



INSTALLATION AND EVALUATION OF
AN EXPERIMENTAL PEPPERMINT
OIL DISTILLATION PLANT

THESIS FOR THE DEGREE OF M. S.
MICHIGAN STATE UNIVERSITY

WILLIAM HENRY KAHL
1955

THESIS

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INSTALLATION AND EVALUATION OF AN
EXPERIMENTAL PEPPERMINT OIL
DISTILLATION PLANT

By

William Henry Kahl

AN ABSTRACT

Submitted to the Michigan State University of
Agriculture and Applied Science in partial
fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

Department of Agricultural Engineering

Year 1955

Approved

Carl W. Hall

8/12/55

This investigation consists of the installation of an experimental peppermint oil distillation plant and the determination of the consistency and efficiency of oil recovery by the apparatus.

The amount of peppermint oil produced on experimental plots and also commercially can be divided into two main phases, (a) the cultural practices, and (b) the distillation process. The distillation process must be highly efficient and consistent before the effect of cultural practices can be determined. Thus, the reasons for the study are:

1. To install a precision experimental peppermint oil distillation plant.
2. To evaluate the efficiency and consistency of oil recovery by the distillation plant.

The installation includes four basic pieces of equipment, (a) a boiler to provide steam, (b) tubs to receive the peppermint hay, (c) condensers to liquify the mixture of steam and peppermint oil vapor, and (d) receivers to separate the water and oil distillate.

The evaluation of the efficiency and consistency of oil recovery was determined by processing a known amount of peppermint oil through the apparatus measuring the amount of oil recovered and plotting the results on statistical control charts.

The long term expected statistical average of the apparatus, determined from the control charts is 7.84 percent oil loss; giving an oil recovery efficiency of 92.16 percent. The consistency of recovery, also determined by the control charts, is a long term expected

standard deviation of 3.01 percent oil loss.

A fast method, applicable to the farmer, of determining the amount of peppermint oil entrained in the distillate going down the drain was investigated with negative results. Two methods were tried (a) freezing the final distillate in an attempt to separate any entrained oil from the water and (b) pH tests of the distillate compared with the pH values of known concentrations of peppermint oil and water.

Many pieces of standard equipment, easily available to the farmer, were used successfully in the installation of the equipment, such items included, aluminum irrigation piping, plastic garden hose, and marine plywood. A jet type condenser was unsatisfactory creating too much turbulence in the receiver.

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To Mr. E. Moore whose farm lies adjacent to the Bath Experimental Muck Farm is extended a word of thanks for his many acts of cooperation during this investigation.

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CHAPTER I

INTRODUCTION

The distillation of peppermint oil from peppermint hay is a process in which steam is passed through the hay to vaporize the peppermint oil; the mixture of steam and oil vapor is then condensed and the resultant distillate is separated; the oil is kept by the farmer and the water disposed.

The site of this investigation is at the Michigan State University Experimental Muck Farm at Bath, Michigan. A research program on the culture of peppermint is carried on by the department of Soil Science at the Experimental Muck Farm. The end product of the peppermint crop is peppermint oil which is classified as one of the essential oils. Peppermint oil is a source of income for farmers located on muck lands. The goal of the peppermint research program is to continuously improve the production efficiency of peppermint oil producers.

The majority of agricultural products indicate efficiency of production by the weight or volume of fiber or seed produced per acre. This is not true of peppermint. A large tonnage of peppermint hay per acre does not mean a large recovery of oil. The amount of hay produced compared to the amount of oil recovered from the hay is not constant; the variance being so large that no rule can be applied. Therefore, since the amount of oil recovered per acre is the criterion of efficiency of production, to be able to compare the effect of cultural practices on experimental plots, the peppermint research program

is dependent upon the accuracy of the method used to recover mint oil from the peppermint hay. Inaccuracies beyond chance variance in the recovery of peppermint oil from the hay ⁽⁴⁾ led to the initiation of this investigation, with the primary purpose of installing precision peppermint distillation equipment.

The research divides itself into two components:

1. Installation of the distillation plant.
2. Testing the equipment after installation to evaluate the efficiency and the degree of consistency of oil recovery by the apparatus.

The departments of Soil Science and Agricultural Engineering of Michigan State University are cooperating in this investigation.

CHAPTER II

REVIEW OF LITERATURE

Hughes⁽⁸⁾ has done some of the more recent research in peppermint oil distillation. He recommends improving field distillation in the following manner:

1. Use aluminum condensers, piping and other apparatus which might be in contact with mint oil rather than galvanized iron or copper since aluminum is not corroded by peppermint oil and does not give the oil an off-flavor as these other metals. Stainless steel is non-corrosive, too.
2. Use automatic control valves on condenser cooling water in order to maintain proper condensate and receiver temperature for the most efficient separation of mint oil from the water ... 110°F to 120°F. is recommended. At this temperature less oil goes down the drain. It is also the temperature for the most rapid demulsification without too great a vaporization loss.
3. The use of steam gages, thermometers, insulation, "warming up" condensers, baffled and larger separators, and the "Opti-stopper" are recommended for the purpose of increasing the efficiency of field distillation of peppermint oil.

The work of Hughes is continuing

Guenther⁽⁵⁾ devotes a chapter, pp. 227-367, including 19 pages of references to the analysis of essential oils. The description and

detailed explanation of the analysis procedure when using the Clevenger apparatus to assay the amount of essential oil present in a given herb should be of particular interest to growers of peppermint. Self-explanatory diagrams are given of the apparatus. It is of laboratory size and will process enough material to produce two to six cubic centimeters of oil. The apparatus is compact and it is claimed an accurate evaluation of large quantities of raw materials can be made by using only small amounts of the herb.

Concerning the yield of peppermint oil per acre, Guenther⁽⁶⁾ reports yield variations of from 10 to 75 pounds with an average of 24 to 25 pounds. The proper time to cut the peppermint hay can be determined by the menthol content of the herb. The menthol content increases as the peppermint approaches maturity; when the menthol content nears 50 percent the peppermint should be cut. Guenther⁽⁶⁾ states that Swift and Thornton have devised a quick viscometric method of determining the amount of free menthol in peppermint oil.

Mentha piperita L, the peppermint grown in America contains l-menthol and l-menthone as its major constituents. Various values for the amount of l-menthol in peppermint oil are given in the literature, Steele⁽¹⁴⁾ gives 80 percent, Geunther⁽⁵⁾ 45 to 65 percent, Bullis⁽²⁾ 43 to 67 percent. Nelson⁽¹¹⁾ in assays of experiments on Verticillium wilt resistant hybrids had total menthol contents ranging from 32 to 56 percent. The U. S. Pharmacopoeia requirement for total menthol content is 50 percent. Accordingly, the loss due to the solubility of the oil in water during distillation is approximately

two percent when taking a value of total menthol content of the oil at 50 percent. Hodgman⁽⁷⁾ gives the solubility of l-menthol in water as 0.04 grams per 100 milliliters of water. Lange⁽⁹⁾ gives the solubility of l-menthol in water as 0.04 grams per 100 grams of water.

Sievers⁽¹³⁾ gives the procedure for the method of handling the oil after it is removed from the receiver. Allowing the water and oil emulsion to stand for a period of time in a separatory funnel before separation is recommended. Filter paper and dry sodium sulfate are useful in removing the final amounts of water. Water if not removed from the oil can aid in chemical changes in the oil if exposed to air. Amber-colored glass bottles completely full placed in a dark cabinet are recommended for storage.

A serious threat to the peppermint industry is *Verticillium wilt* first observed by Nelson⁽¹¹⁾ in 1924. A fungus, of the genus Verticillium, produces the wilt according to Sievers⁽¹²⁾; the organism is able to exist in the soil for extended periods making repression of the disease a problem. Development of wilt resistant varieties⁽¹¹⁾ appears to be the most promising method of controlling the disease.

Moore⁽¹⁰⁾ has an enlightening chapter on the uses of statistical quality control. The definitions and explanations are so clear and easily understood in this exposition that it is a good place of beginning for the initiate to this type of statistical analysis. Abruzzi⁽¹⁾ presents another version of the control chart, as used to analyze work measurement. The difficulties encountered by Abruzzi are apparent

when one considers the inherent subjectiveness of the data gathered,
both the timer and the timed being human.

CHAPTER III

INSTALLATION OF THE DISTILLATION PLANT

The Bath Experimental Much Farm had inadequate peppermint distillation equipment. Recovery of oil had too great a variance. The tub, condenser and fittings were of galvanized iron which is corroded by mint oil⁽⁸⁾ and gives the oil an undesirable off flavor. The capacity of the equipment was too small; 60 samples per week⁽⁴⁾ were all that could be expected. Since an adequate peppermint research program requires at least 500 plots per year⁽⁴⁾ errors will creep into experimental data due to harvesting the plots at varying stages of maturity and also due to the effects of weather on the amount of leaves lost, when the harvesting period has to be extended over too long a time.

The reason for this study is to overcome each of the above deficiencies: by installing precision distillation equipment; by increasing the capacity of the installation to 120 plots per week instead of 60; by installing two stainless steel tubs, two aluminum condensers, aluminum piping; and controlling the temperature of the condensate (110°F to 120°F.) by use of a temperature controlled valve to regulate the flow of cooling water through the condenser.

The information and recommendations given by Hughes, Oregon State College⁽⁸⁾ have been followed to a great extent during this installation.

The Boiler

The boiler is an oil-fired, vertical, fire tube boiler, with a heating surface of 219 square feet, and a working pressure of 100 pounds per square inch. It was purchased second-hand.

Figures 1, 2, 3, 4, and 25 illustrate the installation of the boiler and its fittings. Note the method of offsetting the chimney to take the stress and resultant strain off the boiler and to facilitate cleaning the tubes and making repairs.

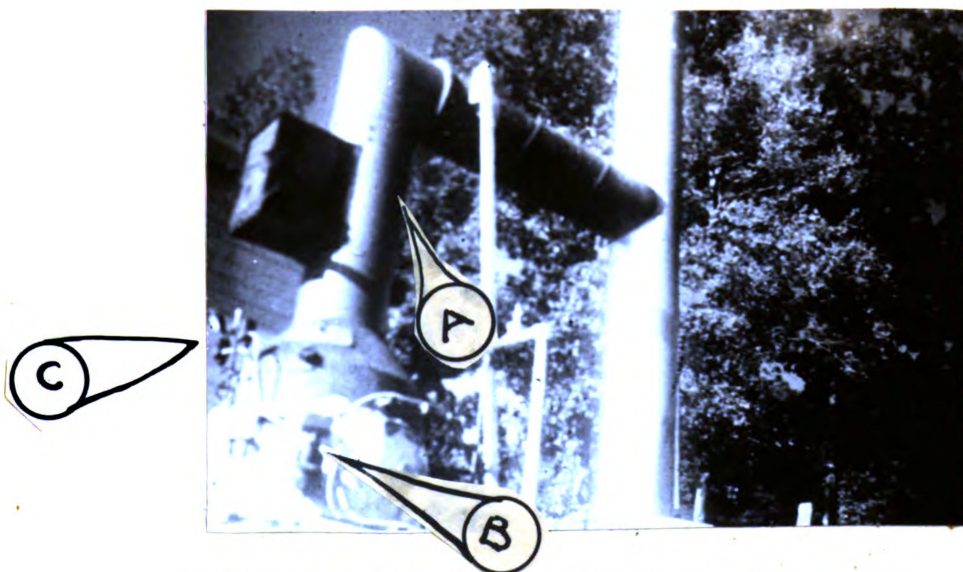


Figure 1. The Chimney in place. The uptake breeching (A) resting on the boiler can be disconnected from the other part of the chimney by removing eight cap screws. Boiler feed water (B) and pressure controls (C) can be seen on the boiler.

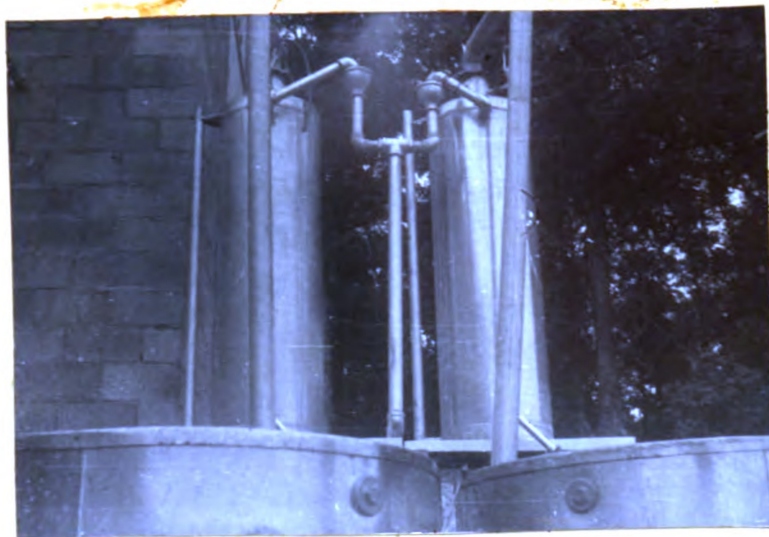


Figure 2. Boiler, chimney, tubs, condensers and four-inch aluminum irrigation piping between tubs and condensers.

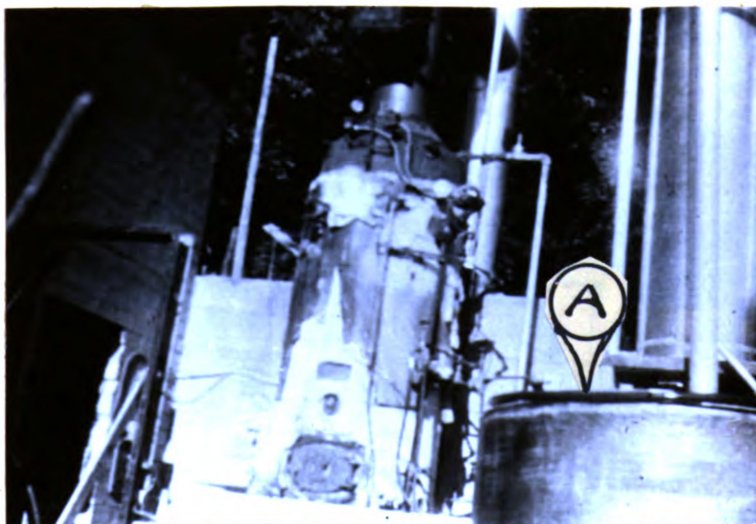


Figure 3. Start of concrete block wall to house the boiler. Note the plastic garden hose (A) on the rim of tub used effectively as a gasket.



Figure 4. The boiler housed.

The Tubs

The tubs into which the peppermint hay is placed for removal of the oil from the fiber and leaves were former dairy processing vats. They are stainless steel tubs with a capacity of 150 gallons, and will hold 500 pounds of green peppermint hay. The bottoms of the tubs were fitted with ten gage steel plates into which one-eighth inch holes were drilled to disperse the steam through the hay. The tubs have sloping bottoms, which provide a steam space between the steel plate and the bottom of the tub. The center of the plate was supported by a two inch length of four-inch diameter aluminum irrigation pipe.

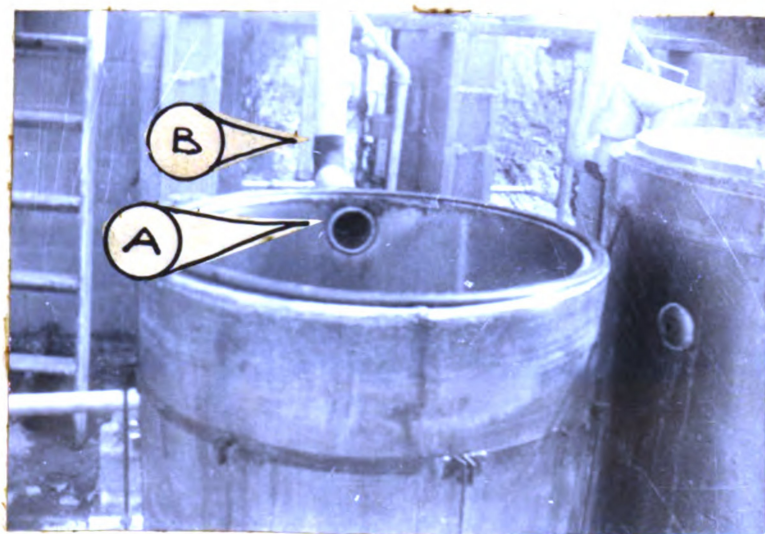


Figure 5. Steam outlet (A) from tub, the four inch aluminum irrigation pipe is malleable enough to shape the flange under which a piece of plastic garden hose was placed to form a vapor seal. The black portion(B) of the pipe locates one of the vapor leaks before repairs.

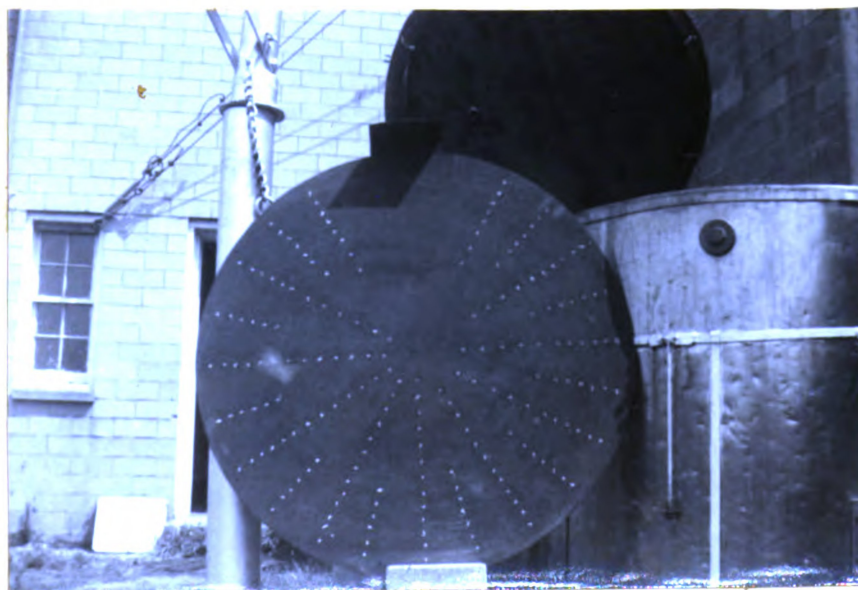


Figure 6. The steel plate which fits into the bottom of the tub. Holes are to disperse steam.

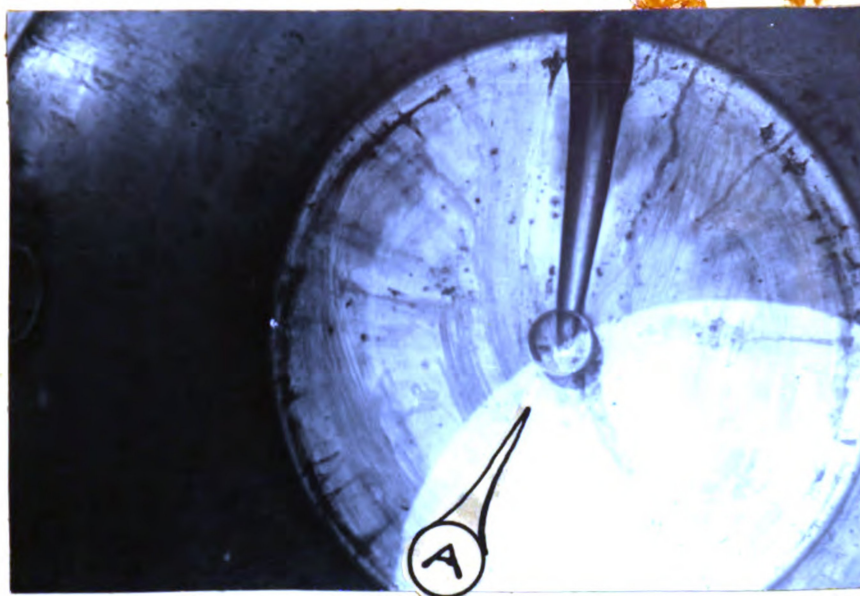


Figure 7. Bottom of stainless steel tub. Groove in bottom is steam inlet. Circular object (A) in center of tub supports center of steel plate.

The one and one quarter inch steam inlet pipe to these tubs was fitted to the regular inlet cock of the tub; this inlet valve serves a dual purpose here since drainage of accumulation of water can be done by simply lifting the plug from its seat. A steam gage⁽⁸⁾ was placed between the tub and a steam flow controlling globe valve.

A four inch outlet hole was drilled in the side of the tub, two inches from the top to provide an outlet for the mixed steam and mint oil vapor. A four inch aluminum irrigation pipe was used to conduct the vapor to the condensers.

An aluminum cover for the tubs was tried (it had a spring loaded clamping device) which could be removed quickly. It proved to be ineffective in that it allowed peppermint vapor to escape from the tub; the cover lacked rigidity causing the loss. Marine plywood three -

quarters inch thick used in the construction of the next tub covers has given satisfactory service to date and as yet they show no sign of deterioration due to the action of steam, mint vapor, or weathering. A common vinyl plastic garden hose cut to the proper length has proved an effective seal between the cover and tub. Gaskets cut from this plastic hose have made tight seals for the temperature and pressure fittings placed on the apparatus, and the joints at the tub outlet and condenser inlet.

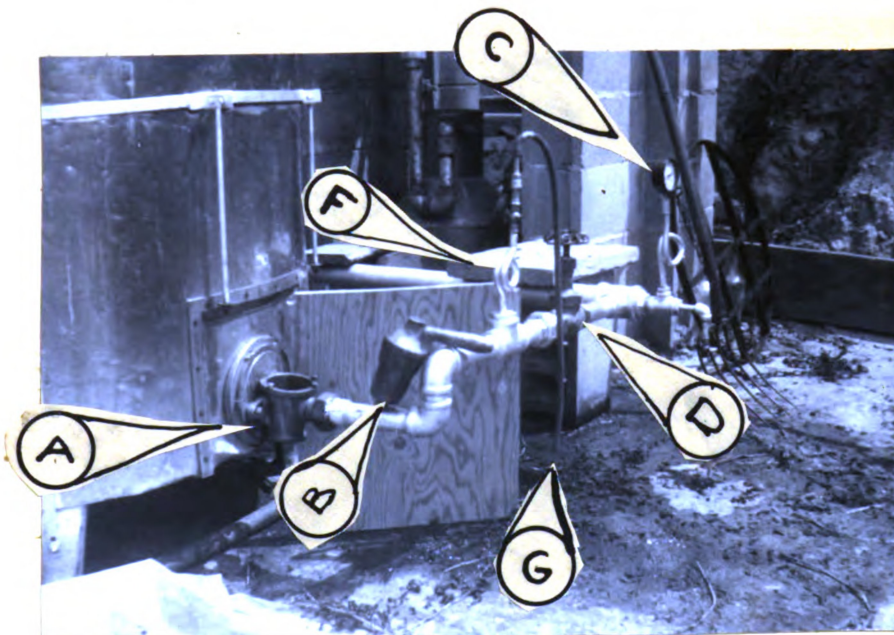


Figure 8. Steam inlet to tub (A), inlet cock (B) is removed from its seat. The steam header pressure gage (C) is at the right. The steam flow controlling globe valve is shown (D). Between the globe valve and the inlet cock is a fitting for another steam gage (E), pressure here was measured by a manometer connected to the end of the hose (G). The manometer would read six inches of water under normal operating conditions, that is, 300 pounds of steam per hour and a header pressure of 20 pounds per square inch gage. The reduction in pressure takes place at the globe valve.



Figure 9. Photograph of marine plywood cover taken in January 1955. The lid has been exposed to the weather and has been used with steam on 176 mint distillation trials. As yet their is no sign of deterioration except a discoloration of the wood. To the right is the condensate temperature control valve (A) and its sensing elements (B).

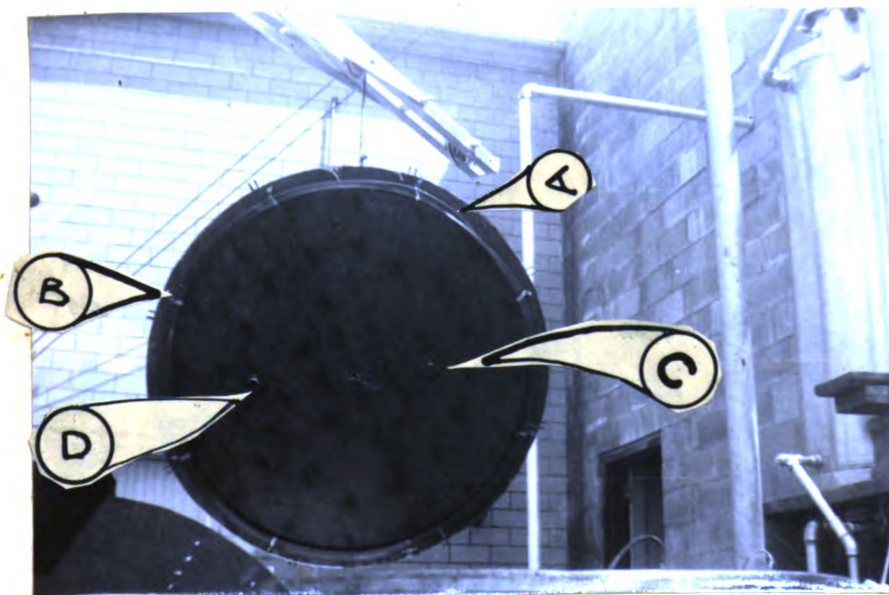


Figure 10. Method of initially fastening the plastic garden hose (A) to the marine plywood cover. Pieces of

Figure 10 (Cont.). plastic clothesline (B) (inner cord-
ing removed) were wrapped once around the gasket and
screwed to the cover. After the gasket had flattened
from use it was nailed (aluminum) along its outer edge
to the cover. Fittings for the pressure gage (C) and
thermometer well (D) can be seen on the bottom of the
cover.

The Condensers

The two aluminum vertical straight tube condensers⁽⁸⁾ were
purchased from the Cobb Manufacturing Company, Jefferson, Oregon
each has a capacity of 1200 pounds of steam per hour. The tempera-
ture of the distillate (110°F to 120°F.) is controlled through a
sensing element in the condensate drain pipe by a bellows type con-
trol valve,⁽⁸⁾ manufactured by the Robert Shaw--Fulton Controls Com-
pany, Knoxville, Tennessee, which regulates the rate of flow of the
condenser cooling water. Three concrete block piers support the
condensers; the condensers rest on two inch planks which span the
distance between the piers.

A jet type condenser was tried using a garden hose nozzle to
regulate the jet of cold water. It did a good job of condensing the
mixture of steam and mint vapor; but caused too much turbulence in
the receiver, 80 percent of the oil being lost down the receiver
drain. In order to use a jet type condenser a receiver of different
design would have to be made which would bring the distillate to a
quiescent condition. The jet type condenser has the disadvantage of
losing all the heat of condensation, because the condensate from the
receiver drain cannot be used for boiler feed water, since it is

possible some mint oil might be entrained in the condensate and the oil would corrode the steel.

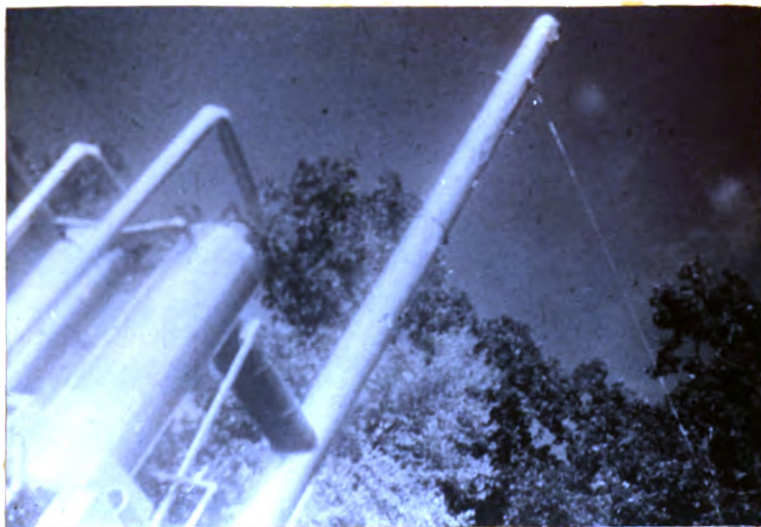


Figure 11. Aluminum condensers.



Figure 12. The aluminum condenser (A), cooling water overflow piping (B), thermometer wells (C), pressure gage connections (D) and manometer. The Manometer (E) read 0.2 inches of water at the elbow (F) connection and 0.1 inch of water less than atmospheric pressure at the condenser connection (G) at normal operating conditions.

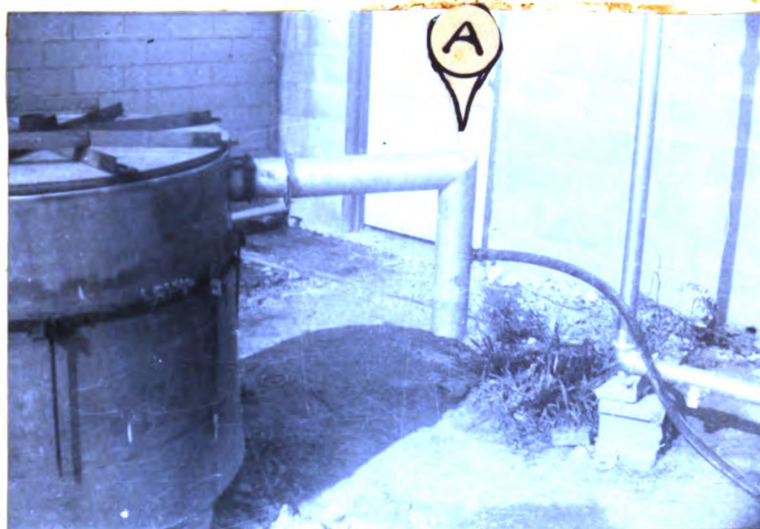


Figure 13. The jet type condenser (A). It caused too much turbulence in the receiver.



Figure 14. Nozzle (from a garden hose) of the jet condenser

The Receiver

The receiver from the former distillation apparatus was put in to use again for the first condenser. Another receiver of similar design, the design follows the principle of the Florentine flask, was fabricated for the second condenser. The one and one-half inch tail pipe from the condensers were provided with return bends to facilitate oil separation and allow the operator to easily observe when either the oil started or stopped flowing.



Figure 15. The receiver. Here it is placed in series with the first receiver to test whether any oil is passing through the drain of the first receiver. Only non-measurable traces were found under proper operating conditions. The quart bottle is used to measure the steam flow by weighing the amount of condensate caught in a period of time. Example: two pounds caught in 30 seconds would give a steam flow of 240 pounds per hour.

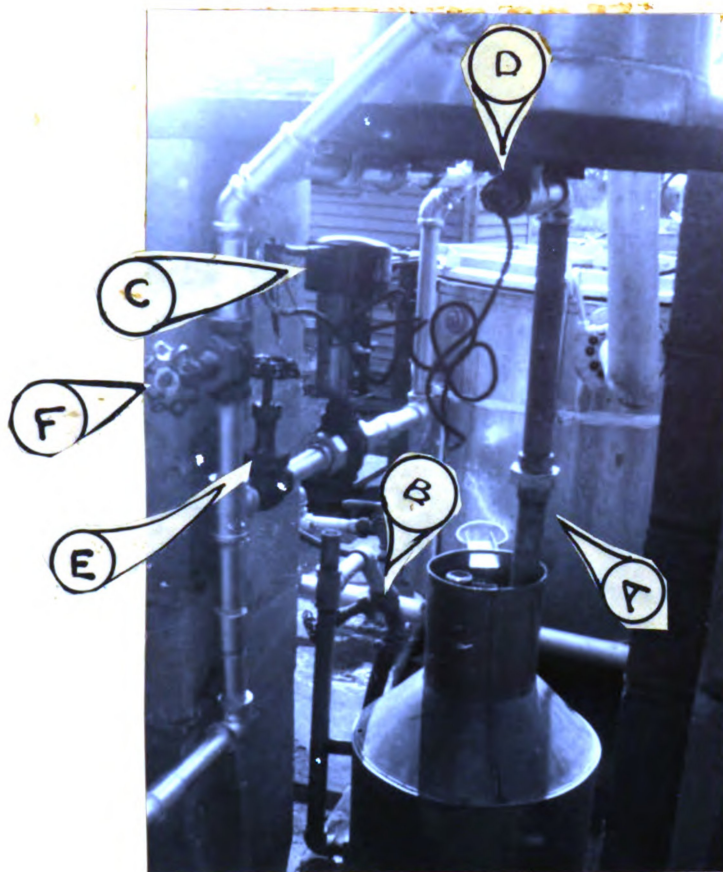


Figure 16. The condensate return bend (A) in operating position in the receiver. Oil bubbles could easily be seen by the operator (coming from the end of the pipe which rises slightly above the top of the water level in the receiver) when the oil started to flow, lack of oil bubbles indicated the oil flow had stopped. The overflow from the receiver was controlled by the globe valve (B). The condensate temperature control valve (C) and its sensing element (D) are shown in operating position. There is a globe valve (E) to shut off the water supply to the control valve, and at the extreme left a by-pass valve (F) to admit more cooling water to the condenser.

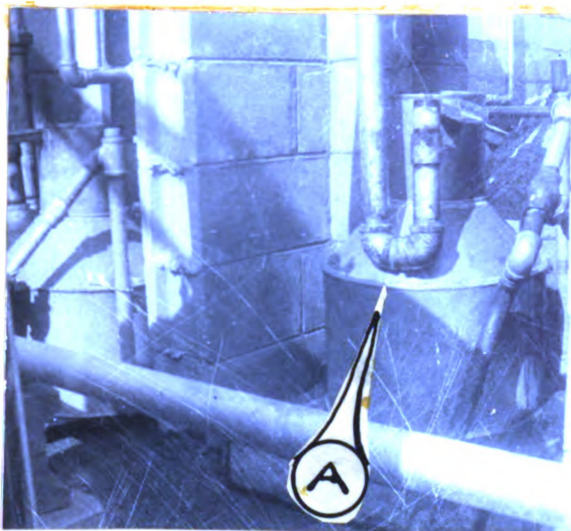


Figure 17. The condensate return bend (A) shown outside the receiver.



Figure 18. Hand operated hoist for removing the processed hay and lifting the tub covers.

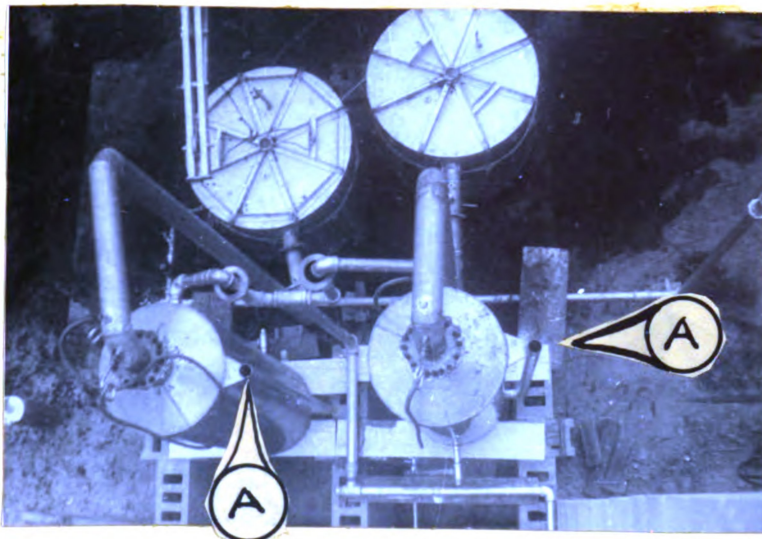


Figure 19. Top view of condensers, piping, tubs, tub covers and concrete block piers to support condensers. Note the three-inch open vents (A) rising alongside the condensers which removes air from the tub and connecting piping when first "breaking a load in".



Figure 20. The method of weighing the green peppermint hay in the field.

Equipment Changes in 1955

The above equipment installed during the summer of 1954 required some changes and additions for the purpose of improving testing procedure, efficiency of oil recovery and consistency of oil recovery. The additions and changes made were:

1. Pressure gages and thermometer wells were installed on the tub lids, condensers and the aluminum piping which conducted the steam and peppermint oil vapor from the tubs to the condensers. Figures 10, 12, 21 and 22 illustrate these fittings. These gages and thermometer wells were installed to give the operator more information concerning the behavior of the apparatus; also temperatures and pressures of these parts of the apparatus could be recorded and used as future comparison references whenever irregularities in operational performance occurred.

2. The rim of one of the tubs became defective and the tub had to be replaced.

3. Two new marine plywood covers, with different construction of metal reinforcement to test warpage, were made.

4. The vinyl plastic garden hose gasket was joined and the ends held together by lacing with a piece of plastic cord (clothesline). That is, holes drilled in each end of the hose one length of plastic cord passed through the holes the cord pulled tight and a square knot tied. There were no vapor leaks at this joint. To hold the gasket in position on the lid pieces of plastic cord were used again (Figure 10).

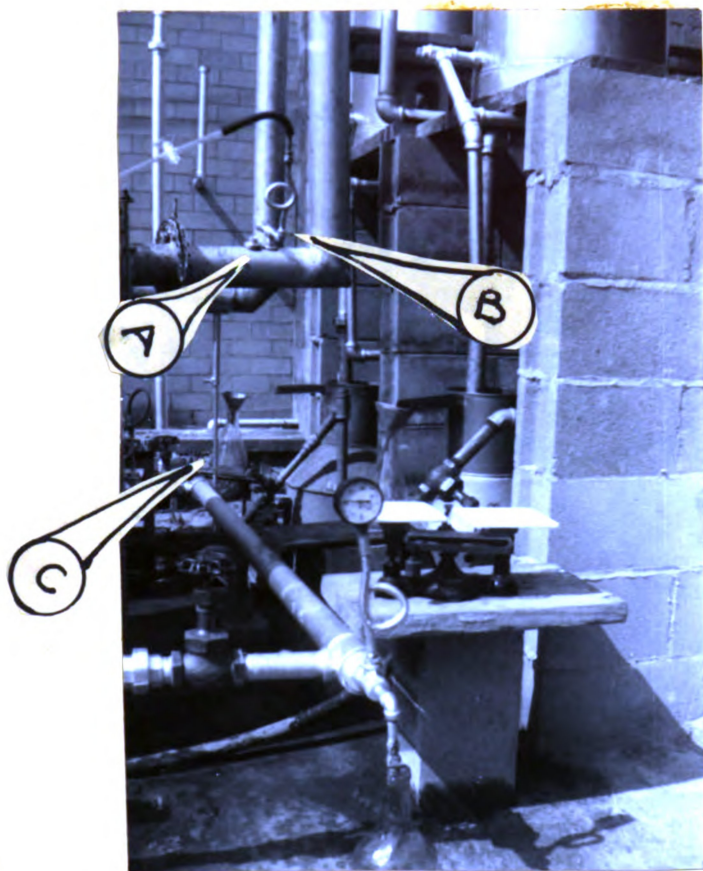


Figure 21. Thermometer well (A) and pressure gage fitting (B) on aluminum pipe on horizontal run conducting vapor from tub to condenser. The pressure at this point measured by a manometer was 0.4 inches of water. The temperature was slightly less than 112°F . The method of catching the skimmed oil (from the receiver) with a funnel and flask (C) is shown.

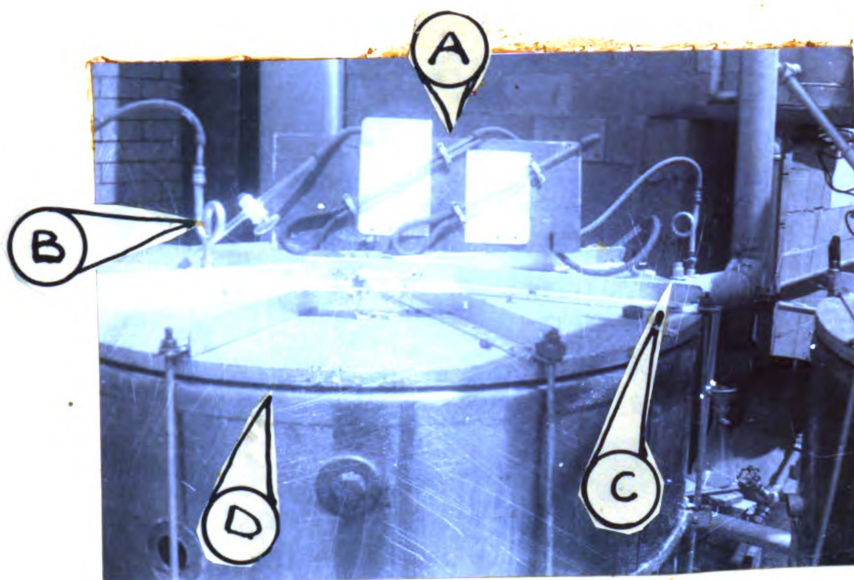


Figure 22. The manometers (A) used to determine the pressure in the tub and the pipe which conducts the vapor from the tub to the condenser. The pressure gage (B) and thermometer well (C) fittings can be seen. The marine plywood cover (D) is in operating position. The manometer reading at the tub cover was 0.5 inches of water under normal operating conditions.

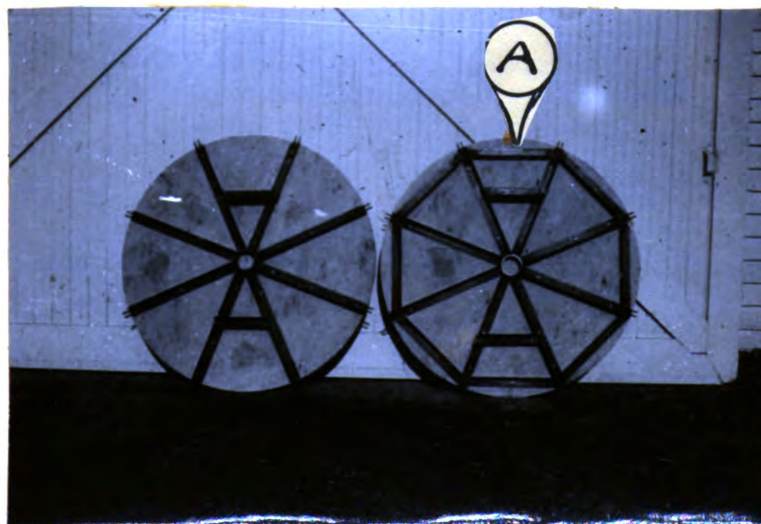


Figure 23. Top views of marine plywood covers. Note different metal reinforcement, used to compare degree of warp after use, and whether the heavier construction was necessary. Trials during the summer of 1955 showed the heavier construction was not needed. The distance between the spokes at the extremities (A) was 16 1/2 inches.

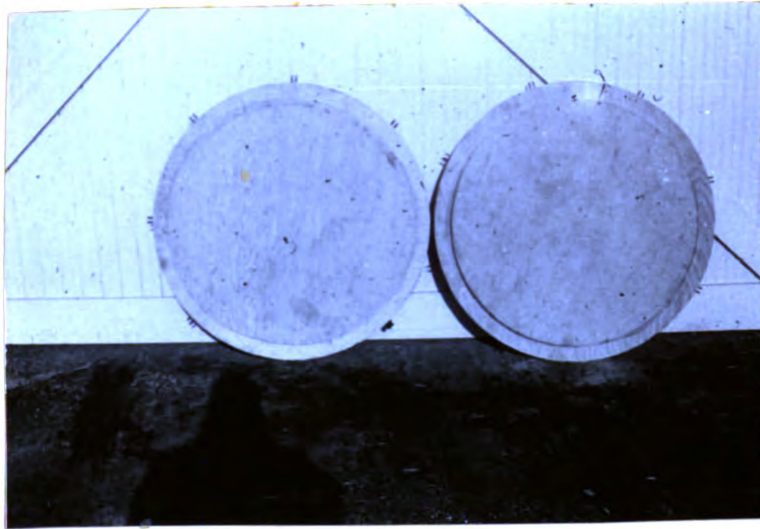


Figure 24. Bottom view of covers shown in Figure 23. The two pieces of $3/4$ inch plywood are fastened together by wood screws.

This method proved effective. In 1954 a way had not been found to fasten the gasket to the cover, this resulted in loss of time during the loading and unloading operation.

CHAPTER IV

EVALUATION OF THE DISTILLATION PLANT

Testing the Equipment After Installation, 1954

Since there is now way of knowing how much peppermint oil is in the hay and because any variance in the amount of oil recovered could be either attributed to varying amounts of oil in the hay or to lack of consistency in recovery of oil by the distillation apparatus, it was decided to place a known amount of previously processed oil into the tub, pass steam through it and see how much of the oil could be recovered in the receiver. The first problem encountered here was how to put the pure oil in the tub so that steam could be passed through it.

A porcelain funnel with filter paper was tried. This method of placing oil in the tub was discarded after five trials, because filters retained portions of the oil. Oil recovery on these five trials was unsatisfactory (Table I) since recovery was never greater than 81.6 percent by weight and losses ran as high as 43 percent. Leaks at the seal between tub and lid (aluminum lid was being used), at the tub outlet, and at the elbows of the four inch aluminum pipe carrying the vapor from the tub to the condenser were responsible for most of the losses.

Next aluminum wool saturated with peppermint oil was used to place oil in the tub. This method was also unsatisfactory because all of the oil did not evaporate. Part of the oil ran out of the aluminum wool and remained on the floor of the tub with only 20.9 percent of the oil by weight being recovered in the receiver.



Figure 25. The blackened portions on the four-inch aluminum pipe locate the vapor leaks before repairs. The wall housing the boiler is nearing completion. The pipe was dismantled welded and then replaced to stop the leaks.

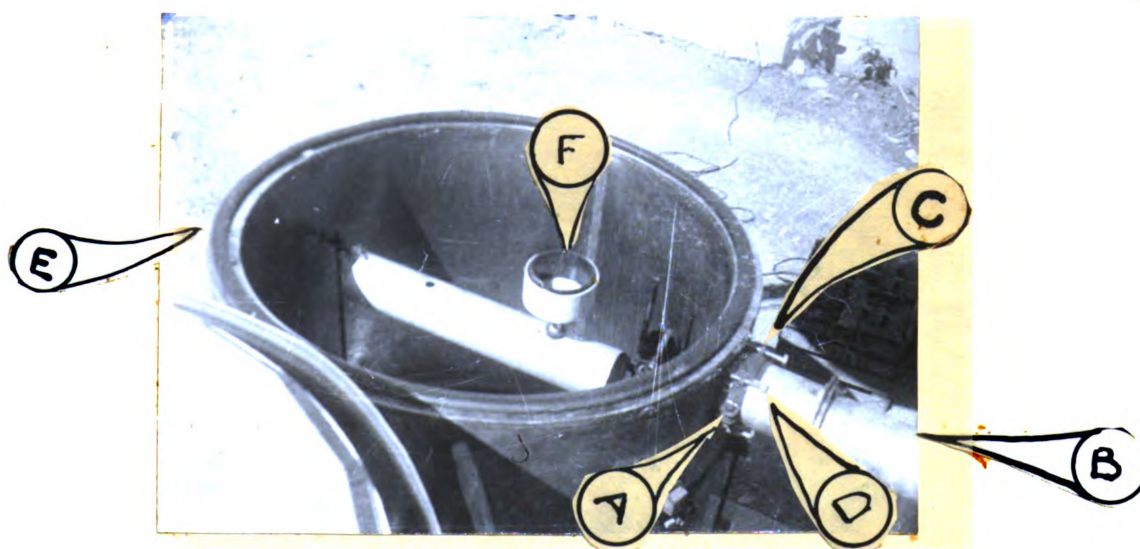


Figure 26. A ring (A) clamped tightly to the aluminum irrigation pipe (B), nuts (C) welded to the ring and cap screws (D) forced against the outer wall of the tub, is the method used to draw the flange of the pipe up tightly against the inner wall of the tub. Shown is the method of using plastic garden hose (E) as a cover seal in 1954. Also, the manner of placing peppermint oil in the tub by using a porcelain funnel (F) and filter. This method of placing oil in the tub was ineffective.

Table I. Oil recovery - vapor leaks in distillation apparatus - porcelain funnel and filter paper method.

Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent recovered	Percent lost	Distillation time, minutes	Steam flow lbs./hr.	Steam pressure (boiler) psi.
442.0	252.5	57.0	43.0	40	375	28
442.1	335.1	75.8	24.2	65	378	28
441.7	284.7	64.6	35.6	90	441	25
439.4	358.5	81.6	18.4	60	126	28
441.5	319.5	72.2	27.8	100	168	28

The next method of placing oil in the tub was to put enough hot water in the bottom of the tub to cover the steam inlet, pour a measured amount of oil on top of the water and after the lid had been fastened, pass steam through the water and oil mixture. This method ("oil on water") was satisfactory.

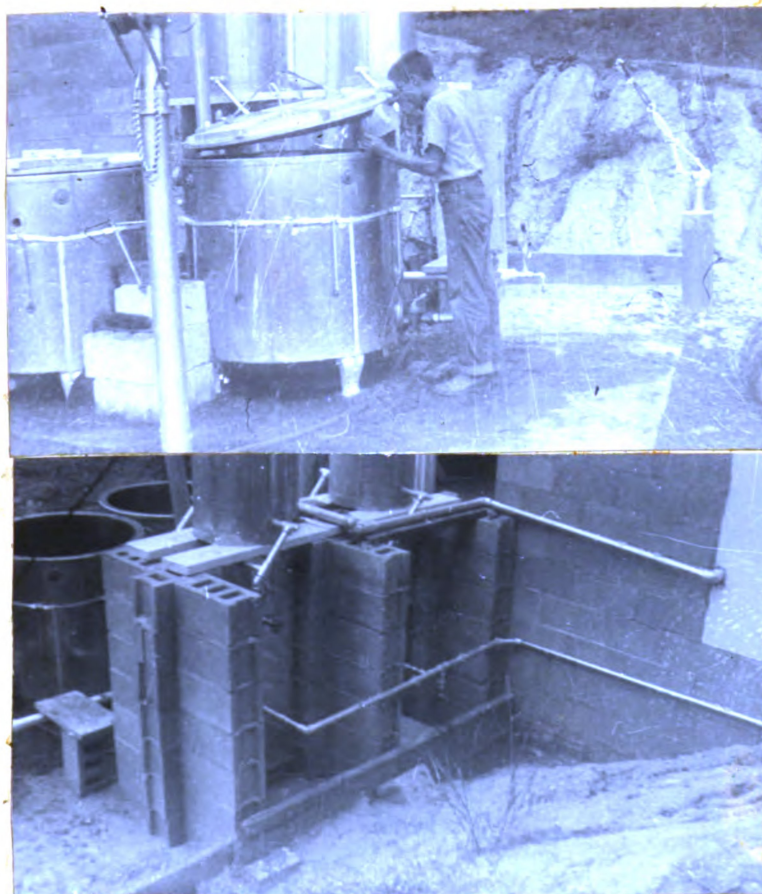


Figure 27. The three concrete block piers which support the condensers. Method of placing peppermint oil in the tub prior to distillation; there is water in the tub up to a level covering the steam inlet, the oil floats on the water and is vaporized by the steam passing through it.

Table II indicates the efficiency of oil recovery which can be expected when vapor leaks are present in the distillation equipment. While making the tests shown in Table II it was not known whether the losses were due to the vapor leaks (this proved to be true as later tests (Table III) showed) or due to oil being passed to the drain by the receiver. In order to check the efficiency of separation of the receiver another receiver was placed in series with it, Figure 15, that is the separated water which ordinarily passed directly to the drain was now passed through this second receiver. No trace of oil appeared at the number two receiver while data for Table II was being gathered.

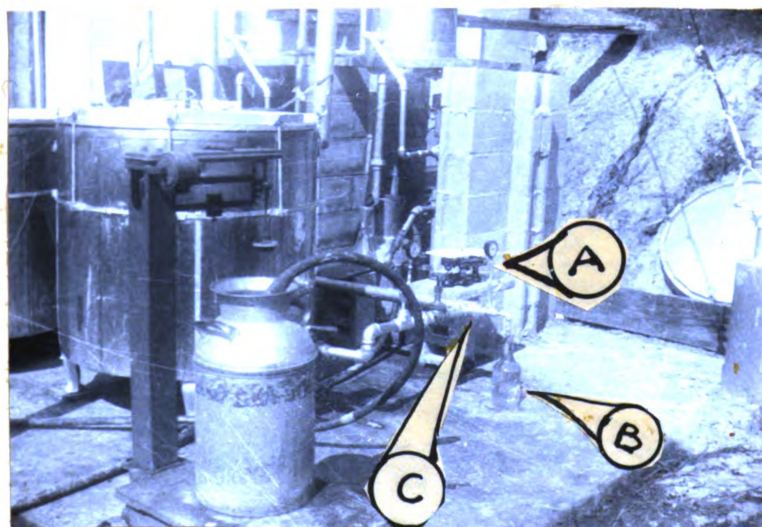


Figure 28. Another method (more accurate than that described in Figure 15) for determining the amount of steam used during a distillation trial. In background can be seen the balance (A) used to weigh the peppermint oil (accurate to 0.1 gram). Also can be seen the gallon bottle (B) used to catch the distilled water, from the steam header (C), used in pH tests.

Table II. Oil recovery - vapor leaks in distillation apparatus - oil on water method

Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent recovered	Percent lost	Distillation time, minutes	Steam flow lbs./hr.	Steam pressure (boiler) psi.
220.3	116.8	52.3	47.7	50	250	28
219.5	147.2	67.1	32.9	55	250	28
438.7	361.1	82.3	17.7	60	333	28
444.2	320.1	72.1	27.9	55	250	28
444.3	282.3	63.5	36.5	45	250	25
446.0	303.3	68.0	32.0	60	250	28



Since the, "oil-water method," assured complete evaporation of the test oil from the tub, and the receiver was separating the distillate properly, the loss of oil could only be attributed to two parts of the apparatus, either the leaking wisps of vapor were the cause of the loss or else the oil was being trapped in the condenser. Accordingly, the piping was dismantled from the condenser to look for oil pockets. There was no oil lodged in the condenser. While the piping was down all leaks were welded and stopped. A piece of plastic garden hose proved effective in stopping the vapor leak at the tub outlet joint. It should be emphasized that these leaks were barely discernable.

Table III gives the data gathered after all vapor leaks were stopped and the aluminum cover had been replaced by the marine plywood cover. With this data control charts were constructed to determine the efficiency and consistency of oil recovery by the apparatus.

Testing the Equipment After the Changes Made In 1955

The amount of oil lost by entrainment with the water discharged from the receiver has been a problem of the farmer who is the processor of peppermint hay into peppermint oil. If the amount of oil lost in this manner during field distillation could be quickly determined the farmer could adjust his apparatus and distillation procedure to obtain the most efficient oil separation at the receiver. Accordingly tests were run to see if this problem could be solved.

Separation of oil by freezing with the hope of being able to measure the oil separated in this manner was unsuccessful. The oil could not be differentiated from the water in the frozen sample.

Next, varied concentrations of peppermint oil and distilled water from the boiler were given pH measurements with a Beckman pH meter to see if a useful relationship existed which could be used to determine the amount of oil entrained in the distillate. These pH values (which referred to known concentrations of peppermint oil and water) were compared with the pH values obtained from the distillate after leaving the receiver. No useful relationship could be found.

Three samples of four measurements each were taken (Table IV) after all changes in the apparatus had been completed for the 1955 season. These samples were plotted on the 1954 control charts (Figures 29 and 30) to observe their degree of control. Then these samples were included with the samples of 1954 to give a new body of control chart data; the control charts were then revised and brought up to date (Figures 31 and 32), that is, to the beginning of the 1955 season.

Evaluation of Tests

The tests were analyzed statistically. The statistical tool used was the control chart. The control chart statistically answers the following:

1. Are the samples from a normal population or lot?
2. Is the process in control, that is, the variance is due only to chance and not to assignable variables?

3. Are the samples random?
4. What is the long range (unbiased if in control) expectation of the mean, and standard deviation?
5. Has a new variable been introduced into the test? This may be due to either unwittingly changing the test procedure, or deliberately introducing a new variable. With the former the chart will go out of control and the operator can immediately search for the cause and prevent gathering needless data; with the latter new variables may be entered one at the time and their effect on the test noted after only a few measurements.

Construction of the Control Chart

The total population or lot of a statistic is all the units of the physical objects which are being examined; the objects are homogeneous and they are exposed to the same conditions. A random sample is as true to the total population as possible. One sample consists of several measurements. For the control chart a minimum of eight samples is recommended⁽⁴⁾; each sample containing four measurements. The number of measurements in a sample will be signified by, n ; the number of samples by, k . The control chart is based upon the $\pm 3\sigma$ limits. Sigma, σ , being the symbol for a standard deviation. Three types of control charts are used, the mean, standard deviation and range charts.

The control chart for means is constructed in the following manner:

1. the average of each sample is determined and denoted by the symbol \bar{X}_1 .
2. The sum of sample averages is determined and is symbolized by $\sum_{i=1}^k \bar{X}_i$.
3. The average of the sum of the sample averages is determined and is symbolized by $\bar{\bar{X}} = \frac{\sum_{i=1}^k \bar{X}_i}{k}$. This value determines

the center line, ϕ , of the control chart.

4. The upper control line, UCL, and the lower control line, LCL, are found by using multipliers from the statistical tables for control charts. These lines may be determined by either the range, \bar{R} or standard deviation $\bar{\sigma}$. Using the range,

$$UCL = \bar{\bar{X}} + A_2 \bar{R}$$

$LCL = \bar{\bar{X}} - A_2 \bar{R}$, where A_2 is the multiplier obtained from statistical tables.

Using the standard deviation,

$$UCL = \bar{\bar{X}} + A_1 \bar{\sigma}$$

$$LCL = \bar{\bar{X}} - A_1 \bar{\sigma}$$

5. After the control lines are established each sample mean, \bar{X}_1 , is plotted on the chart.

The control chart for the standard deviations, σ , is constructed in the following manner:

1. The standard deviation, σ_1 , of each sample is obtained; which is the value determined by performing the following

calculations in sequence; (1) find the deviation of each measurement from the sample mean, $X_1 - \bar{X}$, (2) square this value, $(X_1 - \bar{X})^2$, (3) sum these squares, $\sum_{i=1}^n (X_1 - \bar{X})^2$,

(4) obtain the mean of this sum, $\sum_{i=1}^n \frac{(X_1 - \bar{X})^2}{n}$.

(5) take the square root of this mean, $\sqrt{\sum_{i=1}^n \frac{(X_1 - \bar{X})^2}{n}}$.

Symbolically the entire calculation is denoted thus:

$$\sigma_i = \sqrt{\sum_{i=1}^n \frac{(X_1 - \bar{X})^2}{n}}$$

and the value obtained is the variability from the sample mean \bar{X} known as the standard deviation.

2. Then the sum of the standard deviations of the samples is

calculated, $\sum_{i=1}^k \sigma_i$.

3. The average of the sum of the standard deviations of the

samples, $\bar{\sigma}$, is determined, that is $\bar{\sigma} = \frac{\sum_{i=1}^k \sigma_i}{k}$.

This value is the center line of the control chart.

4. The upper and lower control lines are obtained by the use

of multipliers from statistical tables thus:

$$\bar{C} = \bar{\sigma}$$

$$UCL = B_4 \bar{\sigma}$$

$LCL = B_3 \bar{\sigma}$, where B_4 and B_3 are multipliers obtained from statistical tables available in statistical quality control

texts such as Burr⁽³⁾ page 409.

5. After the control lines are established each sample standard deviation, σ_1 , is plotted on the chart.

The control chart for the ranges, R , is constructed in the following manner:

1. The range, R_1 , of each sample is determined, that is, the difference between the smallest and largest measurement of the sample.
2. The sum of the sample ranges is calculated, $\sum_{i=1}^k R_1$.
3. The average of the sum of the sample ranges, \bar{R} , is obtained,

$$\bar{R} = \frac{\sum_{i=1}^k R_1}{k} . \text{ This value determines the center line,}$$

of the control chart.

4. Using multipliers the upper and lower control lines are determined thus:

$$\bar{C} = \bar{R}$$

$$UCL = D_4 \bar{R}$$

$$LCL = D_3 \bar{R} , \text{ where } D_4 \text{ and } D_3 \text{ are obtained from statistical tables.}$$

5. After the control lines are established each sample, R_1 , is plotted on the chart.

Both the range and the mean charts demonstrate that the tests on the apparatus were in control, which indicates the samples were random and from a normal population with variations due only to chance.

Thus the apparatus can be said to be consistent in recovery of oil with variations due to chance. The efficiency of recovery can be given by the long term unbiased (unbiased because the equipment is in control) estimate of the mean and standard deviation. Burr⁽³⁾ gives the unbiased estimate of the standard deviation, $\hat{\sigma}'$, as either, $\frac{\bar{R}}{d_2} = \hat{\sigma}'$, or $\frac{\bar{\sigma}}{c_2} = \hat{\sigma}'$ where \bar{R} and $\bar{\sigma}$ are the averages of the ranges and standard deviations respectively of a run of samples, d_2 and c_2 are values found in statistical tables on quality control; also the unbiased estimate of the mean, $\hat{\bar{X}}$ is given by $\bar{\bar{X}} = \hat{\bar{X}}$. For this peppermint oil distillation apparatus the long term expected unbiased estimate of the mean efficiency of recovery of oil would be $\hat{\bar{X}} = \bar{\bar{X}} = 7.90$ percent loss or 92.10 percent recovery. The long term expected unbiased estimate of the standard deviation variability would be $\hat{\sigma}' = \frac{\bar{R}}{d_2} = \frac{6.49}{2.059} = 3.15$ percent loss and with a natural tolerance limits of $\bar{\bar{X}} \pm 3\hat{\sigma}'$ giving limits of $7.90 \pm 3(3.15) = + 17.35, - 1.55$ percent loss which should contain all of the future individual measurements when the variations are due to chance only.

It is suggested that these control charts be maintained (the mean and the range charts) by the operator of the installation. Three samples of four measurements each should be obtained at the beginning of each season to determine whether the equipment is in control. If the points fall out of control a search should be made for the cause and a correction made to the apparatus or operation procedure. If the points are in control they should be added to the existing control chart data and a new control chart calculated. If the samples are in

control the apparatus is satisfactory for gathering data on peppermint hay for replications. During the season whenever suspicious results are obtained from peppermint hay replications the apparatus should be tested by the "oil on water" method to determine whether the apparatus is still in control and the variance of the equipment is due to chance and not an assignable cause which could effect the values obtained from the peppermint hay replications. Three more points should be plotted on the control chart and the chart revised at the end of each season.

The four samples obtained at the beginning of the 1955 season were plotted on the 1954 control chart. The apparatus was in control. Including these new samples (1955) with the samples of 1954 to revise the original control charts gave a new mean, \bar{X} , of 7.84 percent oil loss, an upper controlline of 12.4 percent oil loss and a lower control line of 3.32 percent oil loss; a new range, \bar{R} , of 6.20 percent oil loss an upper control line of 14.1 percent oil loss and a lower control line of zero percent loss (Figures 31 and 32). The revised calculation for the long term expected unbiased estimate of the mean became, $\hat{\bar{X}} = \bar{X} = 7.84$ percent loss compared to the 7.90 percent loss of the original chart (Figure 29). The revised long term expected unbiased estimate of the standard deviation became $\hat{\sigma} = \frac{\bar{R}}{d_2} = \frac{6.20}{2.059} = 3.01$ percent loss compared to the 3.15 percent loss calculated from the original chart. Comparing the natural tolerance limits gave $\bar{X} \pm 3\hat{\sigma} = 16.87$ and -1.19 revised percent loss to 17.35, -1.55 percent loss calculated from the original chart.

It is suggested that the mean and range charts be used to keep the peppermint distillation plant in control. The range chart will give the same information required concerning the variability of the process, as the standard deviation, when the number of measurements in a sample is six or less⁽³⁾, and is much easier to calculate. That is, when both charts (the standard deviation and the range) are drawn of the same process the plotted points will fall on the same side of the center line and a proportionate distance; when the standard deviation shows out of control so too does the range. Taking values from the range chart and using the proper multipliers⁽¹⁾ from the statistical tables the unbiased estimate of the standard deviation⁽³⁾ may be calculated if this information is desired.

Table III. Oil recovery - no leaks in the distillation apparatus - oil on water method.

Date 1954	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent lost	Sample number	\bar{X}	R
10/30	250	40	444.2	385.9	13.1			
10/30	300	40	443.6	399.4	9.8			
10/30	350	40	442.5	367.5	17.0			
10/30	375	40	437.8	332.4	23.9			
					<u>63.8</u>	1	15.9	14.1
11/2	300	40	439.4	405.5	7.5			
11/2	300	40	885.8	785.7	11.2			
11/2	250	45	888.1	778.4	12.4			
11/2	350	45	878.8	807.1	8.2			
					<u>39.3</u>	2	9.8	4.9
11/2	300	45	885.2	821.3	7.3			
11/2	300	40	877.8	767.8	12.5			
11/2	300	40	877.4	811.6	7.5			
11/2	250	40	875.3	823.5	6.0			
					<u>33.3</u>	3	8.3	6.5
11/3	300	40	872.9	803.0	8.0			
11/3	300	40	895.3	827.3	7.6			
11/3	337	40	892.6	775.4	13.2			
11/3	300	45	880.6	811.1	7.9			
					<u>36.7</u>	4	9.2	5.6
11/3	337	40	870.6	806.1	7.4			
11/3	337	40	874.9	782.6	10.4			
11/3	337	40	875.7	803.8	8.2			
11/3	300	40	877.3	817.6	6.8			
					<u>32.8</u>	5	8.2	3.6
11/4	300	40	890.4	837.0	6.0			
11/4	300	60	893.7	841.0	5.9			
11/4	300	60	1328.5	1247.5	6.1			
11/4	250	60	1326.4	1167.2	12.0			
					<u>30.0</u>	6	7.5	6.1
11/4	250	60	1319.3	1238.8	6.1			
11/4	250	45	1319.9	1259.3	4.6			
11/4	250	50	1295.3	1237.0	4.5			
11/8	250	45	1334.3	1224.9	8.2			
					<u>23.4</u>	7	5.8	3.7

Table III. (Continued)

Date 1954	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams	Percent lost	Sample number	X	R
11/8	250	50	1334.9	1236.2	7.4			
11/8	250	50	1313.8	1227.1	6.6			
11/8	250	40	1309.8	1231.2	6.0			
11/8	250	40	1319.4	1252.1	5.1			
					<u>25.1</u>	8	6.3	2.3
11/8	250	40	1757.5	1571.2	10.6			
11/9	250	40	1794.5	1760.4	1.9			
11/9	250	40	1766.5	1685.2	4.6			
11/9	250	40	1758.0	1668.3	5.1			
					<u>22.2</u>	9	5.5	8.7
11/9	250	40	1756.8	1667.1	5.1			
11/9	250	40	1753.3	1681.2	4.1			
11/9	250	40	1761.4	1675.1	4.9			
11/9	250	40	1756.8	1663.7	5.3			
					<u>19.4</u>	10	4.8	1.2
11/9	250	40	1751.3	1691.8	3.4			
11/9	250	40	874.6	837.0	4.3			
11/10	250	40	894.5	855.8	4.7			
11/10	250	40	893.0	836.7	6.3			
					<u>18.7</u>	11	4.7	2.9
11/10	250	40	885.1	834.7	5.7			
11/10	250	40	437.1	377.7	13.6			
11/10	250	40	439.1	392.6	10.6			
11/10	250	40	438.5	396.4	9.6			
					<u>39.5</u>	12	9.9	7.9
11/11	375	40	1793.6	1594.4	11.1			
11/11	500	40	1781.2	1489.1	16.4			
11/11	500	40	1751.6	1555.3	11.2			
11/11	500	40	1759.5	1624.1	7.7			
					<u>46.4</u>	13	11.6	8.7
11/12	500	40	1746.3	1601.4	8.3			
11/12	375	40	1339.1	1280.1	4.4			
11/12	375	40	1357.1	1273.0	6.2			
11/12	375	40	1304.7	1285.2	1.5			
					<u>20.4</u>	14	5.1	6.8

Table III. (Continued)

Date 1954	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent lost	Sample number	\bar{X}	R
11/12	375	40	1353.0	1231.7	8.9			
11/12	375	40	1372.6	1239.5	9.7			
11/12	125	75	434.4	395.9	8.9			
11/12	125	75	439.2	409.8	6.7			
					<u>34.2</u>	15	8.5	3.0
11/12	125	75	439.1	400.1	8.9			
11/18	375	40	2229.5	1834.9	17.7			
11/18	375	40	2225.0	2162.7	2.8			
11/19	300	40	1780.4	1722.1	3.3			
					<u>32.7</u>	16	8.2	14.9
11/19	300	40	1780.4	1746.6	1.9			
11/19	300	40	1759.8	1698.2	3.5			
11/19	300	40	1763.5	1562.5	11.4			
11/19	300	40	1754.0	1690.9	3.6			
					<u>20.4</u>	17	5.1	9.5
							<u>134.4</u>	<u>110.4</u>

$$\bar{\bar{X}} = \frac{\sum_{i=1}^k \bar{X}_i}{k} = \frac{134.4}{17} = 7.90 \text{ percent loss}$$

$$UCL = \bar{\bar{X}} + A_2 \bar{R} = 7.90 + 0.729(6.49) = 12.6 \text{ percent loss}$$

$$LCL = \bar{\bar{X}} - A_2 \bar{R} = 7.90 - 0.729(6.49) = 3.17 \text{ percent loss}$$

$$\bar{R} = \frac{\sum_{i=1}^k R_i}{k} = \frac{110.4}{17} = 6.49 \text{ percent loss}$$

$$UCL = D_4 \bar{R} = 2.282(6.49) = 14.8 \text{ percent loss}$$

$$LCL = D_3 \bar{R} = 0(6.49) = 0 \text{ percent loss}$$

Table III. (Continued)

Date 1954	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent lost	Sample number	\bar{X}	R
11/12	375	40	1353.0	1231.7	8.9			
11/12	375	40	1372.6	1239.5	9.7			
11/12	125	75	434.4	395.9	8.9			
11/12	125	75	439.2	409.8	6.7			
					34.2	15	8.5	3.0
11/12	125	75	439.1	400.1	8.9			
11/18	375	40	2229.5	1834.9	17.7			
11/18	375	40	2225.0	2162.7	2.8			
11/19	300	40	1780.4	1722.1	3.3			
					32.7	16	8.2	14.9
11/19	300	40	1780.4	1746.6	1.9			
11/19	300	40	1759.8	1698.2	3.5			
11/19	300	40	1763.5	1562.5	11.4			
11/19	300	40	1754.0	1690.9	3.6			
					20.4	17	5.1	9.5
							134.4	110.4

$$\bar{\bar{X}} = \frac{\sum_{i=1}^k \bar{X}_i}{k} = \frac{134.4}{17} = 7.90 \text{ percent loss}$$

$$UCL = \bar{\bar{X}} + A_2 \bar{R} = 7.90 + 0.729(6.49) = 12.6 \text{ percent loss}$$

$$LCL = \bar{\bar{X}} - A_2 \bar{R} = 7.90 - 0.729(6.49) = 3.17 \text{ percent loss}$$

$$\bar{R} = \frac{\sum_{i=1}^k R_i}{k} = \frac{110.4}{17} = 6.49 \text{ percent loss}$$

$$UCL = D_4 \bar{R} = 2.282(6.49) = 14.8 \text{ percent loss}$$

$$LCL = D_3 \bar{R} = 0(6.49) = 0 \text{ percent loss}$$

Table III. (Continued)

Date 1954	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent lost	Sample number	\bar{X}	R
11/12	375	40	1353.0	1231.7	8.9			
11/12	375	40	1372.6	1239.5	9.7			
11/12	125	75	434.4	395.9	8.9			
11/12	125	75	439.2	409.8	6.7			
					34.2	15	8.5	3.0
11/12	125	75	439.1	400.1	8.9			
11/18	375	40	2229.5	1834.9	17.7			
11/18	375	40	2225.0	2162.7	2.8			
11/19	300	40	1780.4	1722.1	3.3			
					32.7	16	8.2	14.9
11/19	300	40	1780.4	1746.6	1.9			
11/19	300	40	1759.8	1698.2	3.5			
11/19	300	40	1763.5	1562.5	11.4			
11/19	300	40	1754.0	1690.9	3.6			
					20.4	17	5.1	9.5
							134.4	110.4

$$\bar{\bar{X}} = \frac{\sum_{i=1}^k \bar{X}_i}{k} = \frac{134.4}{17} = 7.90 \text{ percent loss}$$

$$UCL = \bar{\bar{X}} + A_2 \bar{R} = 7.90 + 0.729(6.49) = 12.6 \text{ percent loss}$$

$$LCL = \bar{\bar{X}} - A_2 \bar{R} = 7.90 - 0.729(6.49) = 3.17 \text{ percent loss}$$

$$\bar{R} = \frac{\sum_{i=1}^k R_i}{k} = \frac{110.4}{17} = 6.49 \text{ percent loss}$$

$$UCL = D_4 \bar{R} = 2.282(6.49) = 14.8 \text{ percent loss}$$

$$LCL = D_3 \bar{R} = 0(6.49) = 0 \text{ percent loss}$$

Table IV. Oil recovery - no leaks in the distillation apparatus - oil on water method.

Date 1955	Steam flow lb/hr.	Time min.	Amount of oil placed in tub -grams-	Oil recovered in receiver -grams-	Percent lost	Sample number all 1955 data	\bar{X}	R
7/18	250	50	995.0	877.6	11.8			
7/29	300	40	1006.0	892.3	11.3			
7/29	300	40	1000.0	938.0	6.2			
7/29	300	40	1001.2	950.1	5.1			
					<u>34.4</u>	1	18	8.6 6.7
7/29	300	40	1000.4	929.4	7.1			
7/29	300	40	1000.0	938.0	6.2			
7/29	300	40	999.8	946.8	5.3			
7/29	300	40	1000.3	932.2	6.8			
					<u>25.4</u>	2	19	6.3 1.8
7/29	300	45	1000.2	948.1	5.2			
8/1	300	40	1000.4	909.3	9.1			
8/1	300	40	1000.1	887.0	11.3			
8/1	300	45	999.8	950.8	4.9			
					<u>30.5</u>	3	20	7.6 6.4
8/1	300	45	1000.3	929.3	7.1			
8/1	300	40	999.4	887.5	11.2			
8/1	300	40	999.2	933.3	6.6			
8/1	300	40	1000.0	938.1	6.2			
					<u>31.1</u>	3	21	<u>7.8</u> <u>5.0</u> <u>30.3</u> <u>19.9</u>

Using Data of 1954 and 1955:

$$\bar{X} = \frac{\sum_{i=1}^k x_i}{k} = \frac{164.7}{21} = 7.84 \text{ percent loss}$$

$$UCL = \bar{X} + A_2 \bar{R} = 7.84 + 0.729(6.20) = 12.4 \text{ percent loss}$$

$$LCL = \bar{X} - A_2 \bar{R} = 7.84 - 0.729(6.20) = 3.32 \text{ percent loss}$$

$$\bar{R} = \frac{\sum_{i=1}^k R_i}{k} = \frac{130.3}{21} = 6.20 \text{ percent loss}$$

$$UCL = D_4 \bar{R} = 2.282(6.20) = 14.1 \text{ percent loss}$$

$$LCL = D_3 \bar{R} = 0(6.20) = 0 \text{ percent loss}$$

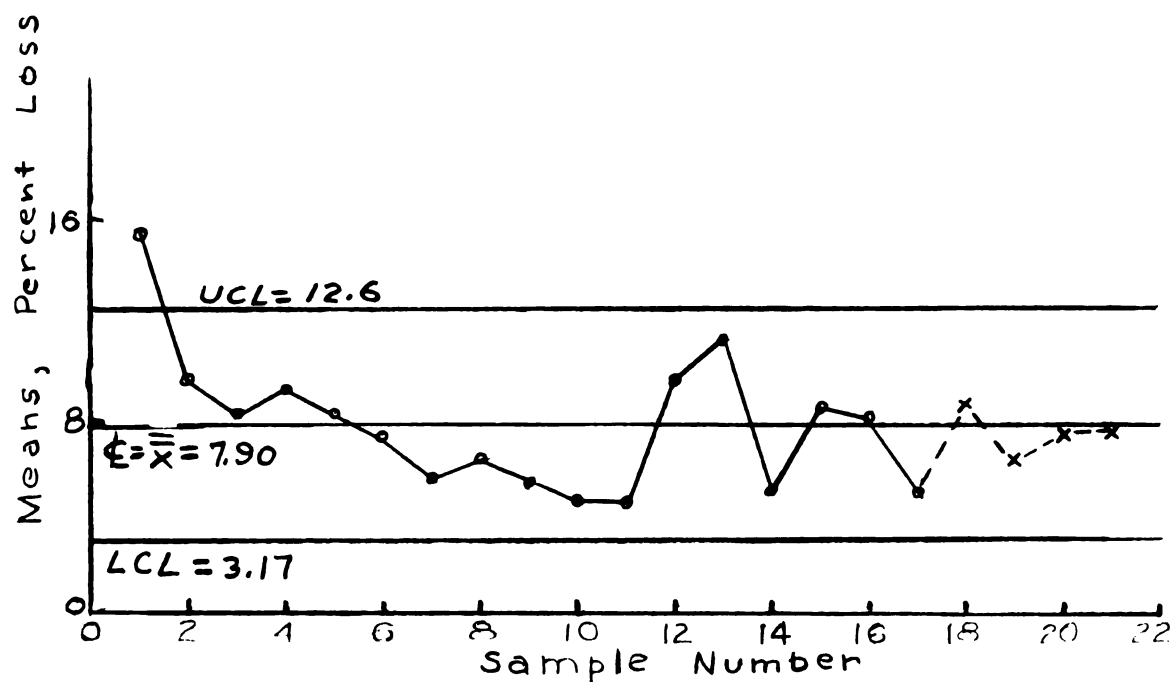


Figure 29. Mean control chart of the distillation plant for 1954. The last four samples were obtained in 1955 and plotted. They were not used to calculate the control lines.

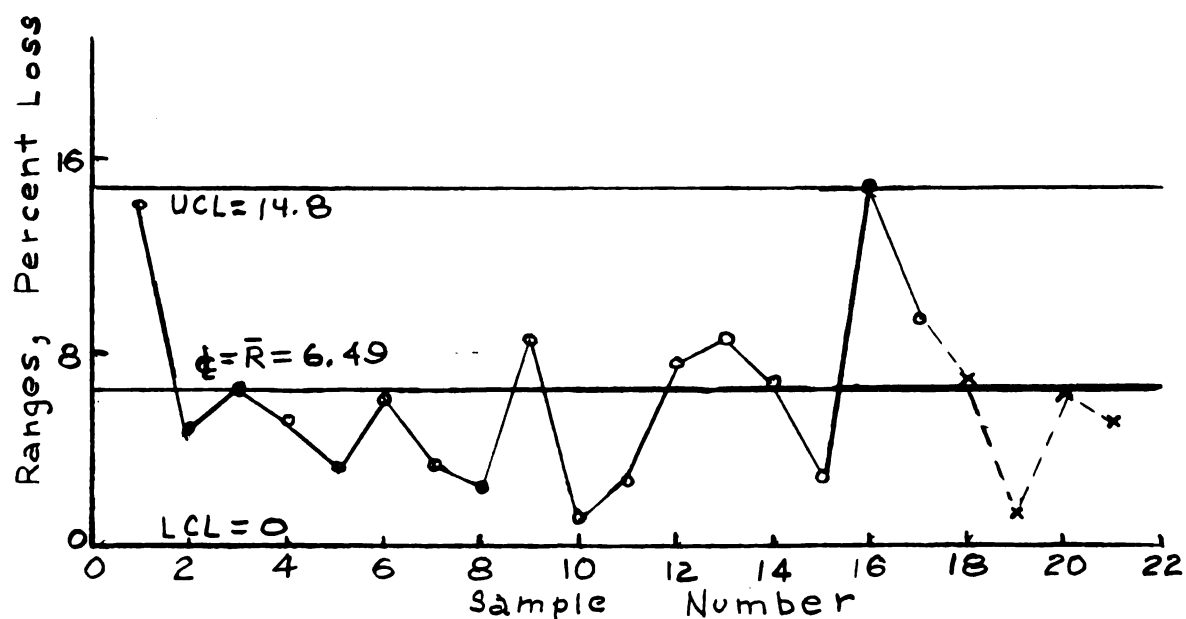


Figure 30. Range control chart of the distillation plant for 1954. The last four samples were obtained in 1955 and plotted. They were not used to calculate the control lines.

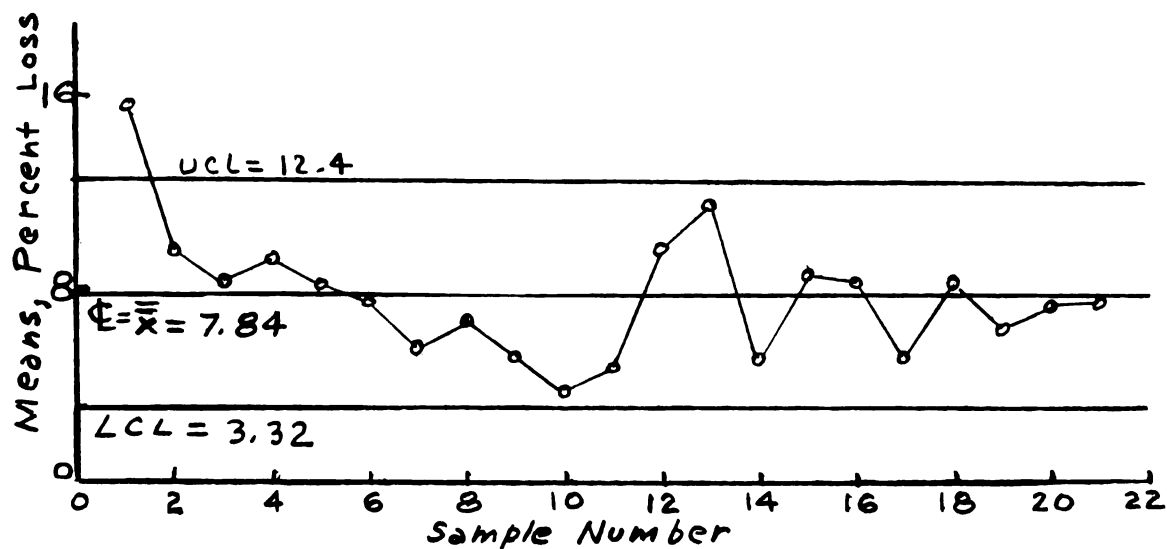


Figure 31. Revised mean control chart of the distillation plant, including tests for 1954 and 1955.

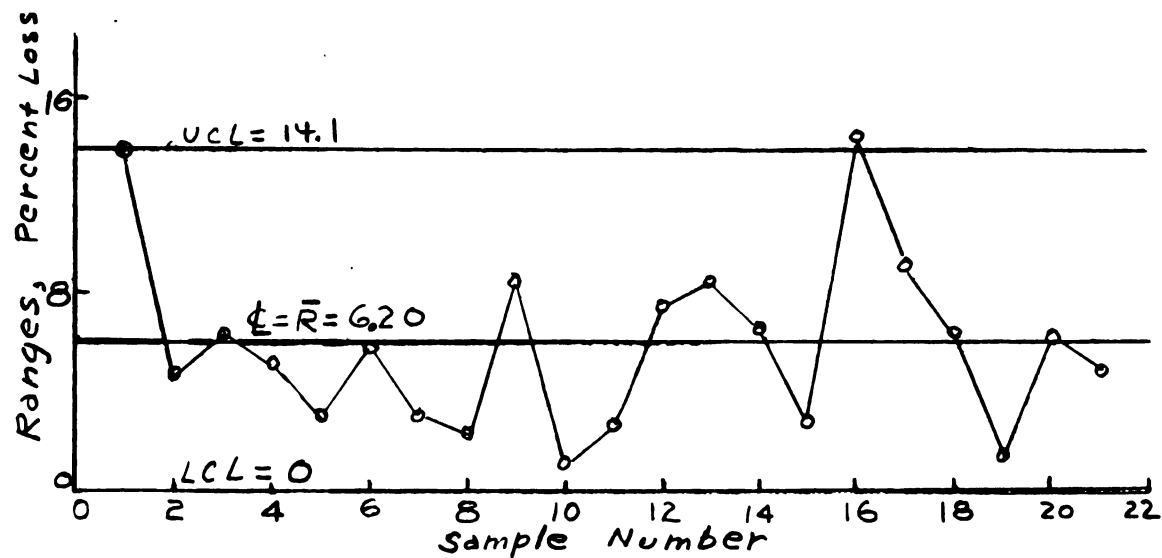


Figure 32. Revised range control chart of the distillation plant, including tests for 1954 and 1955.

Figures 33, 34, 35, and 36 were drawn to indicate the proper steam flow required to obtain the best efficiency of oil recovery for various amounts of oil processed. The points for these curves were taken from Tables III and IV.

A statistical analysis was not used on the curves because it was felt that more data at each rate of steam flow and for each amount of oil processed should be gathered. The curves do seem to give a strong indication that the amount of oil processed should influence the rate of steam flow at which the distillation plant is operated. When yields of 400 to 500 grams of peppermint oil are processed this installation would require a steam flow of between 125 and 250 pounds per hour for the least percent of oil loss, according to Figure 33. When 800 to 1000 grams of oil are processed a steam flow between 250 and 300 pounds per hour is indicated by Figure 34. For 1300 to 1400 grams of oil processed a steam rate of between 250 and 400 pounds per hour is indicated, Figure 35. When 1700 to 1800 grams of oil are processed a steam flow of 250 to 400 pounds is best, at 500 pounds of steam per hour there appears to be an increase in the amount of oil lost, Figure 36. When larger amounts of oil are processed through the still, Figures 35 and 36, the steam flow may be increased up to 400 pounds per hour without increasing the percent of oil lost. When lesser amounts of oil are processed, Figures 33 and 34, the steam flow should be decreased. The best operational steam flow according to these curves should be between 250 and 300 pounds per hour.

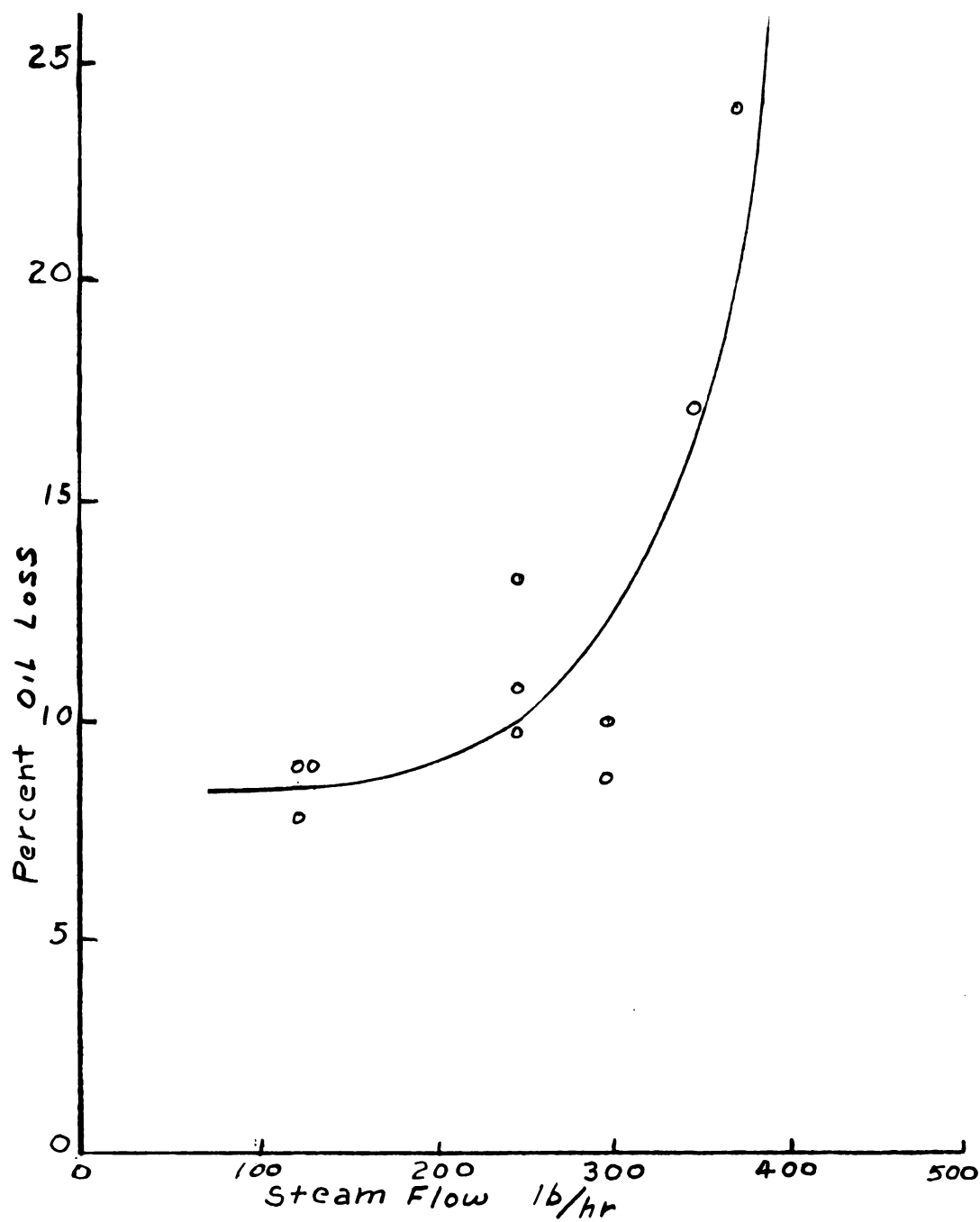


Figure 33. Percent oil loss versus steam flow, 400 to 500 grams of peppermint oil distilled.

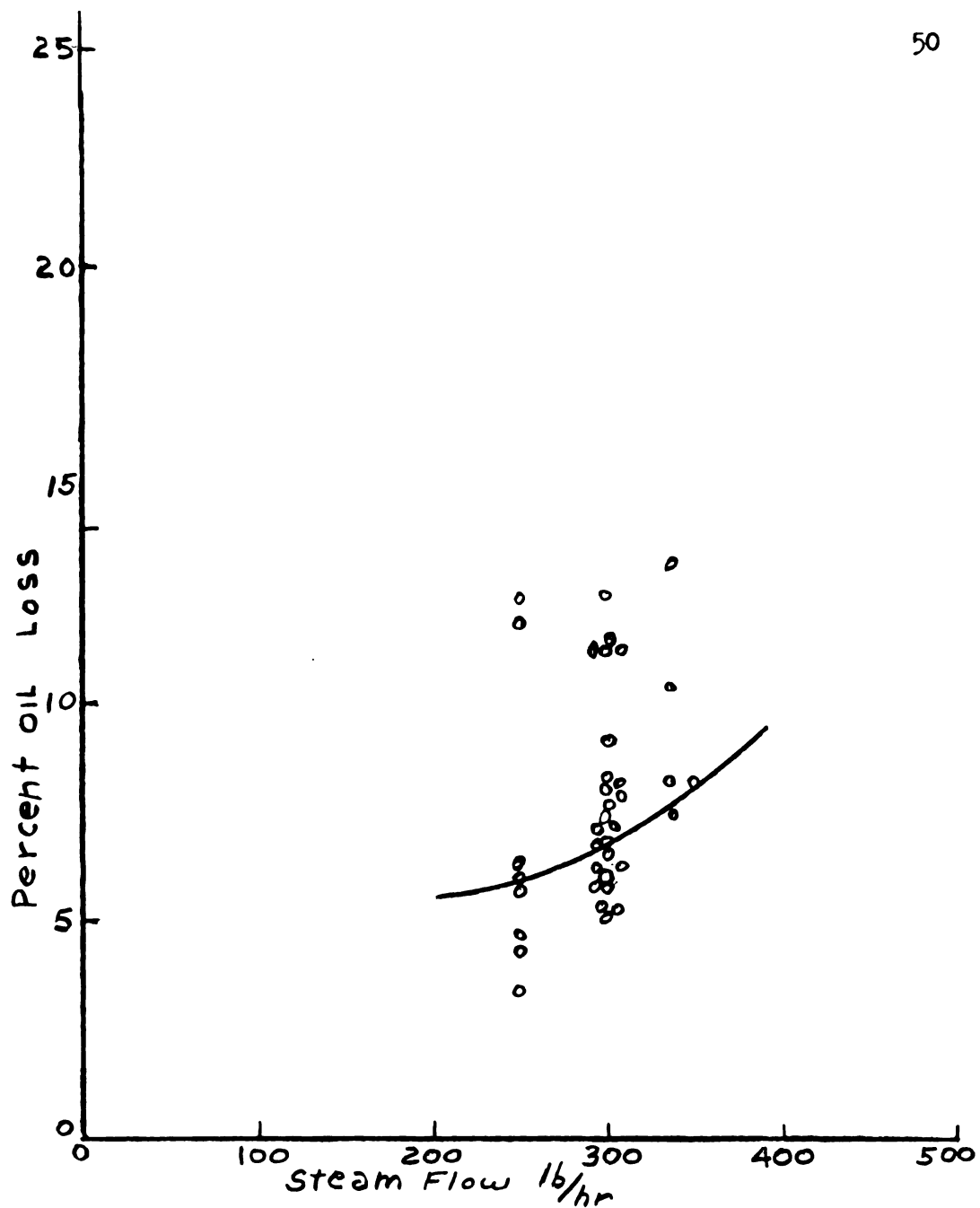


Figure 34. Percent oil loss versus steam flow 800 to 1000 grams of peppermint oil distilled.

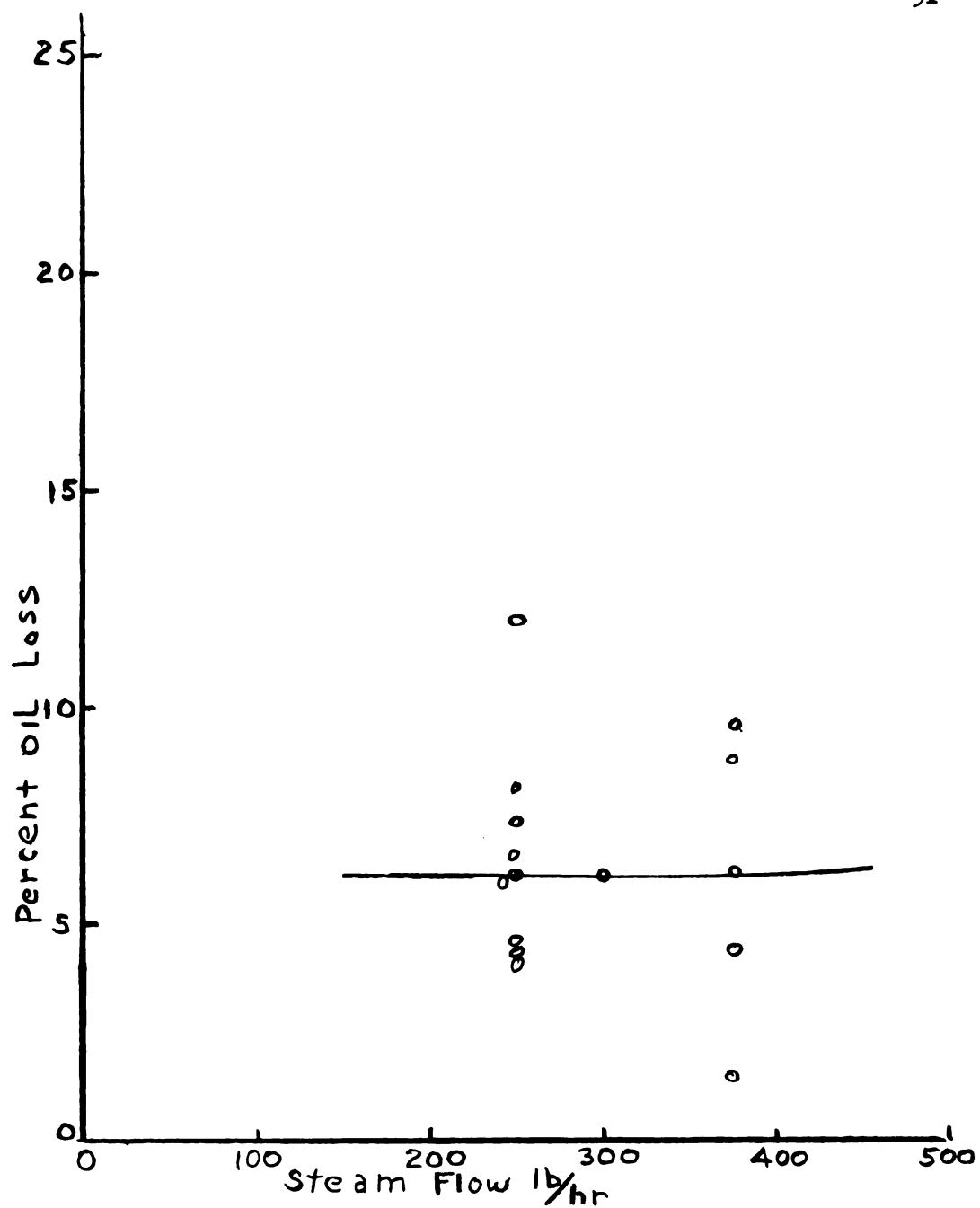


Figure 35. Percent oil loss versus steam flow 1300 to 1400 grams peppermint oil distilled.

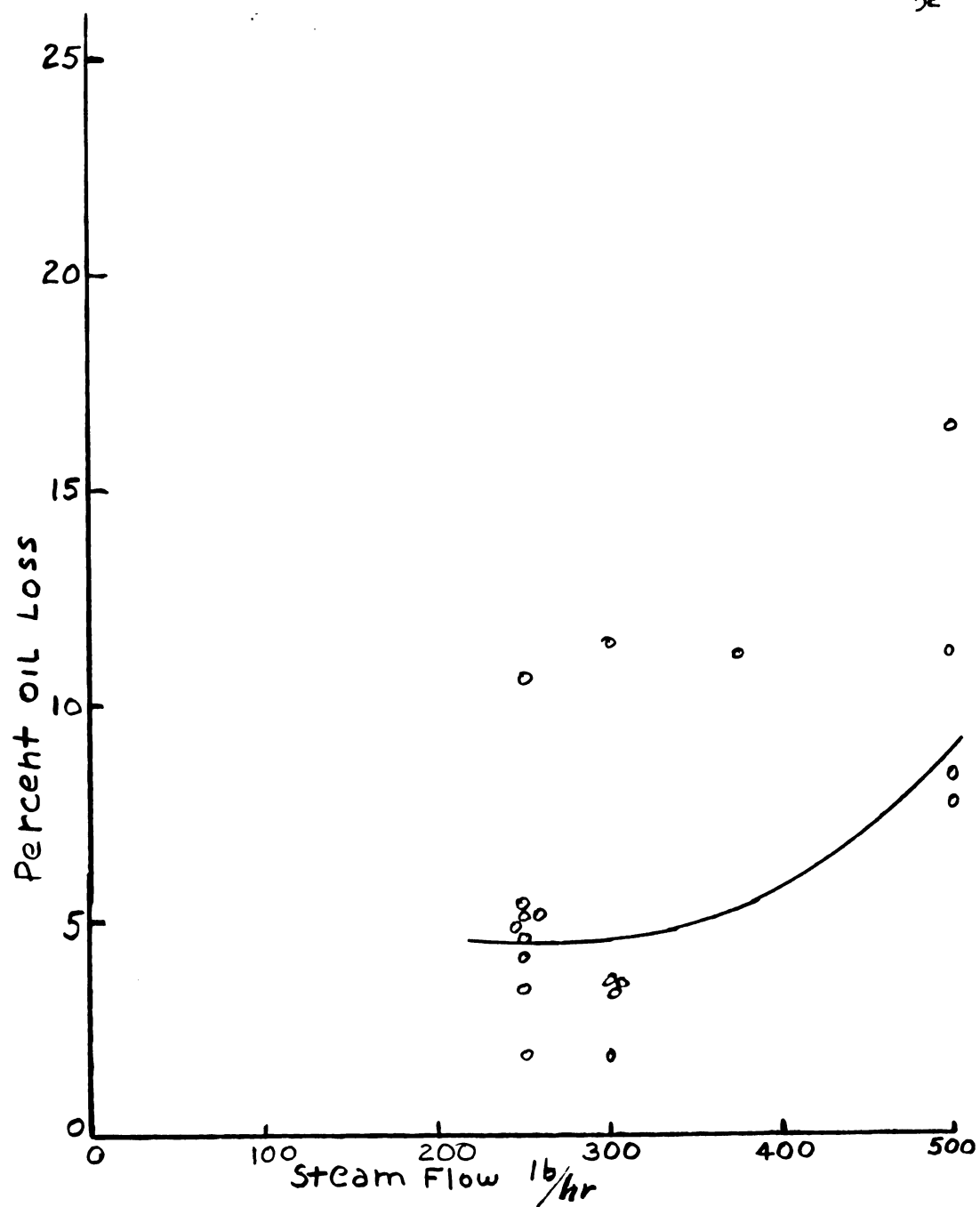


Figure 36. Percent oil loss versus steam flow 1700 to 1800 grams peppermint oil distilled.

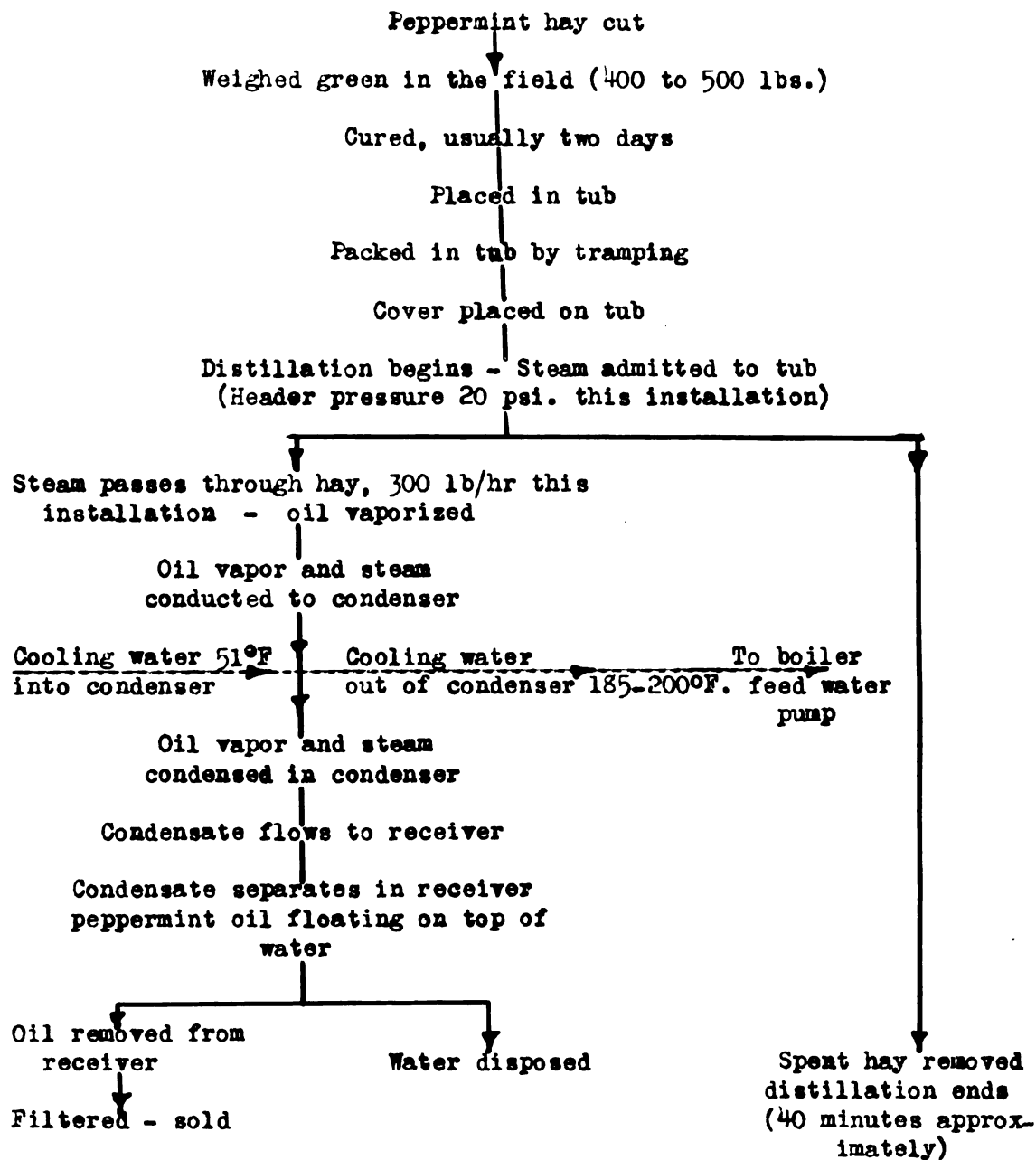


Figure 37. Product flow diagram of the experimental distillation plant.

CHAPTER V

SUMMARY AND CONCLUSIONS

Many standard pieces of equipment easily available to the farmer may be utilized in the construction and maintenance of a peppermint oil distillation plant. Those used in this investigation were:

1. Four-inch aluminum irrigation piping to conduct the vapor from the tub to the condenser.

2. A vinyl plastic garden hose used as a gasket between the cover and rim of the tub. Cut to proper sizes and shapes the plastic garden hose makes excellent gaskets for fittings placed on the apparatus. An example is the gasket placed under the pipe flange of the vapor outlet from the tub.

3. A plastic clothesline used to lace the ends of the cover gasket together and to fasten this gasket to the cover.

4. Marine plywood ($3/4$ inch) was used as a tub cover with no sign of deterioration from steam and peppermint vapors or weathering after a year of service. Tub covers made from marine plywood are satisfactory for experimental stills; whether they would be adequate for commercial stills is not known.

A return bend on the condenser tail pipe will direct the oil toward the top of the receiver; also the operator can observe the oil bubbles rising in the tail pipe showing when the oil has commenced and stopped flowing.

A jet condenser is not satisfactory; although condensing action is good, it gives too great a turbulence in the receiver. There is no way of recovering the heat of condensation.

Pressure gage fittings and thermometer wells placed in the steam header, entrance to tub, tub cover, vapor conducting pipe (tub outlet and condenser inlet) and condenser will give the operator information concerning the performance of the still. By recording the readings of these instruments periodically a permanent history of the apparatus will be available, to refer to, whenever irregularities in performance occur.

Losses as great as 47.7 percent by weight as a result of vapor leaks at the tub, condenser and piping between the tub and condenser were recorded.

When two receivers are placed in series the second receiver will indicate whether oil is being lost from the first receiver and operating methods can be altered to correct this condition. Improper receiver temperatures, failure to warm up condenser and receiver prior to operation and too great a steam flow are the more frequent causes of losses down the drain. No method was found of quickly determining the amount of oil entrained in the water passing from the receiver. Tests on both the freezing of the distillate and pH determinations gave negative results.

Distillation tests were made to determine the efficiency and consistency of oil recovery by the apparatus. The results of these tests were analyzed with the statistical control chart. The method

of processing the oil for these tests was to put enough hot water on the bottom of the tub to cover the steam inlet, pour a measured amount of oil on top of the water and after the lid had been fastened, pass steam through the water and oil. The amount of oil recovered at the receiver was weighed and compared with the amount placed in the tub. The statistical control charts indicated the apparatus was in control and consistent in recovery. Continuation of these control charts, mean and range, can be used as a method of insuring the consistency of oil recovery, by the apparatus, required in determining the amount of oil produced from experimental peppermint plots. The efficiency of the apparatus is given by a mean of 7.84 percent oil loss from the control chart with an expected long term standard deviation of 3.01 percent loss. The mean of the range was 6.20 percent oil loss.

Suggested Future Investigations

1. The actual efficiency of the receiver should be investigated. This study indicates a 7.84 average oil loss with indications that the receiver is largely responsible. It seems that a receiver (perhaps separator is a better word) of more efficient design is needed.
2. A method of quickly determining the amount of oil lost by entrainment with the water from the receiver is needed. Perhaps this problem would resolve itself when determining the efficiency of the receiver.
3. Design a continuous peppermint oil distillation process.

4. Determine the efficiency of commercial distillation plants; which would include the distillation, time and motion studies, and an analysis of the flow of the product. This study should include the synthesis of the most efficient methods.
5. The feasibility of mow drying the peppermint hay with fans or heated air and distilling from the mow. Thus the hay could be harvested in a few days at the optimum maturity period and distilled later. The Clevenger apparatus⁽⁵⁾ and the viscometric method of determining the menthol content⁽⁵⁾ could be used to determine proper maturity.
6. Investigation of the possibility of an improvement in condenser design. The processor should be able to completely dismantle the condenser, to clean inside and outside tubing. Final condenser cooling water temperatures are of the order of 200°F which will deposit many of the solids from the water. Two passes, cast aluminum heads fastened with cap screws and easily removed tubes threaded and screwed into place are suggested.
7. A study of the most efficient size and shape (depth compared with diameter) of tub for time and amount of oil recovery. Included in this determination should be the best methods of steam inlet, that is, one or several inlets, inlet from bottom sides or top, one, two, or three passes of steam through the material. Also, a mechanical means of packing the hay should be devised. A heavy wide (12 inches) flat-rimmed wheel that revolves riding on the hay at the inner wall of the tub is suggested.

8. Is there an engineering method of controlling Verticillium wilt⁽¹¹⁾,
Such as treating transplants with electron beams, high frequency
sound waves, or a method of sterilizing the soil, without harming
beneficial organisms.

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