

THE PHARMACOPOEIAL ASSAY

Of

OIL OF SPEARMINT

By

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SCHOOL OF PHARMACY

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TABLE OF CONTENTS

I.	Introduction.....	
1.	Synonyms for oil of spearmint.....	2
2.	History.....	2
3.	Source and Origin.....	2
4.	Composition.....	3
II.	Spearmint oil in the pharmacopoeia.....	5
1.	United States.....	6
2.	British.....	7
3.	German.....	8
III.	The Chemical Assay of Spearmint Oil.....	10
1.	The neutral sulphite method.....	10
(a)	Process.....	10
(b)	Results.....	11
(c)	Comments.....	12
2.	The phenylhydrazine method.....	13
(a)	Process.....	13
(b)	Comments.....	14
3.	The hydroxylamine method.....	14
(a)	Process.....	14
(b)	Results.....	15
(c)	Comments.....	14
4.	The hydrogen sulphide method.....	15
(a)	Process.....	15
(b)	Results.....	15
(c)	Comments.....	15
IV.	Bibliography.....	16

ASSAY OF OIL OF SPEARMINT

1. Synonyms:

- Huile Volatile (Essence) de Menthe Verte (Fr.)
- Romischmenzol, Krausemenzol, Gruenmenzol (G.)
- Oleum Menthae Crispae

2. History:

Spearmint is one of the mints indigenous to temperate climates, some of which are cultivated, yielding valuable oils. Little appears to be known of the early history of spearmint, although several mints have been in use for culinary and medicinal purposes since very early times. No well defined distinction was made between them until comparatively recently, not even in the treatises on distillation of the fifteenth and sixteenth centuries, during which they were used extensively in the preparation of Aromatic waters. Spearmint may, or may not, have been one of the mints thus used.

3. Source and Origin:

Three kinds of spearmint oil are recognized:

American, German and Russian.

The American oil is distilled, principally in New York and Michigan from the fresh herb of *Mentha*, *Viridis* L. (*Mentha Spicata* L.), large quantities being produced. The same oil is also distilled in England from the same plant. The German oil is produced only in small amounts from *Mentha Crispa* L., or *Mentha longifolia*, in Thuringia where the plant is cultivated for medicinal purposes, only the waste

resulting in the process of drying being used for distillation. The Russian oil is obtained from *Mentha Verticellata*.

Mentha Viridis, from which the American oil is obtained, is regarded as a cultural variety of *Mentha Sylvestris* L., while *Mentha Crispa*, yielding the German oil, is probably a cultural variety of *Mentha Aquatica* L.

4. Composition:

While the composition of spearmint oil varies somewhat with the botanical source, the American, English, and German oils correspond fairly closely as will be seen from the accompanying table. The Russian oil, however, is so unlike the other oils of spearmint that it cannot be regarded as a substitute for them.

	American	English	German	Russian
Specific Gravity	0.92 to 0.94	0.926 to 0.935	0.936 to 0.952	0.88 to 0.89
Optical Rotation	-35° to -53°	-39° to -52°	-38° to -50°	-20° to -28°
Refractive Index	1.48 to 1.489	-	1.489 to 1.493	-
Acid Value	0 to 2	-	-	-
Ester Value	18 to 36	-	-	-
Carvone Value	35% to 66%	38% to 48%	61% to 72%	5% to 10%
Linalol	-	-	-	50% to 60%

The oil of spearmint of the United States Pharmacopoeia is required to contain not less than 43 per cent by volume of carvone and an assay for carvone is given. It is not to carvone, however, that the characteristic minty odor and taste of the oil is due, but rather to the 18 - 36 per cent of ester present. According to Elze⁽¹⁾, who studied the oil remaining after the carvone had been removed from German spearmint oil, the peculiar odor is due to the outer ester of dihydro-cuminic alcohol. Elze also identified l-phellandrene and traces of valeric acid in the oil.

Since spearmint oil is used principally for flavoring purposes, and the value of the oil appears to depend chiefly upon the presence of the ester which is apparently the bearer of the minty odor and taste, it may be that an assay based upon the ester value of the oil would be more rational than the present assay for carvone. This, however, is a matter which requires careful investigation.

(1)Elze - Chem. Zeit., 34 (1910) p. 1175

Spearmint Oil in the Pharmacopoeias.

Oil of spearmint has been official in the United States Pharmacopoeia since the first edition, 1820. No tests were introduced until the edition of 1860. Simple tests for volatility and solubility in water were then included. In 1880, a specific gravity requirement of 0.9 was given and a test for solubility in alcohol. The Pharmacopoeia of 1890 requires a specific gravity of 0.930 - 0.940 at 15° C. and gives tests for solubility in alcohol and other organic solvents. The edition of 1900 requires a specific gravity of 0.914 - 0.934 at 25° C., and an angle of rotation of -35° to -48°, in a 100 mm. tube at the same temperature, also a clear solution in an equal volume of 80 per cent alcohol. With the edition of 1910, the solubility requirement remains the same, the specific gravity is changed to 0.917 - 0.934 at 25° C., and the rotation to -38° to -55° in a 100 mm. tube. A chemical method of assay is also included.

Spearmint oil is found in the British Pharmacopoeia from 1858 - 1914, and in the German Pharmacopoeia from 1865 - 1880, when it was discontinued. It has never been included in the French Pharmacopoeia.

OIL OF SPEARMINT

UNITED STATES PHARMACOPOEIAS

Date	Official	Method of Preparation	Tests	Assay
1820	Yes	Distilled with water	None	None
1830	"	" " "	"	"
1840	"	" " "	"	"
1850	"	" " "	"	"
1860	"	" " "	"	"
1870	"	" " "	"	"
1880	"	" " "	Sp.Gr. 0.900	"
1890	"	" " "	Sp.Gr. 0.930 - 0.940 at 15° C	"
1900	"	Distilled from the fresh or partly dried leaves & flowering tops of spearmint rectified by steam distillation	Sp.Gr. 0.914 - 0.934 at 25° C Angle of rotation -35° to -48° in a 100 mm. tube at 25° C	"
1910	"	Distilled from the flowering plant.	Sp.Gr. 0.917 - 0.934 at 25° C optical rotation -38° to -55° in a 100 mm. tube at 25° C	Assayed for carvone by the neutral sodium sulphite method. The oil should yield not less than 43%, by volume of carvone <chem>C10H14O</chem>

OLEUM MENTHAE VIRIDIS

BRITISH PHARMACOPOEIA

Date	Official	Characters	Assay
1858- 1864	Yes	Colorless or pale yellow, with the odor and taste of spearmint	None
1867	Yes	Colorless or pale yellow, with the odor and taste of spearmint	None
1885	Yes	Colorless or pale yellow, or greenish yellow when recent, but becoming reddish by age, with the odour and taste of the herb	None
1898	Yes	Colorless, pale yellow, or greenish yellow when recently distilled, but becoming darker by age. It has the odour and taste of the herb, sp. gr. 0.920 - 0.940. The oil forms a clear solution with its own volume of a mixture of equal parts of absolute alcohol 90%.	None

OLEUM MENTHAE CRISPAEGERMAN PHARMACOPOEIA

Date	Official	Character	Assay
1865	Yes	soluble in all proportions of alcohol, sp. gr. 0.930 to 0.940	None
1872	Yes	soluble in large proportions of alcohol	None
1873	Yes	mixtures of alcohol with the oil in equal quantities are clear	None
1880	No		
1882	No		
1884	No		
1900	No		

OLEUM MENTHAE CRISPAE

FRENCH PHARMACOPOEIA

Date	Official	Character	Assay
1908	No		

The Chemical Assay of Spearmint Oil

The chemical assay as introduced into the ninth revision of the United States Pharmacopoeia is based upon the determination of carvone by a modification of the neutral sodium sulphite method used for the determination of benzalhyde in the eight revisions. The method follows:

Introduce 10 mils of the oil into a 200 mil flask with a long graduated neck (cassia flask) by means of a pipette, add 50 mil of a saturated solution of sodium sulphite, which has been carefully rendered neutral to phenolphthalein T.S. by means of acetic acid, heat the mixture in a bath containing boiling water and shake the flask repeatedly, neutralizing the mixture from time to time by the addition of a few drops of diluted acetic acid. When no coloration appears, upon the addition of a few more drops of phenolphthalein T.S. and heating for fifteen minutes, cool, and, when the liquids have separated completely, add sufficient of the sodium sulphite solution to raise the lower limit of the oily layer within the graduated portion of the neck and note the volume of the residual liquid. This measures not more than 5.7 mils, indicating the presence of not less than 43 per cent, by volume, of carvone.

The process is based upon the fact that many aldehydes and ketones react with neutral sodium sulphite forming soluble compounds. The sodium hydroxide which is liberated in the reaction is neutralized from time to time in order that the

reaction may go to completion. This process is reputed to give accurate results with carvone. Experience with the U.S.P. assay, however, has not been productive of faith in the method as there outlined. It has been found that a class of students working with the same oil and the same reagents, under as nearly as possible the same conditions, obtain widely different results. In the series of results given in the following table, the reagents were prepared by one student, the samples of oil were all measured out by the same student with the same pipette, from the same lot of oil, (U.S.P. spearmint oil, McKesson & Robbins). They were then distributed among a class of students who followed the U.S.P. directions, heating, shaking, and neutralizing the oil in a bath of boiling water. The individual differences in heating, shaking, and neutralizing were sufficient to bring about the differences in results.

Results obtained by the Class

Sample	c. c. residue	c. c. carvone	% carvone
1	8.6	1.4	14.
2	8.7	1.3	13.
3	8.4	1.6	16.
4	6.5	3.5	35.
5	7.2	2.8	28.
6	9.	1.0	10.
7	8.1	1.9	19.
8	6.2	3.8	38.
9	7.8	2.2	22.
10	6.8	3.2	32.
11	7.4	2.6	26.
12	8.4	1.6	16.
13	7.2	2.8	28.

There were also 11 other samples given out, but the results obtained were obviously incorrect, the per cent ranging from 4 to 9 per cent.

In view of these widely different results, it was thought desirable to run a series of assays upon the same sample in order to see how the results by one operator would correspond.

Sample	c.c. of residue	c.c. carvone	% carvone
1	8.1	1.9	19.
2	8.2	1.8	18.
3	8.5	1.5	15.
4	8.1	1.9	19.
5	8.1	1.9	19.
6	7.9	2.1	21.
7	8.2	1.8	18.
8	7.8	2.2	22.
9	7.9	2.1	21.
10	7.8	2.2	22.
11	7.8	2.2	22.
12	8.0	2.0	20.
Also two samples that involved an obvious error			
1	6.1	3.9	39.
2	6.3	3.7	37.

It was known that in these two determinations a little of the oil spattered out of the flask while shaking, hence the comparatively high results.

Other Methods of Assay

Other methods for determining the quantity of carvone in spearmint oil have been suggested as follows:

Phenylhydrazine method:

This is a method suggested by Parry⁽¹⁾ for the determination of carvone in oil of caraway.

Introduce about 15 mils of oil of spearmint into a tared 300 mil flask and note the exact weight; add 10 mils of an alcoholic solution of phenylhydrazine (1 in 10) (not darker in color than a pale yellow), allow it to stand for one-half hour at room temperature. Then add a few drops of methyl orange T.S. and neutralize the liquid exactly by the cautious addition of half-normal hydrochloric acid V.S. If difficulty is experienced in detecting the end point of the reaction, continue the titration until the solution is distinctly acid, transfer it to a separatory funnel and draw off the alcoholic portion. Now wash the oil with distilled water, adding the washings to the alcoholic solution and titrate the latter with half-normal potassium hydroxide V.S. Carry out a blank test identical with the foregoing, except that the oil of spearmint is omitted, and note the amount of half-normal hydrochloric acid V.S. consumed. Subtract the number of mils of half-normal potassium hydroxide V.S. from the number of mils of half-normal hydrochloric acid V.S. consumed in the original test and this result from the corresponding number of mils required in the blank test; each mil of this corresponds to .075 grams of ketones, calculated as carvone. It shows when calculated from the

(1) Parry - The Chemistry of the Essential Oils, Vol. I, p. 290

weight of the oil taken not less than 43 per cent of ketones from oil of spearmint calculated as carvone.

The crystals obtained are so difficult to dry without decomposition as to render the determination only approximate.

Hydroxylamine Method:

This method was recommended by Kremers and Schreiner⁽¹⁾ in 1897. To a solution of 10 grams of oil in 25 c.c. of alcohol, 5 gm. of hydroxylamine are added, and the mixture is boiled upon a water bath connected with a reflux condenser for half an hour. 25 c.c. of water is added, and the alcohol which carries over a large quantity of limonene is distilled off by the heat of the water bath. Steam is then passed through the liquid until traces of carvoxime come over, as evidenced by the formation of crystals in the last portion of distillate collected separately in test tubes. The tube of the condenser is washed of any carvoxime which is returned to the flask.

The contents of the flask is allowed to cool and carvoxime removed, washed and dried by suction.

The air dried carvoxime is then transferred to a tared glass dish and heated for one hour on a water bath and when cool, weighed. To this weight, .1 gm. is added to make up for quantity lost by heating.

The weight of carvoxime ($C_{10}H_{14}NOH = 164.67$) X
0.9088 gives that of carvone ($C_{10}H_{14}O = 150.11$).

This method of assay has not been found to be satisfactory. The point at which distillation should be

(1) Pharm. Rev., 14, p. 244; (Proc. of A.Ph.A., 45, p. 645)

stopped when the difficulty volatile carvoxime begins to come over is hard to determine. It can give at best only approximate results. Three determinations run upon the same oil, previously reported, gave the following results:

Sample	#1	#2	#3
Weight of oil	10.356	10.422	10.342
Weight of Carvoxime	2.965	3.102	2.884
Weight of Carvone	2.694	2.819	2.620
Per cent of Carvone	26.01	27.04	25.33

Carvone Hydrogen Sulphide Method:

This method of assaying by the determination of the sparingly soluble carvone hydrogen sulphide has also been suggested as follows:

Assay - dissolve weighed portion (about 10 c.c.) of the oil in about 30 c.c. of alcohol. Then add enough ammonium hydroxide to make the solution strongly alkaline. Then pass in hydrogen sulphide until a precipitate is formed, filter and wash with cold alcohol. Dry, weigh, and crystallize from glacial acetic acid and again dry and weigh.

The weight of carvone hydrogen sulphide ($C_{10}H_{14}O \cdot H_2S = 184.18$) X 0.8150 gives that of carvone ($C_{10}H_{14}O = 150.11$)

A series of three determinations run upon the oil reported upon before gave results which checked very closely, but which were lower than those obtained by any other process:

Sample	#1	#2	#3
Weight of Oil	10.078	10.030	10.064
Weight of Carvone		2.186	2.194
Hydrogen Sulphide	2.202	17.77	17.88
Per cent of Carvone	17.86		

What is the chemical relation, if any, between the oils of peppermint and spearmint?

Prov. of A. Ph. A., 33, p. 500;

1. The oils of spearmint and peppermint probably contain hydrocarbons which are identical.

2. These hydrocarbons exist in much smaller proportions than heretofore supposed, and are isolated with great difficulty.

3. Oil of spearmint contains, as the oxygenated portion, carvol, $C_{10}H_{14}O$, which does not solidify at $-23^{\circ}C$, and is precipitated by alcoholic ammonium sulphide.

4. Oil of peppermint contains, as the oxygenated portion pipmenthol, $C_{10}H_{20}O$, which is a crystalline solid at ordinary temperatures, and is not precipitated, when in solution by alcoholic ammonium sulphide.

5. Both oils contain resins almost free from odor, and formed in part during the process of distillation.

Gilman, W. F.

1893

Terpenes of Spearmint Oil

Western Drug, 15, p. 323, (Proc. of A. Ph. A.,
46, p. 695.)

The composition of spearmint oil as ascertained
was; laevogyrate carvol and laevogyrate limonene.

Umnney, J. C.

1895

Spearmint Oil

Pharm. Jour. Trans., 1895, 1045; (Proc. of
A. Ph. A., 43, p. 1039)

Characters and tests:

Sp. gr. at 15° C. .920 to .940, optical rotation
in a tube of 100 mm. -35 to -50. Not less than 35 per
cent should boil between 222°+ and 226° C. One part should
dissolve in one part of alcohol 90% strength, becoming tur-
bid upon further dilution.

Kremers, E. and Schreiner, O.

1897.

Oil of spearmint - Application of the carvoxime method to determine adulteration.

Pharm. Rev., 14, p. 244; (Proc. of A. Ph. A., 45, p. 645)

Carvoxime method as practically applied in the estimation of carvone in adulterated spearmint oil, can be used for the detection of other oils and as cedarwood and Gurjun Balsam which are frequently used. It is shown that it gives better results than the fractional distillation method also using smaller quantities of oil, therefore being more economical.

Schimmel & Co.

1897.

Oil of spearmint, yield and characters.

Schimmel & Co. Semi-Ann. Rep., April 1897; (Pharm. Rev., 15, 115; Proc. of A. Ph. A., 45, p. 645.)

A yield of 0.3 per cent of oil from first cut of spearmint in July and only 0.18 from the second crop in October. Oil of the second crop was less fine in odor, and had a lower sp. gr. and rotatory power.

Tiemann, F.

1898

Ber. 31, p. 3317

Ueber Hydrosulfon saure abkoemin linge des
zimntaldehyds, des Citronellals und des Citrals.

A long article discussing the various compounds
of citral and citronellal.

Tiemann, F.

1898

Ber. 31, p. 3324

Zum Nachweis und zur quantitativen Bestimmung
des Citrals

The application of the sodium bisulphite reaction
to the determination of citral.

Schimmel and Co.

1898

Oil of spearmint - Difference observed between
the German, American and the Russian oils.

Proc. of A. Pb. A., 46, p. 973.

Russian oil shows a striking difference from German
and American oils, by a stale odor, a lower sp. gr. (0.88)
and a lower optical rotation (-23°), while both other kinds
have a sp. gr. between 0.92 and 0.98 and an optical rotation
as high as -43° . The main difference between the two kinds
of oils is due mainly to the relatively high percentage of
linalool and a deficiency of carvone of the Russian product.

"A basic reaction of aromatic and fatty aldehydes".
Am. Jour. Pharm. 76, p. 84

Upon heating a solution of sulphite of soda with the oil, the dihydrodisulphonate of sodium is formed which is soluble in water, and two molecules of sodium hydrate are split off.

"Determination of Aldehydes and Ketones"

Schimmel & Co. Semi-Ann. Rep. April 1905, p. 103.

According to Burgess, 5 c.c. of oil are mixed in a cassia flask ⁽¹⁾ with a saturated solution of sodium sulphite, and the mixture, after adding two drops of phenolphthalein solution, is heated on the water bath with frequent agitation. The sodium hydroxide liberated during the reaction is from time to time neutralized with dilute acetic acid (1:10), until no further red coloration occurs when it is heated. The oil is then brought into the neck of the flask by adding water, and after cooling its volume read accurately. By multiplying the absorbed quantity of oil with 20, the percentage of aldehyde or ketone is found.

Burgess has tested this method with satisfactory results on a whole series of aldehydes and ketones.

(1) Burgess uses for this purpose flasks which differ from our cassia flasks in having at the side (for filling in liquids) which reaches to the bottom of the vessel. We consider the original form of our cassia flasks more convenient and suitable.

Recent determinations by the sulphite method has convinced us that in some cases it is very useful, although according to our present experience it cannot be adapted so generally as Burgess claims. Very good results are obtained in the determination of carvone and can be strongly recommended. But it should not be lost sight of that by this method the carvone-content is ascertained in percentages by volume, and not in percentages by weight and which for example in caraway oil makes a difference of about 3%.

Schimmel and Co.

1906

Russian Spearmint Oil - Constants.

Schimmel and Co. Semi-Ann. Rep. Oct. 1906,
p. 73; (Proc. of A. Ph. A., 55, p. 882.)

Russian spearmint oil differs from American and German distillates, chiefly by its high content of linalool and low carvone content. Sample examined had a stale, feeble odor like spearmint; sp. gr. at 15°C, 0.8873; optical rotation, $-25^{\circ} 16'$; index of refraction, 1.47078; acid number, 1.1; ester number, 15.9; soluble in 2.2 vols. and more 70 per cent, and one or more volumes of 80 per cent alcohol. The American and German distillates in distinction from these constants show the sp. gr. at 15°C. of 0.92 - 0.94, optical rotation -36° to 48° ; soluble in one vol. and more 80 per cent alcohol, the dilute solution having a faint opalescence.

"Characteristics of English Oil of Spearmint"

Pharm. J. London, 25, p. 506; (Digest of Comment on U.S.P., 1907 p. 346.)

Report on the characteristics of two samples of English oil of spearmint. Oil distilled in 1906 had a sp. gr. of 0.931 and was soluble in its own volume of 90 p.c. alcohol. The angle of rotation in a 100 mm. tube was $-50^{\circ} A_D$, temperature 21.5° . Oil distilled in 1907 had a sp. gr. of 0.927, and was soluble in its own volume of 90 p.c. alcohol. The angle of rotation in a 100 mm. tube was $-50^{\circ} A_D$, temperature $20^{\circ}C$.

Evans Sons, Lescher and Webb.

1908

"Analytical Report"

Evans Sons, Lescher and Webb (Analytical Notes, 1908, p. 38); (Digest of Comment on the U.S.P. 1908, p. 397.)

Five samples of spearmint oil examined gave figures ranging as follows: specific gravity from 0.920 to 0.934; optical rotation, $-41^{\circ} 30'$ to $-44^{\circ} 0'$; soluble in from 1 to 4 volumes of 90 per cent alcohol.

Lehn and Fink

1908

"Contrasts of Samples of Oil of Spearmint"

Lehn and Fink's Annual Rep. for 1908, p. 25,

(Digest of Comment on the U.S.P. 1908, p. 397.)

Out of 4 samples of oil of spearmint examined, one contained oil of peppermint; the test by which it was detected is outlined.

Smith, Kline and French Co.

1908

"Assay Report"

Smith, Kline and French Co. Analytical Report,

1908, p. 28; (Digest of Comment on the U.S.P. 1908, p. 397.)

Three samples of spearmint oil examined were of U.S.P. quality except that one had an optical rotation 2° below the limit.

Schimmel and Co.

1908

"B.P.C. Requirements of Oil of Spearmint"

Schimmel and Co's. Semi-Ann. Rep., April 1908, p.

147 - 148; (Digest of Comment on the U.S.P., 1908, p. 397.)

Spearmint oil is soluble in about its own volume of 80 per cent alcohol, the solution becoming opalescent on adding more solvent.

Gane and Webster

1908

"Adulteration with peppermint oil"

Drug Topics, N.Y. 23, p. 235; (Digest of Comment on the U.S.P. 1908, p. 397.)

The enormous demand for oil of spearmint during the past year has exhausted all available supplies. Several samples obviously adulterated with peppermint oil have been met with.

Schimmel and Co.

1908

"Production of Oil of Spearmint"

Schimmel and Co. Semi-Ann. Rep., April, 1908;

(Digest of Comment on U.S.P. 1908, p. 397.)

Schimmel and Co. point out that in America spearmint is produced only in Michigan, the total production being between 5,000 and 8,000 pounds, and that the increased demand for this oil for the manufacture of chewing gum has greatly advanced the price.

Schimmel and Co.

1908

"Report of the Therapeutic Committee of the British Medical Association".

Pharm. J., London, 27, p. 811; (Digest of Comment on the U.S.P. 1908, p. 397.)

It is here suggested that *Oleum Mentha viridis* be deleted from the Ph. Brit., as it is very little used and is unnecessary.

Schimmel and Co.

1909

Hungarian Spearmint Oil - High Carvone content.

Schimmel and Co. Semi-Ann. Rep., April, 1909, p. 85;

(Proc. of A. Ph. A. 57, p. 326.)

Spearmint oils from Hungary examined which were distilled from the dry herb had about the same constituents as the German and American but had a higher carvone content which runs up to 72 per cent against 50 per cent in German and American oils. Also the sp. gr. was somewhat higher than that of other varieties and had greater solubility.

Lehn and Fink

1909

"Adulteration"

Proc. of Am. Pharm. Ass., 57, p. 737; (Digest of

Comment on the U.S.P. 1909, p. 538).

The Committee on Drug market (quoting Lehn & Fink) reports one sample of oil of spearmint containing oil of peppermint.

Schimmel and Co.

1909

"Superior quality of New York Spearmint Oil"

Schimmel and Co's. Semi-Ann. Rep., April, 1909, p.85;

(Digest of Comment on U.S.P., 1909, p. 538).

Assertion is made that little spearmint is cultivated nowadays in the state of New York, which is to be regretted because the oil yielded by the New York herb was superior in quality to the oil from Michigan. They also report that Hungarian spearmint oil is richer in carvone than German and American oils.

Elze, F.

1910

Oil of Spearmint - Odorous Constituent.

Chem. Ztg., 34, p. 1175; (Pharm. Jour. and Pharmacist, 85, p. 787; Proc. A. Ph. A., 59, p. 404)

When l-carvone is prepared from oil of spearmint by Tiermans method, an oil is obtained as a by-product which has a more intense spearmint odor than the original oil. On fractionating this by-product and the saponification of the higher boiling fraction, acetate of Dihydro cuminic alcohol was obtained, which has the characteristic odor of spearmint and to which the odor of the natural oil is due.

Eldred, Frank R.

1910

"Variation of Constants"

Proc. Am. Pharm. Ass., 58, p. 895; (Digest of Comment on U.S.P., 1910, p. 603.)

Four lots of oil of spearmint were found to vary in sp. gr. at 15° C. from 0.933 to 0.940; optical rotation, from -43.3° to -50.2°; and the index of refraction from 1.4880° to 1.4885°; soluble in an equal volume of 80 p.c. (by volume) alcohol.

Evans Sons, Lescher and Webb

1910

"Report on samples"

Evans Sons, Lescher and Webb, (Analytical Notes, 1910, p. 71); (Digest of Comment on U.S.P., 1910, p. 603).

Report on 2 samples of Spearmint Oil, English: sp. gr. 0.959, and 0.9593; optical rotation, -37° and

$40^{\circ} 30'$; soluble in 0.5 volume 90 p.c. alcohol. These were genuine oils, although the sp. gr. is outside the Ph. Brit. limits. Two samples of American oil had sp. gr. of 0.932 and 0.9358; optical rotation $-48^{\circ} 30'$ and $-51^{\circ} 36'$.

Schimmel and Co. 1910

"Proposed Ph. Brit. requirements"

Schimmel and Co's. Semi-Ann. Rep. April, 1910, p. 137; (Digest of Comment on U.S.P., 1910, p. 603.)

Commenting on the proposed Ph. Brit. requirements, states that the minimum limit of sp. gr. at 15.5° should be 0.920. It is soluble up to 1 volume in 1.5 parts of 90 p.c. alcohol.

E. Sachsse and Co. 1910

"Optical Rotation Correction"

Brit. Col. Drug. 57, p. 241; (Digest of Comment on U.S.P. 1910, p. 603).

An optical rotation of -30° to -55° would be more correct as a great many pure American oils would rotate higher than -50° .

Hill and Umney 1910

"Constants and Tests of Spearmint Oil"

Pharm. J. 30, p. 179; also Chem. & Drug., 76, p. 272; (Digest of Comment on U.S.P. 1910, p. 602).

Suggest for the oil distilled from the fresh flowering spearmint, *Mentha Viridis* or *M. Crispa*: colorless, pale yellow

or greenish yellow, becoming darker on keeping, having the characteristic odor and taste of the herb. Sp. gr. at 15.5° , 0.925 to 0.940; optical rotation at 20°C , -30° to -50° .

Forms a clear solution with equal volumes of 80 p.c. alcohol, the solution becoming turbid upon further dilution, soluble in 3 volumes of 90 p.c. alcohol.

Unsigned Article

1910

"Specific Gravity of Spearmint Oil in Ph. Russ."

Chem. Drug., 77, p. 687; (Digest of Comment on U.S.P. 1910, p. 602).

Calls attention to the wide latitude allowed by the Ph. Russ. in the sp. gr. of Oleum Menthae Viridis, 0.900 to 0.940.

Pearson and Sechler

1910

"Report on Specific Gravity of Oil of Spearmint"

Merck's Rep., 19, p. 45; (Digest of Comment on U.S.P., 1910, p. 602),

All samples of Oil of Spearmint they have examined have had sp. gr. from 0.918 to 0.928. The U.S.P. limits might be made narrower.

Jeancard and Satie

1910

"Inexactness of Pharmacopoeial procedures"

Pharm. Era, 1910, 43, p. 143, (Digest of Comment on U.S.P. 1910, p. 602).

Jeancard and Satie think the analytical procedures of the Pharmacopoeia to be inexact.

Schimmel and Co.

1910

"Production of Oil of Spearmint"

Schimmel and Co's (Semi-Ann. Rep. Oct. 1910, p. 131). (Digest of Comment on U.S.P., 1910, p. 602)

A review of the production of oil of spearmint during the past year.

Schimmel and Co.

1911

"Difference of taste between Europeans and Americans"

Schimmel and Co's (Semi-Ann. Rep. April, 1911, p. 106); (Digest of Comment on U.S.P. 1911, p. 487).

Judged by European standards of taste, it seems scarcely credible that the employment of this oil in the manufacture of sweets, chewing gum, etc., should be steadily increasing in the United States.

Evans Sons, Lescher and Webb

1911

"Report on samples"

Evans Sons, Lescher and Webb (Analytical Notes, 1911, p. 66); (Digest of Comment on U.S.P., 1911, p. 487).

Two samples of spearmint oil gave a sp. gr. of 0.9317, and 0.9360; an optical rotation, -34.48°, and 51.30°; refractive index 1.4823 and 1.4892; and were soluble in 0.75 to 1 volume of 80 p.c. alcohol.

Smith, Kline and French Co.

1911

"Report on Samples"

Smith, Kline and French Co's (Analytical Report, 1911, p. 33); (Digest of Comment on U.S.P., 1911, p. 487).

Two samples of oil of spearmint were found to be of U.S.P. quality with the exception that one had an optical rotation 3° above the upper limit of the U.S.P.

Schimmel and Co.

1911

"Increased Acreage of Spearmint"

Schimmel and Co's Semi-Ann. Rep. Oct. 1911, p. 84; (Digest of Comment on U.S.P. 1911, p. 487).

In the states of Michigan and Indiana, 1,727 acres of spearmint are under cultivation, and more than half of this, viz, 921 acres, represents new plantations.

They also (Ibid. April, 1911, p. 133) in discussing the Ph. Russ. VI. requirements for spearmint oil, point out that the dilute solution of spearmint oil in 90 p.c. alcohol has an opalescent turbidity.

Schimmel and Co.

1912

"Hungarian Spearmint Oil"

Schimmel and Co's (Semi-Annual Rep. April, 1912, p. 118); (Digest of Comment on U.S.P. 1912, p. 367).

According to K. Irik, Hungarian spearmint oil constitutes a straw-colored or faintly greenish yellow liquid; sp. gr. at 15° compared with water at 4° , 0.9375 to 0.9513; optical rotation, 44.38° to 49.85° .

Schimmel and Co.

1912

"Acreage of Spearmint"

Schimmel and Co's (Semi-Ann. Rep. Oct. 1912, p. 103); (Digest of Comment on U.S.P. p. 367).

The total area under spearmint cultivation in Michigan and Indiana amounts to 2,057 acres as compared with 1,520 acres in 1911.

Schimmel and Co.

1913

"Increased Consumption"

Schimmel and Co's (Semi-Ann. Rep. 1913, p. 96); (Digest of Comment on U.S.P. 1913, p. 384).

Consumption of spearmint oil has increased to such an extent that available supplies of oil will most probably be insufficient. The bulk of the production is consumed in the United States.

Mann, E. R.

1914

"Report on Samples"

Ann. Rep. Southall Bros and Barclay, 1914, p. 40; (Digest of Comment on U.S.P., 1914, p. 384).

The characters and tests of both samples of oil of spearmint were practically in accord with the requirements of the Ph. Br. V.; sp. gr. was 0.931 and 0.930; optical rotation -43.5° and -51° ; refractive index 1.4865 and 1.4855.

Rabak, Frank

1914

"Variation of solubility"

J. Am. Pharm. Ass. 3, p. 1673; (Digest of Comment on U.S.P. 1914, p. 384).

Oil of spearmint distilled during several successive seasons at Arlington exhibited decided variations of solubility in 80 p.c. alcohol. One sample showed an optical rotation above that of the maximum in the Pharmacopoeia.

Schimmel and Co.

1914

"Yield of 1913 crop"

Schimmel and Co. (Semi-Ann. Rep., April, 1914, p. 92); (Digest of Comment on U.S.P. 1914, p. 384).

Information gathered by our New York firm, states that the 1913 crop has yielded about 10,000 lbs. of oil more than 1912.

Unsigned Article

1914

Oleum Menthae Viridis

Yr. Bk. A. Ph. A. 3, p. 1109

Oleum Menthae Viridis distilled from flowering plant of *Mentha Spicata* Linne, (*Menthae Viridis* Linne) (Fam. Labiatae) added rubric; yielding not less than 40 per cent, by volume, of carvone, Laevogyrate changed from "-35° to -48°" to "-35° to 50°" in a 100 mm. tubes at 25° C. Assay for carvone, assay as directed for carvone under Oleum Cari.

Rabak, F.

1918

Influences affecting the yield of oil of spearmint.

J. Ind. Eng. Chem., 10, p. 275; (Yr. Bk. A. Ph.

A. 7, p. 423).

Yield varies with seasonal conditions. The greatest yield being at the flowering period. Drying reduces oil content. Ester and alcohol content increases with maturity. Freezing increases ester and alcohol content.

Approved Nellie A Wakeman

Instr. in Pharms. Chem.