

Relationship of
Maturing and Weathering
to Yield and Quality of
Peppermint Oil

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Foreword

The production of mint for peppermint oil has developed into an important industry in the state of Oregon in recent years.

From 11,000 acres a yield of 550,000 pounds of peppermint oil was obtained in 1947. It sold for an average price of \$7 per pound.

As has been the case with many specialty crops, the mint producers have been confronted with serious problems. In order to help solve the most vital of these problems, the state legislature appropriated funds for the Oregon Agricultural Experiment Station to conduct research in this field. This bulletin covers that portion of the investigations assigned cooperatively to the Agricultural Chemistry and Agricultural Engineering Departments of the State College.

Dean and Director

Wm. A. Schoenfeld

Acknowledgment

The authors wish to express their appreciation to Virgil Freed for his work in making microscopic examinations and the determination of size and number of oil glands.

Summary and Conclusions

▶ The amount and quality of oil from weathered mint was not materially different from that of freshly cut mint when all shattered leaves from the weathered mint were distilled with the sample. Weathering losses appeared to be the result of a physical loss of leaves rather than a decrease in oil content.

▶ Oil cells of mint increased rapidly in size and number until full-bloom stage. After full bloom, size increased slowly and the number of cells remained about constant. The color of cell contents changed from green to yellow, orange-yellow, and finally to brown late in the season.

▶ The time at which the oil cell size and number approached a maximum coincided closely with the point of maximum per acre yields of dry hay and oil and occurred at about full-bloom stage.

▶ Distribution of oil glands was about 80 per cent on the lower leaf surface, 19 per cent on the upper leaf surface, and 1 per cent on the stems.

▶ Per acre yields of dry hay and oil increased until about full-bloom stage, after which dry hay yield was about constant and oil yield decreased slowly. The oil content of the dry hay decreased slowly and regularly from the first to the last sampling.

▶ The menthol content and ester content of the oils progressively increased from early to late season but the amounts for any given date may vary from year to year.

▶ Specific optical rotation of oils was higher in early and late season samples than in the midseason lots. No orderly variation with maturity was noted in either specific gravity or refractive index of the oils.

Relationship of
**Maturity and Weathering
to Yield and Quality of
Peppermint Oil**

By

D. E. BULLIS, F. E. PRICE, and DALE E. KIRK

WHAT EFFECT do the time of cutting and weathering have on the yield of high quality peppermint oil? To Oregon peppermint growers the answer to this question would eliminate one of their most serious difficulties.

This bulletin presents the results obtained from research conducted to find a satisfactory answer. It deals with (1) the relationship of maturity of peppermint to the yield and quality of oil produced, and with (2) the effects that weathering, particularly heavy rains, may have on yield and quality of oil produced from mint hay. These studies were confined principally to the Lower Columbia mint-growing area where the weathering problem is of vital concern.

The first section of this bulletin is devoted to the experimental procedure of the three seasons of work. The results of these experiments are discussed in the last section.

Procedure of the Experiment

The 1943 season

The project was not set up in time to conduct more than a few tests of a preliminary or exploratory nature on the 1943 crop. About 1,200 pounds of green mint were harvested from the Nelson Gilmore farm, located near the Buena Vista ferry in Marion County, on September 13. The mint was hauled to the College and divided into 50-pound lots. Two of these lots were distilled green the day after arrival. The remaining lots were divided into three series of six samples each. Each 50-pound sample was spread out on a 4-foot by 8-foot sheet of plywood to cure.

Series A was cured inside a well-ventilated building. The other two series (B and C) were cured outside the building in the prevailing weather. At that time the weather was clear and fairly warm.

Series C was thoroughly sprinkled with water after 2, 4, and 7 days to simulate the effects of rain on cut mint in the field.

At intervals of 2, 4, 7, and 11 days, duplicate samples from each series were steam distilled. The yield of oil was determined, and the oil analyzed chemically to determine what significant differences, if any, existed in the oils that came from the variously cured mint lots.

Outline of Objectives for 1944 and 1945 Seasons

The 1944 season

1. Determine the effect of weathering during harvest on loss and on chemical composition of mint oil.
2. Determine the relationship of maturity to the percentage of oil in the dry mint and to the chemical composition of the mint oil.

The 1945 season

1. Determine the effect of weathering during harvest on loss and on chemical composition of mint oil.
2. Determine the relationship of maturity to the percentage of oil in the dry mint and to the chemical composition of the mint oil.
3. Determine the relationship of maturity to the acre yield of mint oil.

Results obtained from the 1944 crop tests were closely confirmed by the more complete 1945 work. This report combines the findings of both years' investigations to avoid needless repetition in the description of procedure and in the discussion of results.

Through the courtesy of William H. Hauptert and C. E. Hermo, a small tract of mint on the Collins estate holdings near Quincy, Oregon, was set aside for experimental use both years.

Samples were harvested each week beginning August 19 in 1944 and August 1 in 1945. Harvesting ended October 5 in 1944 and September 25 in 1945. A 10-pound sample for moisture determination and four 50-pound samples for distillation were weighed out when the mint was cut.

Two of the 50-pound samples were placed in plywood trays 4 by 8 feet by 8 inches deep. These trays were taken to the College where they were kept indoors. The mint was distilled 40 to 42 hours after cutting. Very little curing took place in the trays.

The other two 50-pound samples were placed in trays of the same size but with $\frac{1}{4}$ -inch mesh hardware screen bottoms. Similar screen was used to cover the trays. Blocked up a few inches to

permit air circulation under these samples, the trays were left in the field where they were exposed to the prevailing weather until the following week. The samples were then transferred to plywood-bottom trays and taken to the College for distillation the next day. Approximately 8 days elapsed between cutting and distillation. No leaves were lost in weathering because all the shattered leaves were retained on the screen.

In 1944, only enough mint was cut each week to supply the required samples. In 1945, measured areas were harvested and weighed each week. From the measured area and the total weight, it was possible to calculate the yield of mint hay and the yield of oil per acre at each date of sampling.

In order to obtain samples as nearly representative of the field average as practicable, a strip 10 feet wide by 720 feet long was laid out across the field. This strip was divided into four equal parts. Each of these parts was further divided into nine 20-foot plots. On each harvest date, one plot from each quarter was cut and weighed. The four lots were then combined and four 50-pound samples were then weighed out for distillation. Each of these samples was composed of equal portions from the plots harvested on that date. A smaller amount of hay from each plot was taken to comprise the 10-pound sample for moisture determination.

The changes in size, number, and color of the oil glands on the mint leaves were studied by making counts and measurements from each week's sampling. The average number of cells per unit area was determined by counting three microscopic fields per leaf on 20 leaves. The average size was found by measuring five fields on each of the above counts or totalling 15 microscopic fields per leaf on 20 leaves.

Distillation was carried out in a small steam still with a capacity of 50 to 70 pounds of green mint. It had a conventional water seal lid and was supplied with steam from an automatic oil-fired boiler set at about 25 pounds steam pressure. The vapors were condensed in a multiple tube conductor. In 1943, the distilling time was 40 to 45 minutes and 6 gallons of distillate collected. In 1944, the distilling time varied from 40 to 60 minutes with the steam valve adjusted to yield approximately 7 gallons of distillate. In 1945, the distilling time was 55 to 75 minutes for about 10 gallons of distillate.

Distillate was collected in a glass container of about 1½ gallons capacity provided with a bottom outlet connected to a gooseneck tube so that the water drained out continuously into a 10-gallon milk can.

Distillation was stopped when 8 or 9 gallons had been collected in the can. The oil layer and the water in the glass receiver were

transferred to a 2-gallon milk can. Thus the distillate from each run was divided into two portions: the large one containing mostly water with traces of peppermint oil, and the small one containing most of the oil together with about a gallon of water.

Extraction of oil traces in the larger container of distillate was made by shaking out once with petroleum ether, removing the petroleum ether layer and discarding the water. The smaller vessel containing most of the oil was extracted several times with petroleum ether and the extracts combined. The petroleum ether was removed by distillation on a steam bath using a short refluxing column and a vacuum of about 2 pounds. The petroleum ether used for extraction boiled below 45° C.

The mint oil was weighed to obtain yields and then placed in 35° F. storage until it was analyzed. Analyses were made for: free menthol, total menthol, esters, acid value, optical rotation, refractive index, and specific gravity.

The Data and a Discussion of Results

Graphs 1, 2, and 3 show the oil yields and moisture losses which occurred in the 1943 weathering trials. Only relatively minor differences in yield or quality of the oils from the several lots were observed. These were of no greater magnitude than might be attributed to sample variation under the conditions of the experiment. Weather conditions during the period of the tests were ideal for field curing and were not conducive to losses which occur during rainy weather. Temperatures went as high as 95° F. and relative humidity as low as 22 per cent, which caused the mint to dry out rapidly after it was soaked.

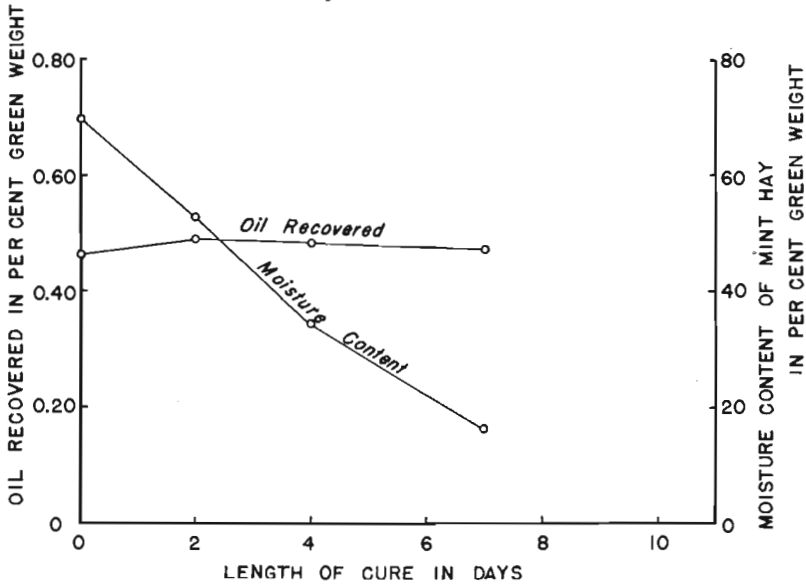
Graphs 6, 7, and 8 do not indicate that weathering of the mint hay in the field for one week after cutting materially affected either the composition of the mint oil or the percentage of oil in the dry hay. These crop graphs show particularly well how closely the total menthol, esters and oil values for green and for weathered mint agree on each harvest date throughout the season. It is believed that the 1945 charts are the more accurate of the two because the 1945 experimental work was more carefully controlled.

Rainy weather caused considerable leaf shattering in cut samples. In these experiments the shattered leaves were retained on the wire screens on which the cut mint was weathered so that no physical loss of plant parts occurred prior to distilling.

Graph 4 depicts the changes in size and number of the oil glands observed through the 1945 season. Cell size increased rapidly until about full-bloom stage after which the size increased very slowly

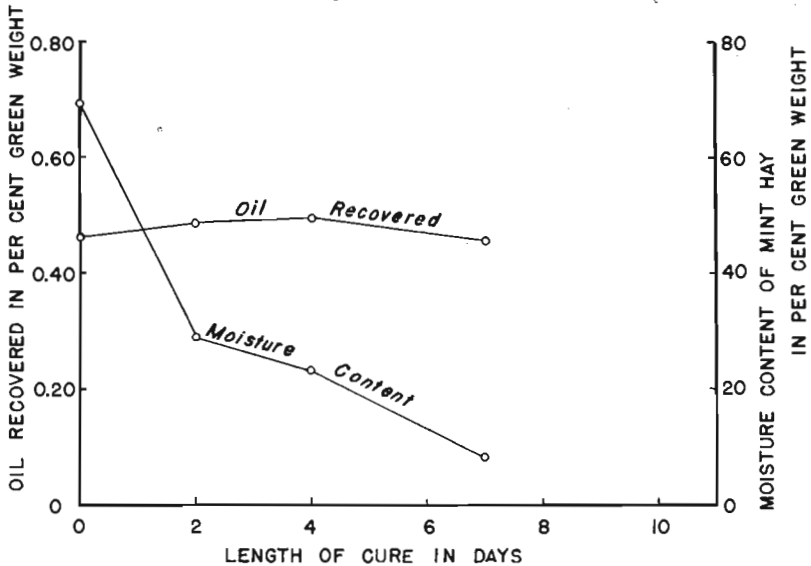
Graph 1

Recovery of Peppermint Oil from Series "A" 1943 Season
Mint Hay Cured Under Cover



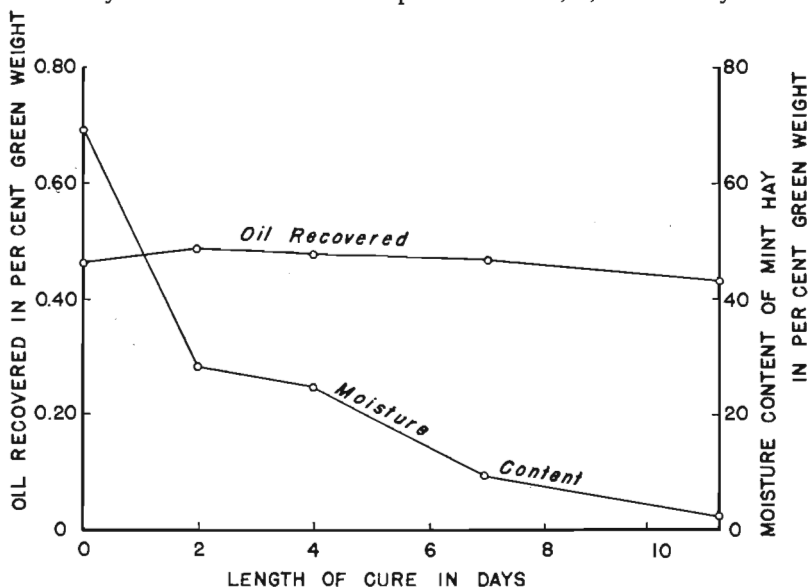
Graph 2

Recovery of Peppermint Oil from Series "B" 1943 Season
Mint Hay Cured in Sunshine



Graph 3

Recovery of Peppermint Oil from Series "C" 1943 Season Mint Hay Cured Outside and Sprinkled at 2, 4, and 7 Days



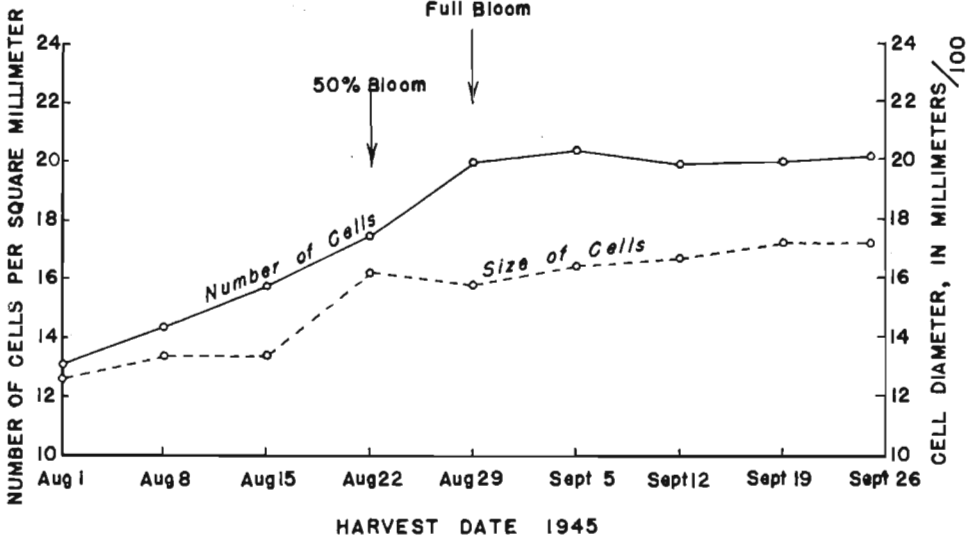
through the remainder of the period of observation (to September 25). The number of cells per unit area also increased until about full-bloom stage (August 29) after which it remained fairly constant for the remainder of the season. The rate of increase in cell numbers was much slower than the rate of size increase during the early part of the season when the principal changes were taking place. The point at which the increase in size and number of oil glands level off also corresponds very closely to the time at which the per acre yield of moisture-free mint hay and of oil are a maximum for the season (see Graph 5). It would thus appear that the factors of oil yield, dry forage yield, oil gland size and number are closely related.

Color of the oil glands progressively changed as the mint attained and passed the optimum harvesting stage. In the immature mint they were green. As the plants matured, the color of the oil gland contents changed from green to light yellow, orange yellow, and finally many of the glands changed to brown.

Distribution of the oil glands may also be of interest. Approximately 99 per cent of them are on the leaves and only 1 per cent on

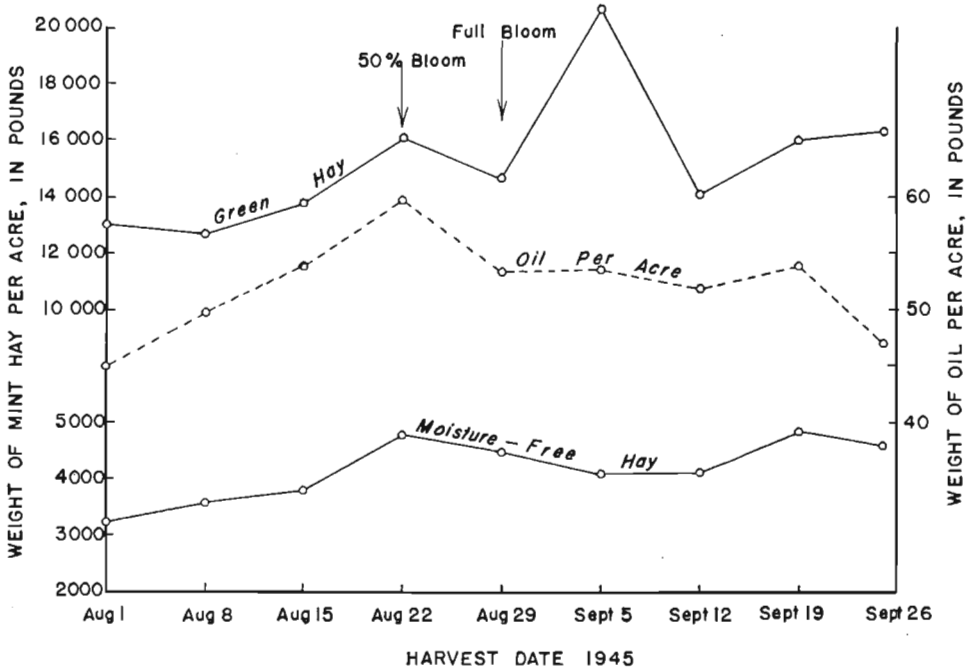
Graph 4

Relationship of Maturity of Mint to Size and Number of Oil Glands on Leaves, 1945 Season



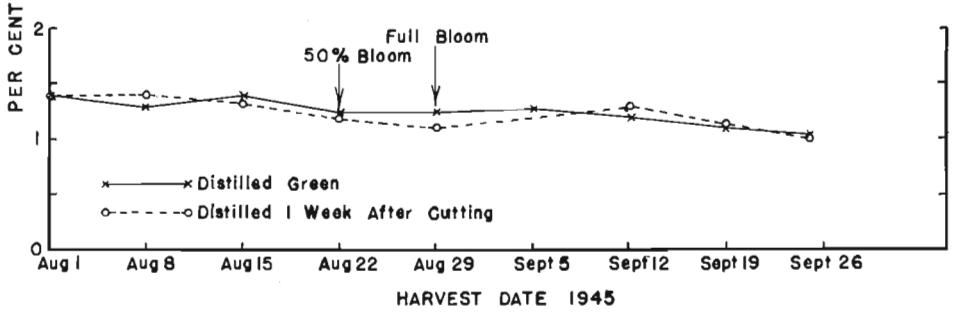
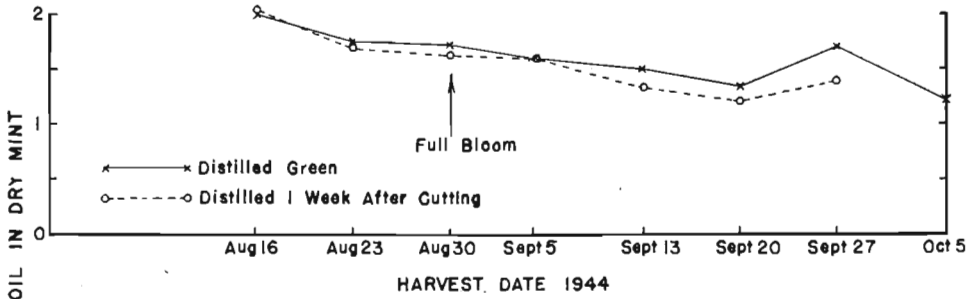
Graph 5

Relationship of Maturity of Mint to the Acre Yield of Oil and Hay, 1945 Season



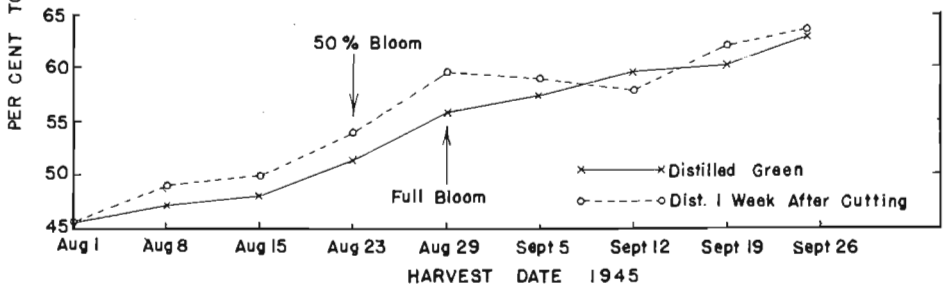
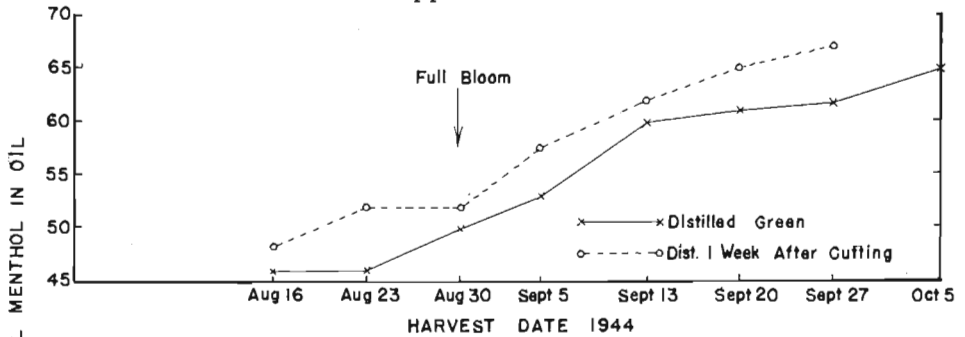
Graph 6

Relationship of Maturity of Mint to Percentage of Oil in Mint Hay



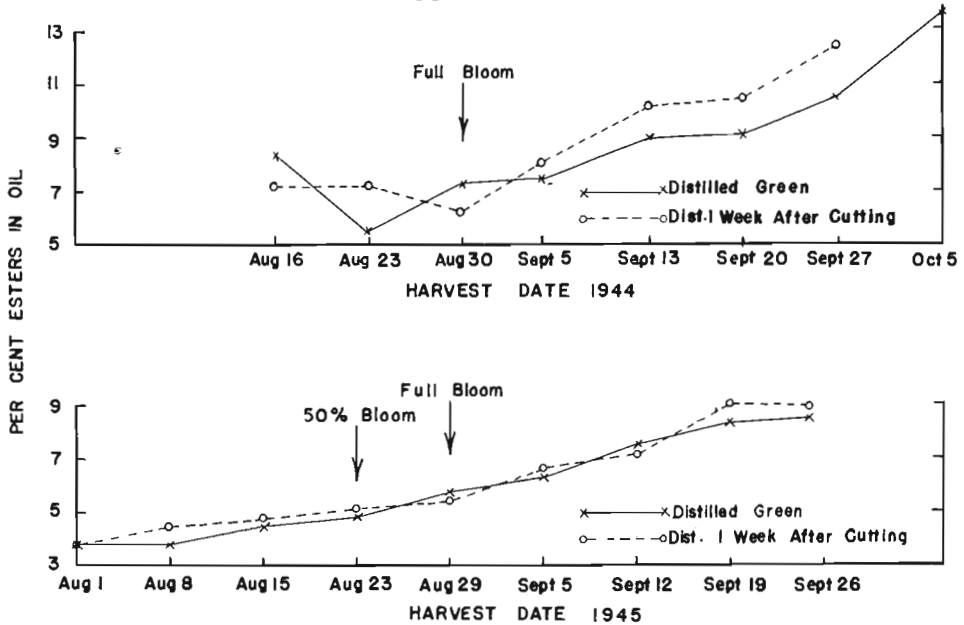
Graph 7

Relationship of Maturity of Mint to Percentage of Total Menthol in the Peppermint Oil



Graph 8

Relationship of Maturity of Mint to Percentage of Esters in the Peppermint Oil



the stems. There are about four times as many on the under-leaf surface as there are on top. This distribution of oil cells explains why serious losses in oil yields result when severe shattering of leaves occurs during periods of wet weather in the harvest season.

Graph 5 shows the per acre yield of green hay, dry hay, and oil. Green hay weight increases with maturity. It also exhibits quite erratic variations caused by the adhering water weighed with the samples harvested during wet weather. The September 5 sample typifies this condition. This sample was cut and weighed during a driving rain and the per acre weight was nearly five tons higher than at any other sampling. When the green weights are calculated to a moisture-free basis, however, it is evident (Graph 5, lower) that the dry weight yield increases up to about the full-bloom stage after which it remains more or less constant for the remainder of the season.

The per acre yield of oil increases when the per acre yield of hay is increasing. The increase in dry tonnage per acre is more than enough to offset the decrease in oil content of the dry hay (see

Graphs 5 and 6). After the dry hay yields become stationary, the per acre oil yield drops off slowly because the oil content of the hay becomes less as the hay becomes more mature.

Graphs 6, 7, and 8 show the relationship of maturity to the oil content of the dry forage and also to the ester and menthol content of the oil. The graphs do not indicate any conclusive differences between the oils from green and cured hay samples. In fact, they agree very closely on the 1945 work in which the experimental errors were probably somewhat lower than was the case in the 1944 tests.

The percentage of oil in the dry hay definitely decreased with maturity in both the 1944 and the 1945 tests, but in 1945 there was less oil in the hay than for corresponding dates for 1944. At first thought, such a decrease may appear inconsistent with the fact that the oil yield per acre is always higher later in the season than at the start of harvest. This is explained by reference again to Graph 5 where it may be noted that the per acre yield of dry hay increases with maturity much faster than the oil content decreases. With advancing maturity the over-all effect is an increase of the per acre oil yields up to about full-bloom stage.

For corresponding dates the ester content of the 1944 crop oils was somewhat higher than that of the 1945 samples. The average total menthol content of the 1944 oils is, however, quite similar to the 1945 oils from corresponding harvest dates.

In both seasons oil samples from mint harvested in midseason had lower specific optical rotation than that from early or late season lots. No explanation in the midseason drop in optical rotation is now known.

In neither of the two seasons was there any orderly variation in the refractive index or specific gravity of the oils with maturity. There was some indication, however, that both refractive index and specific gravity were slightly lower on the first two or three samples than for the remainder of the harvest season.

Table 1. PEPPERMINT OIL ANALYSIS—1944 CROP*

Sample	Free menthol	Combined menthol	Total menthol	Esters	Acid value: KOH per gram	Optical rotation ¹	Refractive index ²	Specific gravity ³
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Mg.</i>	<i>Degrees</i>		
1	38.00	5.64	43.65	7.15	1.28	-24.50	1.4596	0.8758
2	39.47	6.62	46.08	8.39	1.01	-16.63	1.4613	0.8856
D2	43.05	5.68	48.73	7.20	1.06	-21.30	1.4644	0.9040
3	42.03	4.35	46.39	5.53	0.87	-19.86	1.4634	0.8973
D3	46.22	5.56	51.93	7.25	1.94	-22.17	1.4630	0.8940
4	44.56	5.78	50.34	7.34	1.08	-15.82	1.4645	0.8974
D4	46.59	4.97	51.56	6.32	0.64	-18.84	1.4635	0.8937
5	47.00	5.90	52.90	7.48	0.73	-16.88	1.4637	0.8983
D5	51.02	6.45	57.46	8.18	1.03	-18.68	1.4649	0.9067
6	52.70	7.06	59.76	8.97	1.08	-19.90	1.4643	0.9024
D6	53.86	8.04	61.90	10.23	0.72	-21.97	1.4630	0.9008
7	53.74	7.17	60.91	9.10	0.88	-22.44	1.4634	0.9015
D7	56.85	8.32	65.17	10.57	0.96	-25.28	1.4636	0.9000
8	53.53	8.34	61.87	10.58	1.02	-24.45	1.4621	0.8939
D8	57.35	9.84	67.19	12.49	0.83	-27.73	1.4624	0.9005
9	55.22	10.34	65.56	13.64	1.29	-26.59	1.4624	0.8936

* Each value is the average for the analysis of duplicate lots of mint.

¹At 25° C., D line of sodium spectrum.²At 20° C.³Oil at 25° C. referred to water at 4° C.

Table 2. PEPPERMINT OIL ANALYSIS—1945 CROP*

Sample	Free menthol	Combined menthol	Total menthol	Esters	Acid value: KOH per gram	Optical rotation ¹	Refractive index ²	Specific gravity ³
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Mg.</i>	<i>Degrees</i>		
1	42.59	2.96	45.55	3.75	0.36	-29.1	1.4596	0.8883
D1	43.67	2.96	45.63	3.76	0.48	-29.3	1.4599	0.8882
2	43.70	2.99	46.69	3.80	0.38	-25.2	1.4608	0.8870
D2	45.77	3.55	49.31	4.50	0.53	-27.2	1.4629	0.8971
3	44.49	3.56	48.06	4.52	0.66	-24.9	1.4648	0.9036
D3	46.57	3.75	50.32	4.76	0.76	-19.6	1.4650	0.9046
4	47.72	3.78	51.50	4.80	0.78	-15.9	1.4656	0.9052
D4	50.30	4.04	54.34	5.13	0.94	-18.4	1.4651	0.9054
5	51.88	4.45	56.33	5.64	0.82	-16.9	1.4659	0.9080
D5	55.30	4.30	59.60	5.46	0.74	-19.7	1.4635	0.9026
6	52.40	4.96	57.36	6.29	0.88	-18.0	1.4640	0.8942
D6	53.51	5.29	58.80	6.71	0.90	-20.2	1.4641	0.8977
7	53.74	5.95	59.68	7.54	0.47	-20.7	1.4631	0.9005
D7	52.35	5.70	58.05	7.24	0.56	-21.0	1.4625	0.8946
8	53.90	6.60	60.50	8.38	0.50	-23.2	1.4625	0.8974
D8	54.98	7.10	62.09	8.01	0.57	-25.0	1.4631	0.9005
9	56.33	6.66	62.99	8.46	0.59	-25.0	1.4636	0.9026
D9	56.40	7.02	63.42	8.91	0.85	-26.8	1.4628	0.8981

* Each value is the average for the analysis of duplicate lots of mint.

¹At 25° C., D line of sodium spectrum.²At 20° C.³Oil at 25° C. referred to water at 4° C.