary on open (or drip-type) condensers.

It has also been found helpful to install a vent pipe on the lower header of a submerged condenser. This prevents "hanging up" of the condensate during the first part of the run, and also directs the first strong vapors which cannot be condensed away from the operator. This vent should be of at least 1¼-inch pipe, and extend to the top of the condenser tank. Under certain conditions at the beginning of a run, this vent will "fog" and to some operators seem to be losing oil vapor. However, it is felt that relieving the air pressure and the noncondensable vapor presents more advantages than the small loss in the "fog."

c. Actual tests have shown that the installation of an automatic temperature control valve on the cooling water to a submerged condenser will save 50 per cent or more of the cooling water requirements, besides maintaining the condensate temperature at about 110 degrees Fahrenheit, which appears to be about the best temperature to give rapid separation of the oil without encountering heavy evaporation losses. This valve should have its stainless steel control bulb located in the condensate line where it is in contact with the condensate, and should be reverse-acting, and with a range of 100 to 140 degrees Fahrenheit. Control valves of this type should usually be one pipe size smaller than the cold water pipe line in which they are installed. A valve of this type will also automatically shut off the cooling water between runs, and provide hotter water for the boiler, thus saving fuel also.

d. A dial thermometer with a range up to 300 degrees Fahrenheit installed in the vapor line near the top of the still will permit the operator to experiment safely in the saving of steam during the distillation process. It is felt that once a load of hay has completely heated up (which normally takes about 15 to 20 minutes) there is no point in pushing a lot of steam through a load of hay when a smaller amount will bring over the oil just as fast. Tests have shown that the steam pressure to a tub can be "cut back" or reduced considerably during the last portion of a run without affecting the temperature or quantity of the mint oil vapor leaving the tub. Up to 25 per cent saving in steam has been effected on some plants by this procedure (and a corresponding saving in fuel) without any apparent reduction in oil produced. The exact procedure for this will vary for each plant, depending on the type of hay and equipment used, but with the use of the instruments indicated in the foregoing each operator can soon tell what his best procedure should be.

e. The use of a baffled separator can will reduce the losses of mint oil during the early portion of the runs where the mixture of condensate and mint oil may be cooler than the water already in the separator. Using larger separating cans gives the oil more time to separate and reduces the velocity of the water, thus preventing entrainment of oil with the water going out the drain.

f. The use of a device such as the "opti-stopper" will permit cutting down the time necessary to "cook out" the oil in a load of hay, rather than giving each load the same cooking time to be safe. In other words, when this mint flow indicating device stops flowing, all of the oil has been removed from the load of hay.

g. Boilers, steam lines, and tubs (including tops) should be insulated with at least 1 inch, and preferably 2 inches, of high-temperature insulation to prevent continuous loss of heat to the atmosphere. Particularly on a cold, windy day the steam required just to keep the line and tubs hot can be considerable, and this is a direct fuel loss. Portable tubs can be insulated using glass wool or some such light material backed by sheet metal or hardboard. If insulation is not feasible, or is too expensive, the next best thing is to paint the heated surfaces with aluminum paint; this will reduce radiation losses somewhat, but is not as effective as regular insulation.

h. Keeping a mint distillation plant clean will also pay dividends. Keeping boiler tubs clean inside and out, as far as is possible with the type of boiler involved, will save considerable fuel and increase capacity. Burner tips should be cleaned regularly during the season. Strainers on the feed water and fuel lines should be cleaned occasionally to prevent clogging. Flushing the leaves and dirt out of a condenser once in a while will produce a cleaner oil, and the installation of a piece of $\frac{1}{4}$-inch mesh screen wire in the vapor line leaving the still top will keep larger leaves from getting over into the condenser. Removing the "mother" which collects in the separating cans at least once a day, rather than letting it collect for the entire season, will improve the separation process and tend to permit the oil to separate more quickly and completely.

i. Provision should be made for "warming up" a submerged condenser before the first run is made, to prevent the first condensate flow from being too cold. This can be done by simply running a high pressure steam line into the water side of the condenser and heating the water with steam up to about 100 F before the first run is made.

j. Aluminum or stainless steel should be used wherever in contact with mint oil, as the oil in liquid or vapor form is quite corrosive. This applies particularly to condensers, where the combination of heat, moisture, and air causes galvanized iron, copper, or brass to corrode rapidly. Monel metal causes a discoloration of the oil which is not desirable. Where different metals are used in contact with each other, as in a condenser, electrolytic corrosion should be avoided wherever possible.

2. **Conclusions.** The process of distillation of peppermint oil appears to be a field in which several branches of engineering can pool their resources to improve the quantity and quality of mint oil produced as well as reduce the cost of production. The application of various mechanical instruments and controls can simplify and improve the operation of the stills, as well as reduce overhead and fuel costs.

Further study and research should be directed toward improving the condensers and separators used in mint oil distillation in order to improve their effectiveness. Many of the factors affecting the separation of mint oil from steam condensate are not entirely known, as a result of which a considerable quantity of prime oil is unnecessarily degraded to the secondary or "redistilled" condition, or lost completely with the waste water.

Mint still design and layout, and methods of handling the fresh and spent hay, could well be investigated further, particularly in the light of possible future labor costs. Many labor-saving devices, procedures, equipment arrangements, and controls used in other industries could well be applied in this field for the betterment of all.

While it is recognized that the distillation cost itself is not the major cost of producing peppermint oil, yet all the other improvements in methods of irrigation, plant breeding, control of diseases, weeds, and pests are of little avail unless the maximum quantity of high quality oil is obtained from the hay during the distillation process.

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VIII. APPENDIX

1. **Physical and thermal tests on peppermint oil.** For the benefit of those interested in the more technical phases of the distillation of peppermint, some of the physical and thermal properties of the oil not ordinarily found in published literature are discussed in this section. While this material may not be new to some, many of the tests made on the oils are somewhat unusual in their application, and for that reason detailed procedures are given. In other words, for some of the tests no standard procedures were suitable and adaptations were made to acquire the desired results. Also in a rather thorough search through the published literature available, the thermal properties which affect the distillation and separation of the oil were seldom found. From the standpoint of an engineering analysis of the process, determination of these factors was considered essential.

a. DISTILLATION TESTS. Distillation tests were intended to illustrate the wide boiling range and therefore the variation in the composition of compounds present in the oil. They were not intended for any specific purpose in connection with design or operation of distillation equipment.

The tests were conducted by directly distilling the peppermint oil in an Engler flask and measuring the vapor temperatures corresponding with volumes condensed. The resulting curves do not represent true fractional distillations, but do show the boiling ranges and relative volatilities of various oils.

The curves in Figure 17 illustrate the higher over-all volatility of prime oil as compared with secondary or recovery oil. It may be noted that little difference in volatility existed between the prime oil sample and the prime oil collected during the first ten minutes of a field distillation run, except in the higher boiling range. These results suggest that the higher boiling fractions of peppermint oil are not completely removed from hay until the distillation process has progressed for a considerable period of time.

b. DEMULSIBILITY. Demulsibility may be defined as the rate of separation of an emulsion. The emulsion with which we are concerned is that of peppermint oil in water.

These tests were run in order to find the rate of separation of the peppermint oil-water emulsions at various temperatures. It was felt that separation of oil from water in the separating can could be improved if the temperature were controlled at a value favorable to a high demulsibility.

"VOLATILITY"

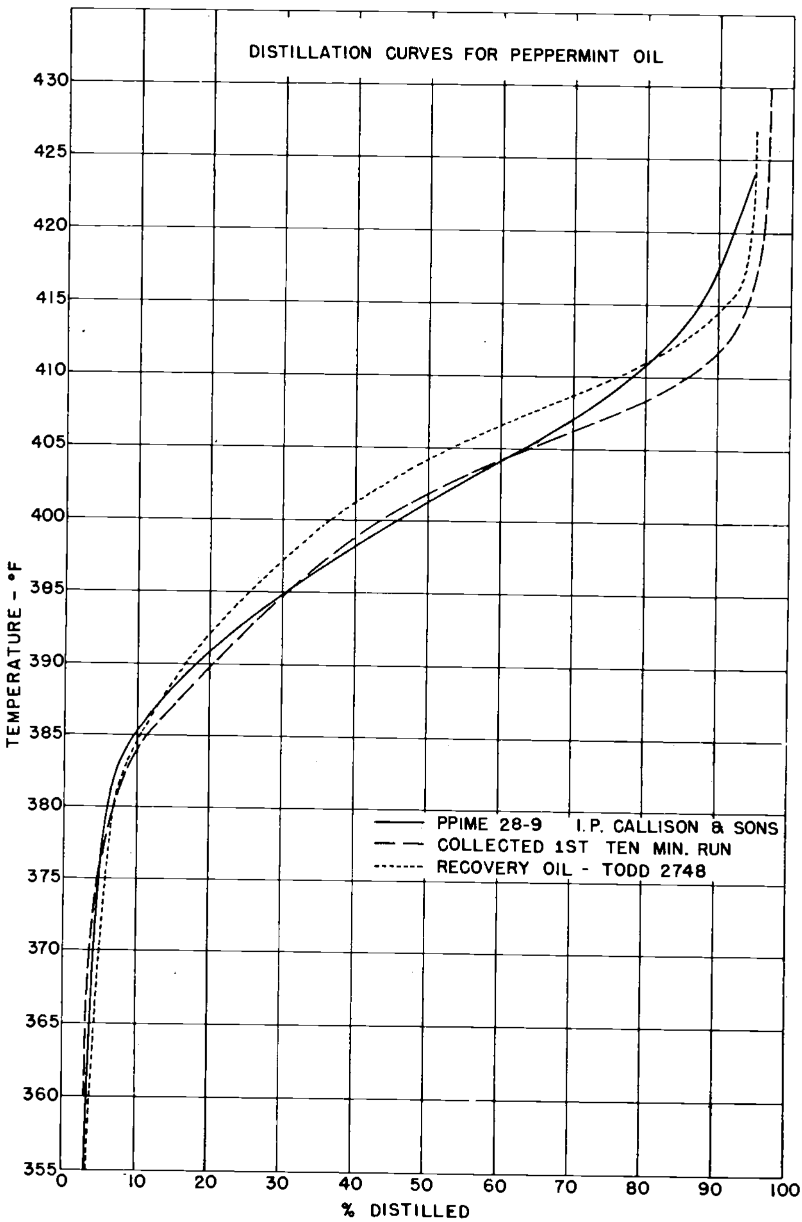

Figure 17. Distillation curves for peppermint oil.

The demulsibility experiments were conducted according to Federal Specification VV-L-791c, Method 32032, with minor variations. The method referred to is designed for the determination of the demulsibility of lubricating oil in water, but proved basically suitable for this work. In brief, it consisted of agitation of volumes of oil and water mixtures in graduated cylinders for a fixed period of time and observing separation time.

It was found that separation improved as temperature increased according to the relationship shown in Figure 18, which indicates very definitely that control of separation temperature is necessary to reduce carry-over of oil particles in water leaving the separating cans.

c. VAPORIZATION LOSS AT VARIOUS TEMPERATURES. The vaporization tests were performed at various temperatures to check roughly the vaporization rates of peppermint oil at atmospheric pressure. It was felt that since results of demulsibility tests indicated improvement in separation as temperature increased (although with decreasing effect), definite data should be obtained to show that operating with high condensate temperatures would result in loss of a considerable amount of peppermint oil through vaporization. The tests were run simply by placing a volume of peppermint oil in a graduated cylinder which was held in a mineral oil bath controlled at a selected temperature for one week. At the end of one week the reduction in volume of peppermint oil was recorded and the test was repeated with a fresh sample at a different temperature.

The results obtained were not quantitative in a strict sense but did serve to assist in fixing a recommended condensate temperature range. Figure 19 is included to illustrate the effect of temperature upon vaporization rate of peppermint oil.

The oils selected for this test were not intended as specific examples, but were intended to show in general the differences ordinarily existing between the properties of prime oil and redistilled or secondary oil.

d. VAPOR PRESSURE DETERMINATION. The vapor pressure of peppermint oil was determined so that steam distillation calculations at various distillation pressures could be made. It is proposed to study, at a later date, the effect of vacuum steam distillation on economy of operation and on oil quality. The vapor pressures were determined by evacuating a flask and measuring pressure and temperature. The thermometer was fitted with a wick which was continuously supplied with new oil. At the reduced pressure the thermometer registered the boiling point of the oil at the pressure of the system.

The vapor pressure of prime oil is only 4.0 mm Hg at 250 F, and for redistilled oil 3.3 mm Hg at the same temperature by this method. The latent heat of vaporization of peppermint oil was calculated from the slope of the prime oil curve at about 212 F which is the steam distillation temperature at normal atmospheric pressure.

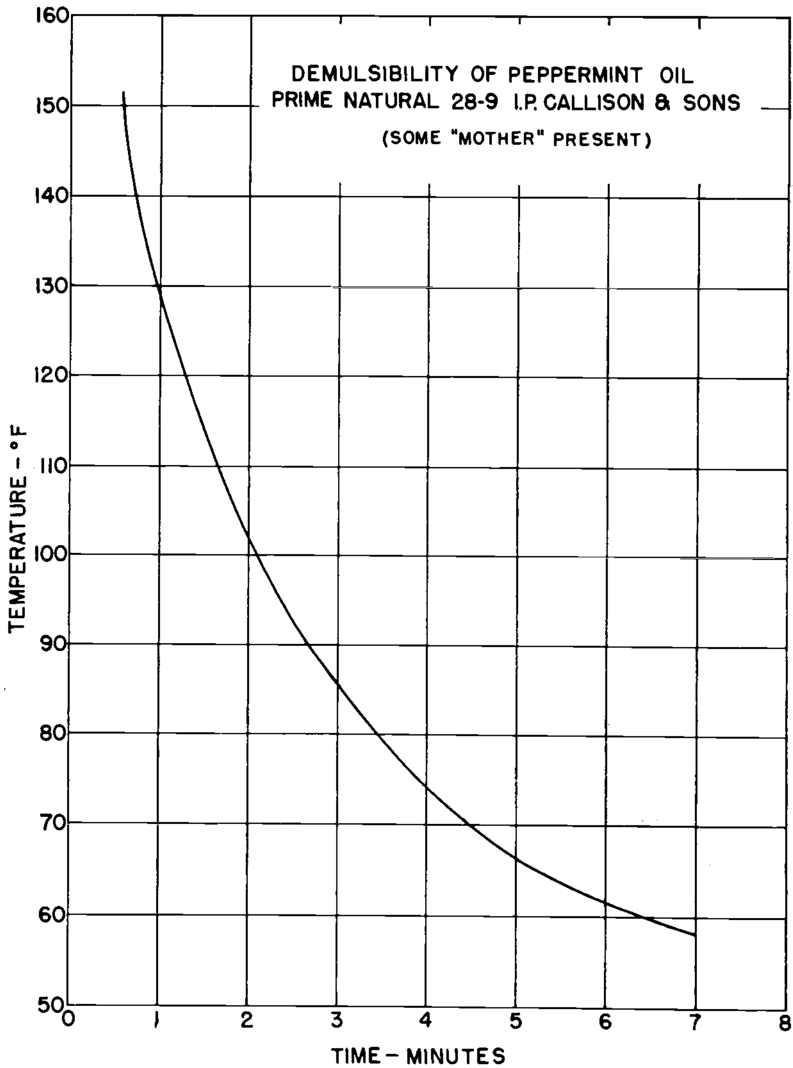

Figure 18. Demulsibility of prime peppermint oil in water.

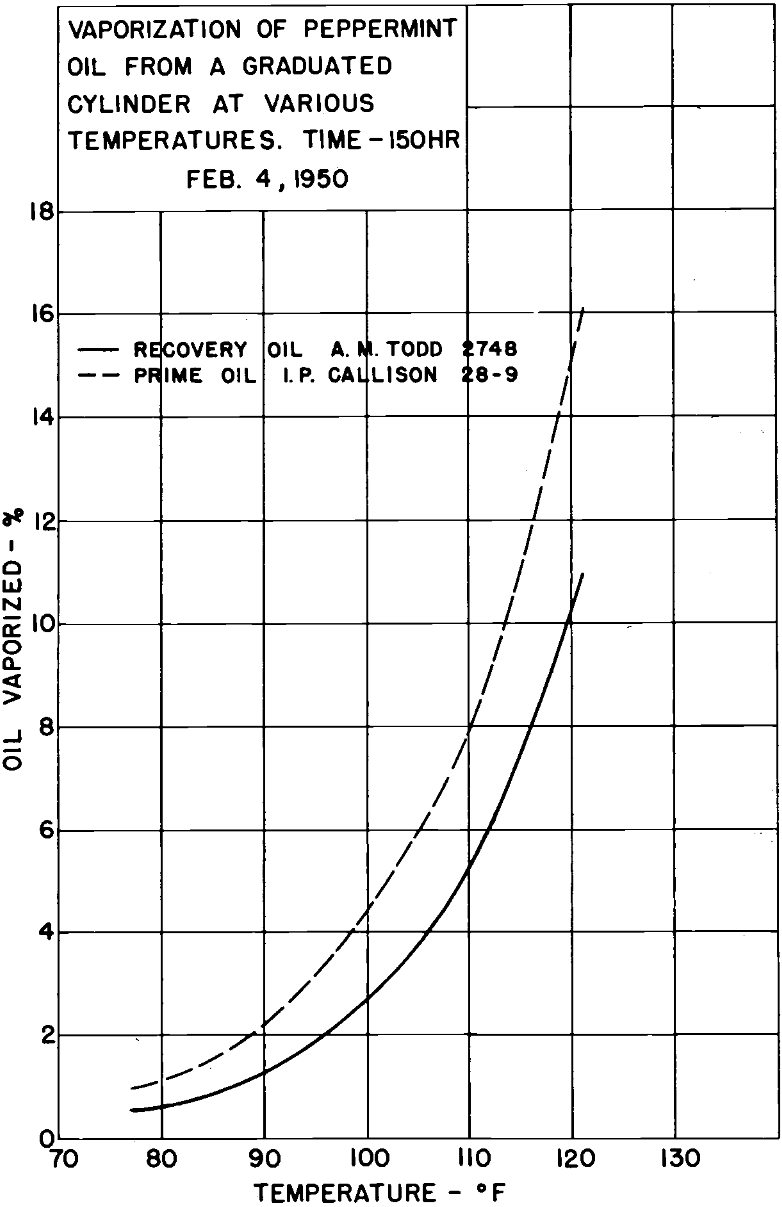

Figure 19. Vaporization loss of peppermint oil at various temperatures.

The calculated result was 115 calories per gram or 207 Btu per lb, assuming the average molecular weight of peppermint oil to be the same as that of menthol.

In this connection, it might be well to point out that the heat of vaporization of steam at atmospheric pressure is about 970 Btu per lb, and that in both the tub during steam distillation and in the condensation process in the condenser there is much more steam than mint oil present. Test runs have shown a minimum ratio of about 10 lb of steam per lb of mint oil, but most of the time the ratio is much greater than this. Thus as far as heat addition or removal is concerned, the heat of vaporization of the oil itself is insignificant.

It was noted that the vapor pressure of prime oil was considerably higher than that of recovery oil, which agrees with the results of the distillation and the vaporization tests. This fact reflects again the higher volatility of the prime oils and therefore a difference in composition of prime and secondary oils.

e. VISCOSITY. In an attempt to determine the factors which affect the demulsibility or rate of separation curve, tests were run on a sample of prime oil as supplied by the A. M. Todd Company according to the ASTM Procedure, "Test for Kinematic Viscosity," (D-445-46T) Method B. A modified Ostwald viscosimeter was used. The results of the tests from 60 to 180 F are shown in Figure 20, along with the established values for water for the same temperature range. Although water is much less viscous than peppermint oil, that is not so significant as the fact that the viscosity of peppermint oil has a much greater variation over the same temperature range. With the viscosity decreasing as the temperature increases, the curve takes a shape very similar to the demulsibility or time of separation curve, and the change in viscosity might well be the reason for the shape of the demulsibility curve. Visual inspection corroborates this, for at lower temperatures the oil collects in heavy globules which seem to have considerable difficulty in combining with each other as they rise to the surface. In warmer water, the mint oil globules are usually much smaller and more active, and combine with each other quite readily.

This leads one to believe that there also is a considerable variation in surface tension with temperature. Although this property was not determined analytically, surface tension for practically all liquids decreases with an increase in temperature.

f. SPECIFIC GRAVITY. As mentioned before, peppermint oil being a complex organic liquid obtained from natural sources, its physical properties are quite variable. Specific gravity of prime oil, according to the standards of USP XIII Peppermint Oil, may vary from 0.896 to 0.908 corrected to 25 degrees Centigrade (77 F). However, tests made on samples of prime oil indicate that the specific gravity decreases about 4 per cent over the range of 60 to 150 degrees Fahrenheit. Water, on the other hand, decreases about 2 per cent between those same temperatures. This decrease in density at higher temperatures also would affect the time-of-separation curve, as lighter oil would be expected to rise more rapidly.

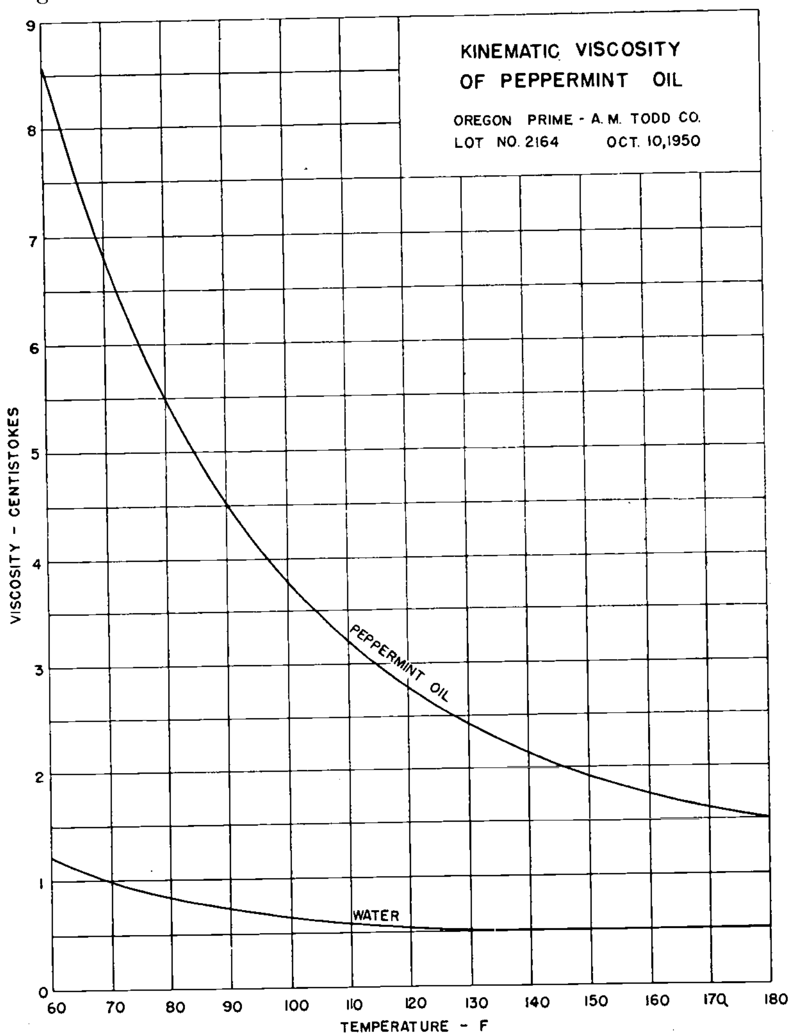

Figure 20. Viscosity of peppermint oil.

g. CENTRIFUGAL SEPARATION OF PEPPERMINT OIL FROM WATER. In order to eliminate the present redistilling or recovery process which produces an inferior grade of oil, the centrifugal separation of the oil and water was attempted. It is not certain that the centrifugal separation of the oil from the oil-water mixture which is normally redistilled will produce prime oil, and actual field investigation will be necessary to confirm the belief that "redistilling" oxidizes the oil and reduces its quality.

In the experiment a DeLaval No. 202 Oil Clarifier, a continuous three-bowl type separator, was used. The oil and water were mixed continuously in a 100-ml beaker equipped with a stirrer. The oil-water mixture was added at the maximum capacity of the separator, about 50 gal/hr. The separator was operated continuously, oil recovery measurements being taken as soon as steady state conditions had been reached. It was found that 20 to 30 per cent of the oil was recovered from a 0.1 per cent concentration of oil in water and that 10 to 20 per cent of the oil was recovered from a 0.05 per cent concentration, but that recovery was insignificant below the 0.05 per cent concentration. It is felt that the average concentration of peppermint oil in the water leaving the separating can is so low that further investigation of centrifugal separation appears unwarranted. This matter will be investigated further, but depends first upon finding a reliable method of measuring the amount of peppermint oil in the separator outflow.

h. SOLUBILITY OF PEPPERMINT OIL IN WATER. Solubility determinations at various temperatures were made because it was considered possible that oil was being carried out of the separating cans in solution with the water. It was felt that if it could be determined that peppermint oil is less soluble in water at some particular temperature, separating temperature could be controlled at a level which would insure improved oil recovery. Test procedures were based on simple volumetric methods using a 50-ml Cassius flask, but solubilities throughout a practical temperature range were so low that differences could not be detected. More exact methods of solubility determination such as refractive index or optical activity could be used, but the results obtained at the time indicated that solubility variation with temperature had a relatively minor effect on separation, and therefore these experiments were discontinued. On the basis of more recent field tests of separating cans, however, it appears that the solubility in water, though very low, still is significant.

One method recently developed for measuring the small quantities of peppermint oil in large quantities of water is that of using a 1,000-ml sample of the mixture, such as taken from the overflow of a separating can, and distilling it in a 2,000-ml erlenmeyer flask. The mint oil in this procedure is caught in a reflux condenser and trap, such as is ordinarily used to measure quantities of water in samples of crankcase oil. Quantities of peppermint oil of the order of from 5 to 15 ml (or cc) per gallon of original sample have been obtained from water which had previously been thought free of oil. While this appears to be an insignificant percentage, ranging from 0.13 to 0.40 per cent, it has been calculated that for a still with three tubs running on a 24-hour basis, approximately fifty dollars worth of valuable oil per day could be lost in the separator overflow.

There is some question also as to whether these small quantities of oil are actually in solution with the water or are simply in such a finely divided state that they would not rise to the top in any reasonable length of time. The answer to that question, however, is academic, for if the oil is being lost in the separator overflow, the important thing is to prevent the loss, if it can be done economically.

As stated before, this report is a progress report, and further research is being aimed toward minimizing this separator loss. Although the use of a redistill appears to collect some of the oil which would otherwise be lost, yet the apparent necessity for a redistill is simply evidence that the first separation process for the prime oil was not completely effective. Every effort will be made to improve the prime oil separation process so that the use of a redistill may not be necessary.

i. CORROSION TESTS. The corrosivity of peppermint oil-water mixture on various metals was determined to assist in choosing proper materials for still construction. These tests were run on materials in use at the present time and on other materials that might possibly be used. The tests consisted of placing the metal samples in test tubes along with 4 cc of peppermint oil and 12 cc of water and maintaining the temperature of the tubes at 180 F by means of a controlled mineral oil bath for a seven-day period. Air saturated with water vapor was bubbled through the test tube solutions to simulate conditions present in the condensers where most of the corrosion appears to take place. At the end of the test period, the metal samples were cleaned and reweighed, the difference between original and final weights indicating degree of corrosion.

From the tests it was apparent that copper, brass, ferritic iron, copper-bearing mild steel, Corten mild steel, cold-rolled mild steel, and galvanized steel should not be used since these metals corrode severely, and/or impart color to the oil. It also was noted that the presence of corrosion products in peppermint oil from the above metals greatly reduced demulsibility (or reduced the rate of separation) of the oil from water. Monel metal darkened oil and reduced demulsibility even though corrosion was low compared to the metals mentioned previously, and it cannot be recommended.

Three aluminums, 61-ST, 2-S, and 3-S, were about equal in corrosion resistance and, in general, should be very satisfactory as still tub, condenser, and separating can materials. Occasionally, a definite deleterious effect on demulsibility was indicated during tests. However, the laboratory tests were quite severe as compared with actual service.

Stainless steel (18-8) was the best material tested from the standpoint of corrosion, oil discoloration, and demulsibility reduction. It might be well to mention that mixing of materials in tubs, condensers, or separating cans easily might cause electrolytic (or battery-type) corrosion. In other words, if a condenser is to be made of aluminum, it should be made completely of that one metal to avoid possible pitting or disintegration due to electrolytic action.

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LIST OF PUBLICATIONS

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