

various dextrans should prompt other investigators to examine this action further. This paper has been concerned primarily with studies leading to the relative effects produced and not to their causes.

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## PEPPERMINT OIL

# Relation of Maturity and Curing of Peppermint Hay to Yield and Composition of Oil

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The yield of peppermint oil per acre is higher in the lower Yakima Valley of south central Washington than in any other known area. Peppermint plants bloom profusely and give maximum yield at time of full bloom. Oils contain an appreciable portion of blossom oil, high in menthofuran, and some practical method is needed to reduce the number of blossoms at harvest time. Retiming of irrigation and fertilizer application and clipping of blossoms might be of value. Harvest at an early stage of plant development might improve odor and flavor, but at the expense of total yield. Total menthol and esters increase with plant development. The optical rotation of the oil may or may not decrease to a minimum at the time of maximum yield. Length of curing after cutting has only a minor effect on composition.

PEPPERMINT OIL is obtained by the steam distillation of the peppermint plant (*Mentha piperita*). The Pacific Northwest is the source of about 60% of the oil produced in the United States; about 21% comes from the state of Washington (7).

Peppermint is grown on muck soils in the midwestern states of Michigan and Indiana, and on somewhat lighter soils in the states of Washington and Oregon. There are two main areas in the West which, according to the buyers, produce somewhat different peppermint oils, although the same plant, *Mentha piperita*, is grown in both areas, as well as in the Midwest.

In the older production area in Washington and Oregon, west of the Cascades in the Willamette and lower

Columbia valleys, peppermint is grown on heavy alluvial soils which are high in organic matter. There is occasional irrigation during the dryer parts of the growing season. The peppermint oil produced there is somewhat similar to that produced in the Midwest.

The second area, located in south central Washington, is in the lower Yakima Valley and along the Columbia River immediately below its junction with the Yakima River. Peppermint is grown on mineral soils that have developed under semiarid conditions, in an environment characterized by wide temperature fluctuations and more hours of sunlight of much higher intensity than in the other areas. Cultural conditions and practices in south central Washing-

ton may vary from those in other areas. This is the only known area growing peppermint entirely under irrigation. Irrigation is principally by furrow, but sprinklers are used to some extent.

There has been a rather rapid increase of peppermint oil production in this area. The yield of oil per acre in south central Washington is over three times that obtained in the Midwest and almost twice that of Oregon and western Washington.

Plant diseases and insect damage have decreased both acreage and yield in Indiana and Michigan. Rust has become a problem in Oregon and western Washington. As yet the lower Yakima Valley in Washington is free of these plant diseases and has only a small infestation of the mint flea beetle.

The published standards for American peppermint oil are those of the U. S. Pharmacopoeia XIV (11) for pharmaceutical use, which are for oils rectified by distillation, and neither partly nor wholly dementholized. To comply with these standards, the rectified oils must contain not less than 5% esters and not less than 50% total menthol. The physical constants must be within the following limits: specific gravity  $\frac{25^\circ}{25^\circ}$  0.896 to 0.908, optical rotation  $\frac{25^\circ}{D}$   $-18^\circ$  to  $-32^\circ$ , and refractive index  $\frac{20^\circ}{D}$  1.4590 to 1.4650. These standards were influenced largely by the composition and characteristics of the midwest-type oil, which earlier comprised most of the American production and still commands a price premium. Guenther (8) summarized data which show that many oils produced in other countries do not meet U.S.P. XIV standards.

The buyers of peppermint oil give much consideration to the odor and flavor of the oil, in addition to conformance to the U.S.P. XIV specifications. The standards for food, dentifrice, and other than pharmaceutical uses are less well defined. Most manufacturers desire an oil with an odor and flavor suitable for their individual products. Oil consumers are accustomed to the midwest flavor and odor and may consider anything else inferior. Methods for measuring odor and flavor are much less definite and tangible than the methods for measuring the physical and chemical properties. A buyer desires a natural oil which can be easily converted by rectification and blending into an oil that meets his requirements or the pharmaceutical specifications. If the natural oil obtained by steam distillation from the peppermint hay does not meet these standards, it means a loss of oil when the buyer rectifies it.

About 50% of all peppermint oil is used in chewing gum, 13% in confections and dentifrices, 2% in food flavoring and extracts, 32% is exported, and only about 3.5% of the American peppermint oil production is used for pharmaceutical purposes (10).

Information regarding the factors influencing yield, composition, physical properties, odor, and flavor of natural peppermint oils is limited. Cultural practices, together with climatic and soil conditions, may be factors. The amount, time, and method of irrigation, length of day, and amount and intensity of sunlight may have an important influence on the quality of the oil. The length of drying or curing of the hay before distillation may influence the yield, composition, and quality of the oil (13).

The stage of development of the peppermint plant at the time of harvest differs in the different areas of production. It appears to be about the same in the Midwest as in Oregon and western

Washington. In south central Washington mint plants develop more rapidly and are cut at a definitely later stage of development. No previous studies have been made in south central Washington to determine the effect of the stage of plant development or the length of the hay-curing period on the quality of peppermint oil.

### Literature Review

Sardanowsky (15) at Lubny, Russia, reported that the menthol content of the oil increases with the age of the plant and reaches a maximum toward the end of the blossoming. The ester content of the oil increases during the formation of the flower, declines a little during blossoming, and increases thereafter.

Rutowski and Trawin (12) at Moscow, Russia, showed that the menthol content of the oil increases with growth, while the menthone content decreases.

Bauer (7) reported the change in yield and menthol content of oils obtained from peppermint grown in different parts of Germany and cut at two different stages of development. The second cutting always produced some oil with a higher menthol and ester content. He then cut the plants, grown at Leipzig, at four different stages, before and at the time of budding and at the time of and after blooming. The yield of oil usually increased, but the menthol and ester content varied in several different ways, increasing and decreasing with no definite trend.

Ellis (7) reported on peppermint grown in Indiana and cut at several different times but did not mention the maturity of the plant, except to state that the last cutting was at half bloom. The ester and menthol content usually increased, while the optical rotation and refractive index reached a maximum and then decreased. The yield of oil steadily increased.

St. John and others (13, 14) reported that the peppermint grown in the Yakima Valley was harvested at a definitely different stage of development than in the other producing areas, and had been grown under very different conditions. Experimental plots were cut every week from the middle of July to September. The menthol content of the oil (determined by the viscosity method) tended to rise with the increasing maturity and the optical rotation reached a minimum at the time of full bloom.

Tornow and Fischer (17) reported that the natural peppermint oils produced in eastern Washington frequently do not conform to the U.S.P. XIV standards for rectified oils. On the average they have a less negative optical rotation and a slightly higher specific gravity and refractive index.

Bullis, Price, and Kirk (3) investigated, on a pilot plant scale, the effect

of curing on the yield and quality of the peppermint oil from the coast area of Oregon. The rain falling every few days on the drying peppermint presented a problem. The quantity and quality of oil from weathered mint were not materially different from those of freshly cut mint, if all the shattered leaves were gathered and distilled. Weathering losses appeared to be the result of a physical loss of leaves rather than a decrease in oil content. Rain during harvest and weathering are not a problem in eastern Washington.

Bullis, Price, and Kirk also reported that the menthol and ester content of the oil from peppermint grown in Oregon increased with the maturing of the plant during a period from before to well after full bloom. The optical rotation of the oil decreased to a minimum at full bloom and increased after that. The refractive index and specific gravity had no orderly variation but were at their maximum at full bloom.

### Experimental Methods

**Stage of Plant Development** The data reported show the effect of plant development on the yield and composition of commercially produced eastern Washington oil. This work covered five seasons, the first two of which were devoted to pilot plant studies to determine more definitely the experimental plan to be utilized.

**Pilot Plant Studies.** Peppermint hay was harvested from representative fields of several growers. In 1945 and 1946, samples were harvested in eastern (south central) Washington at weekly intervals from July 31 to September 5. In 1946, mint was also harvested at 2-week intervals in a western Washington field near Kelso. The growing conditions and the stage of plant maturity on a given date vary materially between eastern and western Washington. All samples were dried, and distilled in the laboratory at Pullman. The optical rotation of the oils was determined, and the menthol was estimated by a viscosity method (16). Later work raised a question concerning the validity of this method for the determination of free menthol in Washington-type oils.

**Commercial Harvests.** The experimental field work was done on a peppermint ranch in the Kennewick area in eastern Washington. Hay from  $\frac{1}{8}$ -acre plots was cut at intervals from a 6-year-old peppermint field as indicated in Table I, including one cutting after the commercial harvest date. Irrigation was discontinued one week before each harvest.

The peppermint was cut with a mower equipped with a modified swather attached, so that about one half of the windrow was folded over on the other half. The hay was bunched and rolled

into cocks with a dump rake, soon after cutting, so that the hay would be tough and shatter-resistant. At the expiration of half of the experimental drying period, the cocks were turned with pitchforks to promote uniform drying. This is the harvesting procedure followed by growers in the Kennewick area. The drying of the haycocks was not uniform. The hay on the inside of the cock remained green. Even after 96 hours of curing, some haycocks still contained green peppermint hay that had hardly wilted. Because of the unevenness of the drying, it was impossible to obtain representative samples for determination of moisture. The hay was cured for 3 days in 1948, 2.5 days in 1949, and 2 days in 1950. The hay was weighed on the truck by means of portable truck scales (Loadometer).

The cured hay was steam-distilled in the ranch still. Subsequent to the earliest harvest, distillation was complete in about 55 minutes. The temperature of the distillate was about 120° F. and that of the condenser water about 165° F. as it left the condenser. When no odor of peppermint could be detected in the steam escaping from a petcock in the top of the distillation tubs and no further oil appeared in the distillate, the distillation was considered complete. This is commercial practice. The vapors were condensed in a submerged aluminum tubular condenser. The oil and condensed steam were separated in a conventional gravity-type separator. The distillation time was noted. A representative sample of the weighed oil was taken for analysis.

Because of the much lower yields for the first harvests in 1949 and 1950, only enough hay was available to fill a pilot plant still twice. In 1950 it was necessary to utilize peppermint from a differ-

ent field, owing to uneven growth that year and to mint flea beetle infestation.

The peppermint oil samples were analyzed according to U.S.P. XIV procedures (77) for the examination of rectified peppermint oils. The refractive index, specific gravity, and optical rotation were determined, and the oil was analyzed for alcohols and esters. The total alcohol was calculated as total menthol. The difference between this and the esters is tabulated as free menthol. Ketones, calculated as menthone, were determined by means of a cold oximation procedure obtained from the A. M. Todd Co. While menthol, menthyl acetate, and menthone are the major components of peppermint oil, many other alcohols, esters, and ketones are present in the oil. As the drying of the hay was not uniform, no representative samples could be obtained for determination of moisture. The physical properties and composition of each sample are given in Table I.

**Curing** Selection of Material. Peppermint from an eastern Washington ranch was cut and allowed to cure for varying periods and then steam-distilled. The oils were analyzed to determine the influence of length of curing on physical properties and chemical composition.

The plots were selected from a weed-free, uniform field of peppermint. The same area was used in 1949 and 1950. Approximately 1<sup>2</sup>/<sub>3</sub> acres were divided into five strips, each having an area of 0.301 acre and extending completely across the field. Cutting dates and drying intervals are shown in Table II. As the hay for the second and third studies in 1950 was cut before the regular harvest, the yield and quality of the peppermint oils differ from those of the first curing study in 1949.

## Experimental Data

**Stage of Plant Development** Representative yield and optical rotation data for 1946 are shown in Figure 1. In 1946, the yield increased to a maximum on about August 10. Optical rotation progressively decreased from about -25° to about -15° at the time of maximum yield and thereafter increased slightly.

Botanical observations were made on plant development at each cutting of mint hay throughout the 1946 season (73). In the Kennewick area full bloom was reached at the August 5 harvest. The first spike was differentiated in the mint cut on July 15, but no flowers had opened on July 22, and on July 29 the flowers extended over one half or more of the length of the spike. At full bloom on August 5, the lower half of the first spike was past the flowering stage, and the hay had reached a maximum maturity. Thereafter, there was little formation of new leaves, while the stems continued to grow. This resulted in an increased proportion of stems to leaves in the harvested hay, which might have an unfavorable effect on the odor and flavor of the oil distilled. By August 26 very few flowers remained.

The development of the mint plants from the eastern Washington area was about a month ahead of that of the western Washington area. The first spike was not differentiated until August 13. The mint cut on August 30 in western Washington was at the same stage of maturity as that cut on July 29 in eastern Washington. In western Washington, Oregon, and the Midwest, harvesting is done at an earlier stage of plant maturity than in eastern Washington.

1948. Data for 1948, 1949, and 1950

Table I. Analytical Data on Maturity and Yield

Sample No.	Date of Harvest	Oil Yield, Lb./Acre	Refractive Index, 20° C.	Sp. Gr., 25° C.	Optical Rotation	Esters, %	Total Menthol, %	Free Menthol, % (by Diff.)	Menthone, %	Maturity Indicated by Blossoms
1948										
1	8-9-48	59	1.4612	0.9020	-19.4	4.8	38.2	33.4	37.2	
2	8-19-48	83	1.4620	0.9035	-20.4	4.8	41.7	36.9	31.2	
3	8-30-48	84	1.4618	0.9055	-20.1	5.4	47.7	41.7	27.7	
4	9-8-48	72	1.4618	0.9035	-17.7	5.6	50.5	44.9	24.3	
5	9-15-48	76	1.4621	0.9043	-17.6	7.2	55.2	48.0	18.6	
1949										
1	7-20-49	43	1.4591	0.8993	-20.3	3.0	32.4	30.0	42.6	Few
7	8-2-49	90	1.4612	0.9006	-18.2	3.1	36.1	33.6	35.0	Many
11	8-16-49	110	1.4601	0.9025	-18.5	4.3	40.2	36.9	33.9	Full bloom
16	8-23-49	114	1.4601	0.9026	-20.2	4.5	44.7	41.2	31.1	Some fading
26	8-30-49	111	1.4604	0.9019	-22.5	5.1	48.4	44.3	27.3	Majority gone
29	9-6-49	108	1.4611	0.9043	-22.4	5.6	52.5	48.0	21.5	None
1950										
1	7-18-50	56	1.4581	0.8971	-21.1	2.2	30.6	28.8	46.5	None
2	8-1-50	96	1.4621	0.9017	-16.4	3.4	36.5	33.8	37.8	Many
5	8-14-50	125	1.4636	0.9065	-10.7	3.2	38.0	35.4	29.2	Nearly full
12	8-21-50	138	1.4636	0.9074	-9.7	3.6	40.5	37.7	29.0	Full bloom
15	8-28-50	136	1.4628	0.9057	-12.2	4.0	42.6	39.4	26.7	Majority gone
16	9-4-50	127	1.4621	0.9050	-15.9	5.0	48.0	44.0	24.2	None

**Table II. Relation of Curing Time to Yield, Physical Properties, and Composition of Oil**

Sample No.	Curing Time, Hours	Distillation Time, Min.	Oil Yield, Lb./Acre	$n_D^{20}$	$D_{20}^{20}$	D	Esters, %	Total Menthol, %	Free Menthol, %	Menthone, %
Harvested August 14, 1950 (just before full bloom)										
6	9	65	90	1.4653	0.9063	-10.2°	3.48	40.0	37.3	30.0
7	28	78	106	1.4638	0.9066	-10.5	3.70	39.5	36.6	28.9
8	53	65	117.5	1.4633	0.9066	-11.3	3.43	37.7	35.0	29.8
9	72	60	116.75	1.4629	0.9056	-12.4	3.72	39.3	36.4	30.5
10	108	65	115.2	1.4631	0.9073	-12.7	3.88	39.1	36.0	29.8
Harvested August 21, 1950 (full bloom)										
11	8	74	90.0	1.4643	0.9080	-9.6	4.08	39.9	36.7	28.5
12	25	74	102.0	1.4640	0.9093	-9.0	3.91	40.7	37.6	29.3
13	48	52	140.0	1.4637	0.9074	-10.8	3.82	41.5	38.5	29.0
14	72	53	132.6	1.4639	0.9076	-10.5	3.70	41.4	38.5	27.8
15	96	55	134.5	1.4635	0.9068	-12.2	4.02	42.0	38.8	29.0
Harvested August 30, 1949 (just after full bloom)										
1	7	95	94.1	1.4620	0.9049	-17.6	4.66	45.4	41.8	25.2
2	28	85	97.4	1.4618	0.9047	-18.4	4.85	46.2	42.4	25.1
3	53	90	97.4	1.4616	0.9039	-19.7	5.12	47.5	43.4	23.8
4	77	..	90.8	1.4613	0.9049	-20.8	5.20	48.2	44.1	23.8
5	122	..	99.0	1.4618	0.9045	-19.9	5.20	48.3	44.2	23.4

are shown in Table I. In 1948 the yield of oil varied around 80 pounds per acre at all harvests except the earliest.

The refractive index and the specific gravity were essentially constant and at about the center of the range specified

for pharmaceutical oils. The optical rotation was above the minimum of  $-18^\circ$  for the first three harvests, but slightly below for the September harvests. Total menthol was above the standard for the last two harvests, slightly below for the third, and somewhat lower for the two earlier harvests. Menthone progressively decreased from 37.2% in the oil from the earliest harvest to 18.6% at the September 15 harvest.

**1949.** The maturity work in 1949 was a replication of that in 1948. This was the seventh year for this mint. Harvests were at 2-week intervals as shown in Table I. Very few blossoms were present on July 20, but by August 16 the field was in full bloom. The fields presented a more uniform appearance in 1949 than in 1948. The mint hay was cured for approximately 2.5 days.

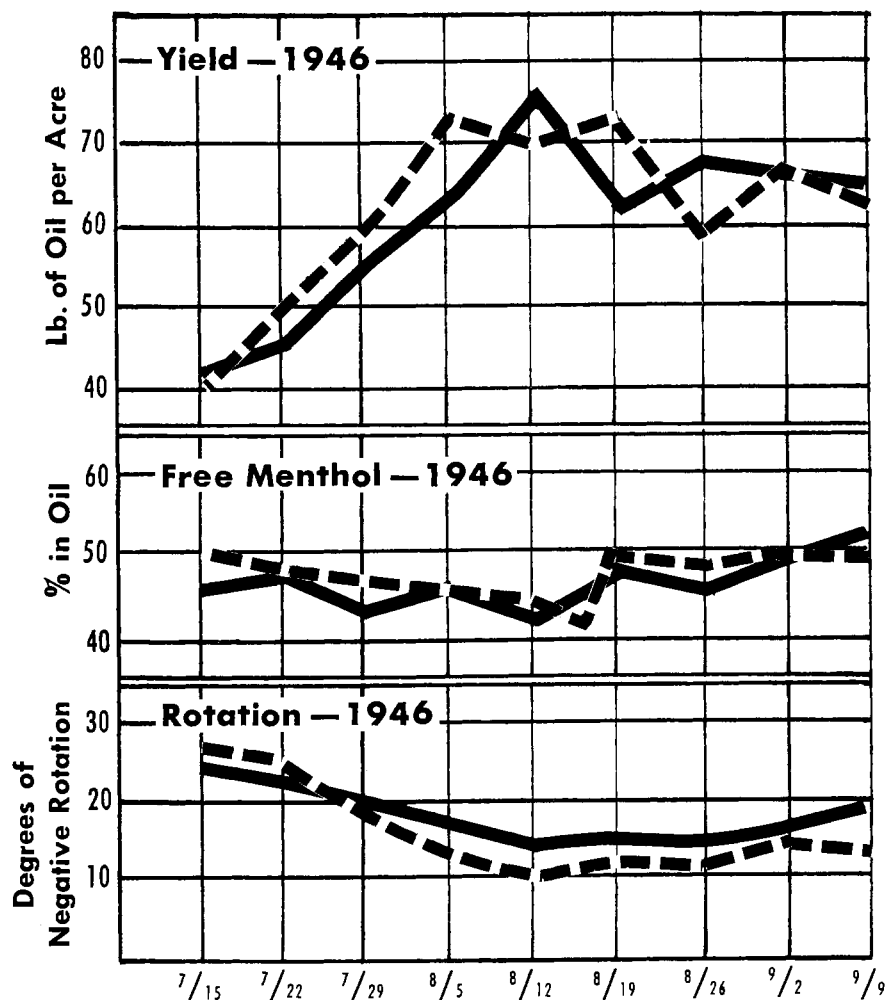
The yield increased rapidly from 43 pounds per acre on July 20 to 110 pounds and above on August 16 and 23. The yields were much higher than in 1948. Maximum yield was obtained a week after full bloom.

The refractive index increased slightly as the season progressed. All values were within the Pharmacopoeia limit and only slightly above the minimum. The specific gravity increased during the experimental period. All values except that for July 20 were within the Pharmacopoeia range and not greatly above the minimum. The July 20 value was only a trifle below the minimum.

The optical rotation varied from  $-20.3^\circ$  at the first harvest to  $-22.5^\circ$ , but at the second and third harvest dates was  $-18.2^\circ$  and  $-18.5^\circ$ . The oils were all above the minimum standard.

The esters varied from 3 to 5.6%. At the harvests on August 30 and September 6, the esters were above the 5%

**Figure 1. Composition and yield of peppermint oil from hay harvested at weekly intervals in 1946 from two different growers**



minimum. Total menthol varied from 32.4% on July 20 to 52.5% on September 6. Total menthol on September 6 was above the minimum, and on August 30 it was 48.5%. Menthone decreased from 42.7 to 21.5% during the experimental period.

While maximum yield was obtained on August 23, the ester content did not rise to the minimum until August 30, and the total menthol until September 6. Maximum yield was obtained a week after full bloom, and esters and total menthol reached the minimum 1 and 2 weeks, respectively, after full bloom. The yield varied little between August 16 at full bloom and any of the later harvest dates.

**1950.** The new field selected for the 1950 work was very uniform in growth. Mint was harvested on the dates shown in Table I. No blossoms were present on July 18, and full bloom was reached August 21. Blossoming was much more extensive than during either of the previous seasons, perhaps because of suboptimum irrigation early in the season. The cut hay was cured for 2 days. Data reported in this paper tend to show that this may be about optimum curing time in this area.

The yield of oil rapidly increased to 125 pounds per acre on August 14 and 138 pounds on August 21, and was materially higher than in the two previous years. Maximum yield of oil was reached at full bloom and a week past full bloom. The yield was high after all blossoms were gone and also in the week preceding full bloom.

The refractive index varied little after the first harvest and was well within the Pharmacopoeia range. The first harvest value was slightly lower. The specific gravity of the oil was somewhat lower in the first harvest than in the remainder. All, however, were within the Pharmacopoeia limits.

The optical rotation in 1950 progressively became less negative through the August 21 harvest and then increased somewhat. All rotation values were below the standard, except the one for the first harvest on July 18. The optical rotation was low in 1950 and reached a minimum of less than  $-10^\circ$  at full bloom on August 21.

The esters progressively increased throughout the season. The early values were unusually low, and reached the standard (5%) only at the last harvest on September 4, when all blossoms were gone. The total menthol progressively increased throughout the season. At the last harvest date it had not as yet reached the Pharmacopoeia minimum of 50%. The menthone progressively decreased throughout the season from 46.5 on July 18 to 24.2% on September 4.

**Second Growth.** If eastern Washington peppermint were harvested at a date substantially earlier than is customary, a second growth would result.

In 1946, second-growth hay was harvested on October 14 from a plot first harvested on July 15. The second-cutting plants were at a much earlier stage of maturity than those of the first cutting. The average second yield from two growers' fields was 20 pounds of oil per acre. The total yield from both cuttings was 80 pounds per acre. The optical rotation ranged from  $-28^\circ$  to  $-33^\circ$  in contrast to  $-18^\circ$  in the first cutting oil. However, the oil was not considered to have a good odor or flavor. There was a poor stand the next year.

## Discussion

**Stage of Plant Development** Oil yields were materially lower in 1946 and 1948 than in 1949 and 1950. The maximum yield was from August 20 to 25 in the latter three years and about August 15 in 1946. These larger yields continued through to about September 1, although there was a tendency toward some decrease in yield as the season progressed further. The largest yields came immediately after full bloom.

From hay harvested in the Kelso area in 1946, the maximum yield of oil per acre was produced about August 30. This hay was at an earlier stage of maturity than that of the eastern Washington hay at the date of maximum yield. The stage of maturity actually corresponded to that of the hay cut on July 29 in the Kennewick area. To obtain an optical rotation of the oil above the Pharmacopoeia minimum, the Kennewick hay should have been cut about July 22 in 1946.

The intervals between experimental harvests in 1948, 1949, and 1950 were a week or 10 days, and a more specific harvest date cannot be suggested. Each year consideration must be given to a number of factors, and it would not seem desirable to select the harvest date on basis of yield alone.

The optical rotation (levorotatory) sometimes appears to decrease to about the date when yield is at a maximum, and then increase. It appears from the 5 years' results that it might be necessary to harvest during the latter part of July to secure an oil with an optical rotation above the U.S.P. XIV minimum of  $-18^\circ$ . Oil produced in 1948 and 1949 had a rotation of about  $-18^\circ$  at the time of maximum yield, but in 1946 and in 1950 rotation was below  $-18^\circ$ . In 1950, when yields were the highest, the optical rotation varied around  $-10^\circ$  during the period of maximum yield and did not again reach  $-18^\circ$ . However, in 1949 when the maximum yields were about 110 pounds per acre, the rotation was above  $-18^\circ$  throughout the season. Thus, peppermint harvested during this period may or may not have an optical rotation above  $-18^\circ$ . The optical

rotation may not definitely be expected to increase to  $18^\circ$  if harvest is delayed until after September 1.

Observations on the 1946, 1949, and 1950 harvests suggested a relation between stage of development and yield and a less definite relation between stage of development and optical rotation. In each of these 3 years the maximum yield was obtained between full bloom and the later stage where blossoms no longer remained. In 1949, the optical rotation had begun to increase preceding full bloom; in 1946 and 1950 it was lowest following full bloom.

The total menthol progressively and rather rapidly increased throughout the season. In 1948 and 1949, the total menthol did not reach 50% until after September 1. In 1950, when the yields were the highest, the value was 48% at the September 4 harvest. Total menthol may or may not reach 50% if harvest is delayed until September 1 or later. The menthone progressively decreased from the earliest to the latest harvest date during each of the three seasons.

The sum of the total menthol plus menthone is very roughly the same for each harvest during the 3 years, but may vary somewhat from year to year. In 1950, it was lower during the period when maximum yield was obtained, owing both to lower menthol and to slightly lower menthone.

Esters were low in 1949 and 1950 and reached 5% about September 1. In 1948, esters varied from 4.8 to 7.2% and reached the 5% level by the time of maximum yield. Esters may, in general, be expected to reach the 5% level by about September 1, but they may reach the 5% level at an earlier date.

The 1948 results indicated that oils from hay harvested in late August or early September met the chemical requirements for pharmaceutical oils, although they did not meet buyers' requirements for buyers' purposes. It was difficult to correlate results with plant maturity in 1948, as all stages of maturity appeared present at each harvest.

Flavor was evaluated on 1948 oil samples by buyers, who concluded that the samples did not meet their requirements for odor and flavor. The flavor of the oil from early harvest before August 1 may be somewhat more acceptable, but is not considered by buyers equal in quality to midwest oils. It would seem that improvement in odor might be secured at the expense of higher yield and improvement in composition.

The results of Bullis, Price, and Kirk (3) coincide with those of the present investigation. They report maximum oil yield at about full bloom, an increase in menthol and esters throughout the season with a lowering of rotation, and a later increase after the time of full bloom.

In 1946, the hay was harvested at periodic intervals on a large ranch in southwestern Washington near Kelso. The maximum yield of 65 pounds per acre was obtained about August 30. Based on botanical observations (13), the peppermint plants at a given date were at an earlier stage of development in western than in eastern Washington. A given stage of maturity was about one month later in western Washington. The optical rotation was approximately constant at about  $-30$  and the menthol remained at about 70% throughout the period covered during the 1946 season in western Washington.

Maturity was studied in 1953 on samples collected at weekly intervals from August 10 to 31 from three growers in the Kennewick, Prosser, and Mabton areas. Approximately 100-pound samples of hay were cut, cured, and distilled in a pilot plant still. There was variation in the composition of the oil obtained from the different ranches. The spread in optical rotation was from approximately  $-10$  to  $-18$ . When taken together, the rotation curves for the oil from the three ranches in 1953 do not seem to show consistent trends. The menthol progressively increased during this 3-week period from an average of approximately 44 to 48% on August 31. The esters progressively increased from about 5 to 7% during this period. Menthone decreased, but not consistently, considering an average of the three ranches.

Menthofuran (6) increased somewhat between August 10 and August 31, but varied rather materially among the three ranches. All showed a fairly high menthofuran content, varying on August 31 from 16 to 20%.

Although there were variations among the different ranches, the 1953 results in general confirmed those obtained in 1948, 1949, and 1950.

**Curing** The effect of length of curing on composition was slight. Guenther (8) states that oil is removed from green hay with more difficulty. The time of distillation decreased in some cases with increased dryness of hay, permitting the distillation of more hay per day. Dry hay occupies less volume, so that more can be distilled in the tub. Both factors are advantageous, as they reduce the cost of operation. Longer curing might further increase this advantage. The dry hay is also easier to handle. Care must be exercised to prevent shattering and loss of leaves, as they contain the oil.

Evaluation of odor and flavor by several buyers and users of peppermint oil showed that the samples from the curing studies were not acceptable to them for flavoring purposes. Oil from the uncured hay had a slightly better odor. It is understood that peppermint oil from eastern Washington (usually

less than 10% and almost always less than 50%) is blended with peppermint oil from other areas to form an oil that is satisfactory for a specific use (14).

**Other Factors Related to Quality** Studies have pointed toward an interesting relation between the menthofuran content of peppermint oil and its quality. Menthofuran was first identified and isolated from American peppermint oil in 1948 by Bedoukian (2) and others. Studies initiated in 1948 by Watson suggested an interesting relation between the menthofuran content of peppermint oil and quality. Later Watson and Elliott showed an apparent relation between the extent of blossoming and the quality of the oil, particularly as judged by odor and flavor. Menthofuran has a rotation of  $+95$  (5). Blossom oil is high in menthofuran (4) and has a positive rather than a negative rotation. In this area data at present available indicate that it has a positive rotation of 18 to 33, depending on the stage of blossom development. Leaf oil has a negative rotation of  $-18$  to  $-24$ . Even 5% of blossom oil intermixed with the leaf oil would thus materially reduce the negative rotation of the oil, and might also contribute to an undesirable odor. Menthofuran is readily oxidized by air (18). Peppermint is harvested at a more advanced stage of development in south central Washington than elsewhere and the blossoming is therefore much heavier and more extensive than in other areas. The resulting oil contains a greater quantity of menthofuran and perhaps accompanying compounds. This may offer a logical explanation for differences in composition, including optical rotation, and differences in odor and flavor attributed to eastern Washington oils by the buyer.

The results presented in this paper point toward the importance of plant development in determining the quality of peppermint oil. This is strengthened by the studies made so far on blossoming and menthofuran. Practical methods of culture which will produce a maximum yield of a higher quality oil are thus needed. Delay of plant growth early in the season by some revision of cultural practices such as retiming of irrigation and fertilizer application, and perhaps clipping blossoms during midseason to produce hay that is less mature at harvest date, may prove to be of practical importance. The unusually high yields of peppermint oil in south central Washington justify further work of this type.

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