OIL OF PEPPERMINT AND ITS CONSTITUENTS

A Thesis submitted for the Degree

of

GRADUATE IN PHARMACY

by

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The following synonyms are those of the principal plants from which Oil of Peppermint is derived:

MENTHA PIPERITA. L. (152) *

MENTHA.

balsamea	(Willd.)
glabrata	(Vahl.)
hircinia	(Hull.)
hispidula	(Poepp.)
Kahirina	(Tersk.)
languii	(Geig.)
napolitana	(Tenore.)
odora	(Salisb.)
officinalis	(Nees.)
piperita	(Stokes.)
Tenuis	(Frank.)
)Menthella ( )Requienii	(Perard.)

MEXICAN SUBSTITUTE. (113)

yerbabuena piperita

hedeoma piperita

* Numbers refer to corresponding references.

MENTHA ARVENSIS L. (152)

MENTHA.

agretis (Sole.)
allionii (Bor.)
augustifolia (Schreb.)
anomala (Herib.)
arguta (Opiz.)
atrobirens (Host)
austriaca (Jacq.)
badensis (Fellm.)
barbata (Opiz.)
bracteolata (Opiz.)
compestris (Schur.)
carnithiac (Host.)
carniolica (Host.)
coerulea (Opiz.)
crenata (Becker.)
dalmatica (Tausch.)
deflexa (Dunn.)
densiflora (Opiz.)
divaricata (Host.)
dubia (Chaix.)

ehrhartiana	(Lej & Court)
elata	(Host.)
elegans	(Lej.)
elliptica	(Lej.)
flagelliferae	(Schur.)
fontana	(Weihe.)
gentilis	(Georgi.)
glabra	(Colla.)
gracilis	(Sole.)
grabeoleus	(Opiz.)
Hakka	(Siebolt.)
Hellebrantii	(Ortin.)
Hostii	(Bor.)
javanica	(Blume.)
laniiflora	(Tenore.)
lanceolata	(Benth.)
lapponica	(Wahlent.)
lata	(Opiz.)
latifolia	(Host.)
latissima	(Schur.)
laxa	(Host.)
longifolia	(Host.)

maculata (Host.)

marrubiastrum (Schulz.)

melissaefolia (Host.)

minor (Opiz.)

montana (Host.)

mesana (Lej.)

muellenana (Seh.)

multiflora (Host)

mesiana (Opiz.)

nemorasa (Host.)

nitida (Host.)

nobilis (Weihe.)

nummularca (Shreb.)

obtusata (Opiz.)

ocymoides (Host.)

oreganifolia (Host.)

ovalifolia (Opiz.)

ovata (Schur.)

palatina (Schulz.)

pilosa (Spreng.)

plicata (Opiz.)

polymorpha (Host.)

praecox (Sole.)
practicola (Opiz.)
procumbens (Thuill.)
prostata (Host.)
pulchilla (Host.)
pulegium (Luce.)
pimula (R.Grah.)
rivalis (Sole.)
Rothii (Nees.)
rotundata (Opiz.)
rubrohirta (Lej.)
salebrosa (Bor.)
sativa (Roxb.)
scordiastrum (Schulz.)
scribae (Schulz.)
serotina (Host.)
slichoviensis (Opiz.)
speckmoseriana (Opiz.)
stachyoides (Host.)
suaveoleus (Host.)
subcordata (Collg.)
subspicata (Weihe.)

tenuifolia (Host.)
tortusa (Host.)
varians (Host.)
verticellata (Linne.)
villosa (Becker.)
virgata (Salisb.)
viridula (Host.)
Wohlwerthiana (Schulz.)

HISTORY.

Peppermint oil was first obtained from mentha piperita. Distillation of peppermint oil first accomplished in America by Mr. Burnette in 1816, in Wayne County, New York. He obtained about forty pounds of oil from wild plants. In 1835 the industry was established in Michigan, in St. Josephs County on White Pigeon prairie, about two miles from the village by that name; a distillery being erected the following year. Up to this time and for ten years later, the distilling apparatus used was very crude, being the same as had been used in England with a few slight modifications. It consisted of a copper kettle, in which the plants were placed, immersed in water, to which direct heat was applied from a furnace beneath; a condensing worm of the

usual character being connected with the kettle by a pipe from its apex.

In 1846 new distilling apparatus came into use, in which steam was allowed to pass through the plants. The plants were placed in wooden containers, and the steam entered through the bottom through a pipe.,

The primitive distilleries had a capacity of about fifteen pounds of oil in twenty-four hours, the new way could produce from seventy-five to one hundred pounds. This system of steam distillation originated in St. Josephs County, Michigan. (95)

Whether or not peppermint was among the mints used during the Middle Ages can no longer be determined. Whether it agrees with the mints now in use, is not known. As far as known the only specimens of mentha piperita which are several hundred years old, are found in the herbarium of the British Museum in London. John Ray, the English naturalist, had obtained them in 1696 in England.

The principal places of production are the states of New York, Michigan, and Indiana. Whereas formerly the state of New York controlled the market, Michigan has superseded it during the last ten years, and produces at present at least four to five times as much. On the other hand the New Yorkoil

is preferred as its quality is said to be much better than Michigan.

The peppermint industry in Japan had its seat in the vicinity of Yonezawa, but since a few years, has also started in the province Bingo Bitchu, and recently also on the northern most island Hokkaido (Yezzo). (194)

In England the peppermint culture and oil distillation have their seat in Surrey, Hertfordshire, and Lincolnshire, in the vicinity of the towns Mitcham, Maddon, West Croyden, Wallington, Carshalton, and Market Deeping. (194) Peppermint industry reached its height in England in 1850. (95)

In Thuringia the peppermint industry has almost decreased, but it is cultivated to a great extent in the vicinity of Leipzig.

Italy and Russia produce peppermint oil to a similiar extent. (194)

STATISTICS.

State of New York. (102)

1855. ---------- 33000 pounds.

1856.---------- 25000 pounds.

1857.---------- 15000 pounds.

Highest price per pound $ 4.00

Lowest price per pound $ 1.25.

State of Michigan. (175)

1861.---------- 15000 pounds.

1862.---------- 18000 pounds.

1863.---------- 24000 pounds.

1864.---------- 11000 pounds.

Price, 1861, $ 2.25

1862, $ 2.75

1863, $ 3.00

1864, $ 5.00

In 1880 the United States produced 70,000 pounds. (89)

America,

New York ---------- 35000kilos.

Michigan ---------- 27000 kilos.

England,

Mitcham---------- 5000 kilos.

--- ---------- 1400 kilos.

France ---------- 4600 kilos.

Germany ---------- 400 kilos.

Italy ---------- 1200 kilos.

Japan ---------- 64000 kilos.

Production in 1897. (194)

America,

W.Michigan---------- 79000 kilos.

S.Michigan---------- 55000 kilos.

N.Michigan---------- 25000 kilos.

E.Michigan----------13000 kilos.

New York---------- 37000 kilos.

Indiana ---------- 32000 kilos.

Various localities--- 10000 kilos.

Total---------- 251000 kilos.

Japan,

Uzen----------	1200 piculs.
Bingo----------	800 piculs.
Bitchu----------	150 piculs.
Bezin----------	50 piculs.
Yamate----------	7 piculs.
Aki----------	5 piculs.
Yamashire----------	5 piculs.
Slumaire----------	5 piculs.
Suruga----------	5 piculs.
Is. of Sikoku -------	5 piculs.
Total	2232 piculs.

One picul = 60.479 kilos.

Proportional production of the world. (194)

North America----------	90000 kilos.
Japan----------	70000 kilos.
England----------	9000 kilos.
France----------	3000 kilos.
Russia----------	1200 kilos.
Germany----------	800 kilos.
Italy ----------	600 kilos.
Other countries----------	400 kilos.
Total----------	175000 kilos

ADULTERATIONS.

Oil of peppermint has been found adulterated with the following substances:

alcohol (82,130,86,87,96,106,102,138,156,145,157)

cedar oil (154)

camphor oil (151,155,157,159,164)

copaiba oil (135,157,158)

coniferous oils (153)

fatty oils (86,87,106,138)

fireweed oil (90,92, 102, 135,155,)

ginger oil (86)

hemlock oil (91, 101)

lard (96)

mustard oil (86)

menthol abstraction (106,155,164)

oil of mentha arvensis (95)

pennyroyal oil (151,155,157,161)

cheaper peppermint oil (82,86)

sassafras oil (145)

spearmint oil (60)

turpentine oil (82,86,91,92,94,96,101,102,131,133,134,151, 158,159,160)

TESTS.

ALCOHOL has been named as having been freely used as an adulterant, but the writer does not believe it to be so used to any considerable extent, though it is always a possible addition. Magenta makes a reliable test for its presence. This aniline product is insolube in oil of peppermint, free from alcohol and moisture, though most samples of the commercial oil will dissolve it in a small proportion. In examining twenty samples of the oil only one was found not to dissolve the dye at all, and two others took it up in very small proportion, the rest on warming dissolved noticeable quantities. If alcohol were added at all it would scarcely be in less than five per-cent quantities, and this amount is easily detected by shaking the oil with a few grains of the reagent for fifteen or twenty seconds; use no heat. With five percent or more of alcohol the oil will be colored a distinct red. Light tints may be disregarded, and are probably due to the presence of small amounts of moisture. One or two trials on known material will quickly teach the analyst how much color is permissible, and render the detection of any important quantity of alcohol an absolute certainty. In the examination of eighteen samples of oil only two were found to contain alcohol, and even these two contained

it in small amounts, probably added in concealing some other adulterant. (157)

ALCOHOL may be detected by agitation with an equal volume of water, when a milky emulsion is produced. (87)

ALCOHOL can be detected in a solution by means of a mixture of one part potassium dichromate in ten parts nitric acid, specific gravity 1.30. Alcohol if present, is at once betrayed by the pungent odor of ethyl nitrite. (184)

Oil of peppermint, when heated on a water-bath in a flask provided with a well-cooled condenser, should not yield a distillate having the characteristics of alcohol. (192)

CAMPHOR OIL may be detected by adding to the suspected peppermint oil five cc of nitric acid to one drop of the oil, and the mixture gently agitated and allowed to stand for about three hours. If it has a yellow color the original oil was pure but if it has a bright red color it has been adulterated. (192)

CAMPHOR OIL may be detected according to the following test. Place a drachm of nitric acid, specific gravity 1.42 in a test-tube, and add one or two drops of the suspected oil, and after gentle agitation set aside for a few minutes. The mixture will be of a yellow color, and if pure will remain so. If as low as five percent of the oil be present the mixture will

turn red in fifteen or twenty minutes. A perfectly good quality of the oil will change color very little on this treatment, and assumes a light straw-yellow color, which becomes lighter if anything, on standing; but on applying this test to commercial oils it will be found very often that they will assume a reddish-brown color. This is not to be mistaken for the color produced by oil of camphor, The reddish-brown color fades on standing two or three hours, but the oil of camphor, a distinct red, will remain for thirty or forty hours and perhaps even longer. The reddish-brown color often obscures for a time the test for oil of camphor, but on allowing the test-tube to stand over night, the brown will fade and leave a reddish color if the oil of camphor be present. This test will reveal with perfect ease as little as five percent of canphor oil. Another advantage of it is that it can be applied for the detection of oil of camphor in other oils besides peppermint which gives no color. (157)

CASTOR OIL may be detected by the characteristic stain it leaves upon the paper. (87)

CONIFEROUS OILS may be ascertained by Heppe's test, by means of nitroprusside of copper. The adulterated oil becomes lighter in color, and the copper compound retains its grayish

color; while a sample of pure oil would become yellowish-brown, while the copper-compound contained in it would become dark-green or almost black. On pouring off the oil and adding alcohol to the salt, the difference in color between the two becomes still more noticeable. It is however impossible to tell by chemical tests what kind of coniferous oil has been added, since they all yield the same reaction. (153)

COPAIBA OIL was detected by the following method. The iodine absorption as determined from a single sample was found to be 250. According to Flueckiger the boiling point of this oil is very high, 245°C. and over, and it was thought that these two properties might be of service in its detection. An oil was taken, and after determining its iodine absorption it was fractionated, retaining a fourth in the retort; the same oil was adulterated with about twenty percent of copaiba oil, and after fractionating in like manner the iodine absorption was determined for the residue. The three figures were 110, 129, 193, so that it will be seen that the oil of copaiba increases the absorptive power of the last fraction remaining in the retort, but its own absorptive power, when perfectly pure, is not sufficiently high to make it increase this power very much over the variation which may perhaps be found to occur in

the case of different samples of oil of peppermint. However, by comparing the iodine absorption of the undisturbed oil with that of the last one-fourth fraction remaining in the retort, some data will be found which will be sugestive of the probable purity or sophistication of the oil as regards copaiba. In this connection it must be mentioned that in fractionally distilling for this last purpose a small retort is to be preferred to the regular fractional flask, because the higher boiling point of the latter portions leads to the condensation, and dropping back into the flask of the oil to an extent which brings the distillation to a close. (157)

OIL OF MENTHA ARVENSIS, DETECTION. Take one drachm of chloral hydrate and c.p. sulphuric acid, adding twelve drops of alcohol; when this solution is mixed with a like quantity of pure oil of peppermint, a yellow-brown color is produced which is maintained from ten to twelve hours, and thirty-six hours later has a slight tendency to the cherry color, or intermediate between the cherry and the brown. It was noticed that when the true oil of peppermint was mixed with an equal portion of mentha arvensis, a deficiency in the intensity of the cherry color was plainly noticed. As pennyroyal shows a similar reaction, proves relation of the oils which the botany

of the plant show, and is a question of interest. (95)

MENTHOL ABSTRACTION can be detected by simply immersing the oil in a freezing mixture; if menthol has been abstracted none will crystallize out of the oil. (185,192)

PENNYROYAL OIL may be detected as follows. One drachm of chloral hydrate, one-half drachm sulphuric acid c.p., are rubbed together in a glass mortar, and alcohol added drop by drop until a clear solution resulst. In a watch glass put a few drops of the oil to be tested, and with a glass rod add an equal quantity of the test solution, and rub briskly for a moment. After standing for a few moments if there is an adulteration with pennyroyal, the mixture will assume a dirty green color, which grows darker on standing. Pure oil of peppermint assumes a cherry-red color. (87)

PENNYROYAL OIL may also be detected as follows. The only test for the oil of pennyroyal which the writer has seen is the chloral sulphuric test. It has already been pointed out that this test is unreliable, and the writer's work simply confirms that of previous investigators in this respect. Oil of peppermint alone gives it to deep a color for it to be easy to distinguish the less marked olive-green color which oil of pennyroyal gives, and it is only when the amounts become consider-

able that it is easy to note its appearance, and even then the reaction does not seem to be uniform. The writer has not search ed the commercial oils particularly for this adulterant, but causally noted its presence in two oils, one of which contained it in such quantities as to be distinctively noticeable by its odor. (157)

SASSAFRAS OIL may be detected in the same manner as oil of camphor given above. (192)

TURPENTINE OIL may be detected by the cohesion figure, which forms more slowly than that of peppermint, and breaks up less rapidly. (183)

OIL OF TURPENTINE. It is generally spoken of as a probable adulterant of essential oils, and is specifically mentioned as being added to oil of peppermint. Among other tests great stress has been laid on the rotation of polarized light, the fulmination with iodine, and the solubility in aqueous alcohol. With the specific rotary power the writer has had no experience but nevertheless is justified in warning against to implicit reliance in this test. Its positive results, when coupled with fractional distillation, may perhaps be, and doubtless are, trustworthy, but as to its negative results, it must not be forgotten that fresh oil of turpentine rotates polarized light

in the same direction as oil of peppermint, and according to a statement now before the writer, nearly as far as the lower average of peppermint oil (-35°). It is highly probable that this known fact of the difference between American and French turpentine has already taken advantage of to nullify the optical test for turpentine in oil of peppermint.

Oil of peppermint is completely soluble in its own volume of eighty percent by weight of alcohol, but on the other hand, oil of turpentine is as completely insoluble in an equal volume of alcohol of this strenght. This difference between the two oils has been recommended as a means of detecting the latter when added to the former, but whoever first announced this test must certainly have overlooked the fact that the two oils are mutual solvents, and that therefore their solubility when in mixture will be greatly different from either of the oils when separate. This is such a common occurence in solubilities that it is somewhat surprising that it should not have been taken into consideration. As a matter of fact, eighty percent alcohol is worthless as a test for the presence of turpentine in oil of peppermint. The usual test of equal volumes will overlook the presence of thirty percent, and only begins to show the presence when forty percent of turpentine has been

has been added, and increasing the amount of dilute alcohol employed, does not, as one might suppose, increase the exactness of the test. Further, when a little alcohol,is also added with the adulterant, as we have reason to suspect is sometimes done, then fifty percent of turpentine escapes notice by this test. Long before such amounts are reached the adulterant may be detected by the sense of smell. This test, it will thus be seen, is a false reliance, and is doubtless in favor with certain unscrupulous dealers in essential oils.

Distillation with chlorinated lime and water is mentioned for the purpose of directing attention to the fact that chloroform is yielded in small amounts, and may be detected by filtering the distillate through a well-wetted filter paper and applying the isonitrile test for chloroform. It was long ago recorded that turpentine yielded chloroform when thus treatdd, and in experimenting to determine whether it might be made a means of detecting turpentine in oil of peppermint it was noted that this latter oil also yielded chloroform.

The iodine absorption factor has also been used for detecting turpentine. Experience to date justifies the statement that an oil yielding, from the fraction of onexdrachm from one ounce less than 125 forits iodine absorption is not very likely to

to be adulterated with turpentine. Only once thus far has the writer found an oil having a higher absorption in the original than this, and as already stated that oil is still open to question. If an oil exceeds 125 it is a suspect and running over 185 may be discarded. With much turpentine present the fraction will have an absorptive power over 225. After careful fractional distillation the turpentine may often be detected by its odor. The absence of alcohol should first be proven. (157)

PROPERTIES.

Properties are divided into three classes:

a. chemical

b. physical

c. medicinal

Chemical Properties.

Action of chemicals.

ACETIC ACID. (142)

Twenty parts acetic acid and shaken with one part of peppermint oil; the oil will acquire a faint blue color, in the course of one-half hour, and this becomes gradually more intense.

ACETIC ACID. (192)

If five drops of the oil be added to one cc. of glacial acetic acid, and the mixture gently warmed, the liquid will assume a blue color, with a red flourescence.

ACETIC ACID AND DILUTE ALCOHOL. (147)

A mixture of one cc. glacial acetic acid and one drop of oil of peppermint, slightly warmed, shows the color very beautifully, it being blue in transmitted, and blood-red in reflected light, and after diluting with alcohol until the blue tint has nearly disappeared, the red reflection is still observed in the sunlight on pouring the liquid out in a thin stream, and looking vertically into it. Menthol and oil of crisped mint do not show the reaction.

ACETIC ACID AND NITRIC ACID. (192)

If two cc. of the oil be mixed with one cc. of glacial acetic acid, and one drop of nitric acid added, the liquid will soon acquire a green, greenish-blue, blue, or violet tint with a copper red flourescence.

GLACIAL ACETIC ACID AND NITRIC ACID. (18)

Ten cc. of the oil, five cc. of acetic acid, and five cc. of nitric acid will show two bands, one in the red, and the other in the orange part of the spectrum. After five minutes

a third band is found in the green, and after twenty minutes they unite. The mixture viewed by transmitted light is green. After five minutes standing a blood-red flourescence is visible after minutes the color by transmitted light has changed to a pure blue.

ALCOHOL AND DILUTE HCL. (143)

The mixture with the oil became turbid, then red, violet, and finally deep blue.

ALCOHOL AND CONCENTRATED HCL. (143)

The mixture became deep olive green and finally of a deep violet color.

ALCOHOL AND SALICYLIC ACID. (143,147)

An alcoholic solution of the oil, upon the addition of salicylic acid gradually acquires a copper-green color. On adding the solution to salicylic acid (melted) a blue-green color is at once produced, soluble in alcohol.

BROMINE. (127)

Bromine colors the oil a lemon yellow color.

BROWN TEST PAPER. (132)

Brown test paper is changed by oil of peppermint.

CHLORAL HYDRATE. (140)

The rose coloration which takes place in shaking chloral.....

hydrate with peppermint oil has been investigated by the author. He has come to the conclusion that the color is produced in the oil of peppermint and not in the chloral hydrate, and that it only occurs when the chloral hydrate used is acid, it being more intense in proportion as the chloral hydrate is more acid. But he has not yet been able to experiment with perfectly neutral specimens. In this case the reaction would be due to the formic acid contained in the chloral hydrate, or possibly to hydrochloric acid resulting from partial decomposition.

CHLORAL HYDRATE. (139)

The mixture of chloral hydrate and peppermint oil changes from a brown to a red color, and soon becomes a dark red color, which is dissolved by ether, alcohol, and chloroform. By boiling the color is not changed. Addition of sulphuric acid intensifies it. If chloroform is added after the addition of the sulphuric acid, it takes on a violet color.

CHLORAL HYDRATE. (144)

The following reaction took place between seven samples of oil and chloral hydrate:

	5 min.	1 hour.	heated.
1.	none	l.violet	slight red.
2.	none	none	none.
3.	l.violet	green	green.
4.	yell.-brown	yell.-brown	yell.-brown.
5.	none	none	yell.-brown.
6.	none	none	none.
7.	none	red-brown	green.

The great difference produced in color, as seen above, indicates that some of the oils must have been adulterated.

CHLOROFORM AND BROMINE. (143)

Ten to fifteen drops were mixed with one drop of peppermint oil. The oil was colored splendidly violet, then cherry-red, then discolored.

CHROMIC ACID. (133)

Produces no reaction.

COPPER NITROPRUSSIDE. (133)

Produces a green color.

FERRIC CHLORIDE AND CONCENTRATED SULPHURIC ACID. (146)

The color produced is red-brown, then dark-red brown, and finally black-brown.

FERRIC CHLORIDE? CHLOROFORM? AND CONC. SULPHURIC ACID.(146)

Color is produced with peppermint oil which is drak red brown and finally blood red.

IODINE. (126,~~143~~ 134)

Iodine dissolves in peppermint oil without any heat being evolved.

IODINE. (133)

Iodine fumes in contact with oil of peppermint.

The above statement is refuted. (192)

SUGAR? HCL., AND ALCOHOL. (192)

If one cc. of the oil be dissolved in five cc. of alcohol and 0.5 gms. of sugar and one cc. of HCL be added, and the mixture gently heated, a deep blue or violet color will gradually be produced.

HYDROCHLORIC ACID. (140)

Hydrochloric acid induced a rose color rather slowly. Upon the addition of ether this became faintly green. When water was added the underlayer was rose colored, but the ether retained its green color. In some experiments a blue color was produced.

NITRIC ACID. (140)

Nitric acid caused first a rose coloration, then red, soon

becoming greenish. Upon adding ether and water and shaking, the underlayer was rose, and the ether rising to the top, took a violet-blue gray color. The blue and green tints were rapidly altered by the action of air and light. These observations were made upon pure and quite colorless peppermint oil. When the yellow or greenish-yellow tinted oils, frequently met with in commerce were employed, the phenomena of coloration were much more intense with sulphuric and hydrochloric acids; whilst with nitric acid the ethereal layer acquired a magnificent green color and a strong red flourescence. The rose coloration first produced by the acids had a violet reflection. Moreover, when chloroform was employed in the place of ether, sometimes a violet or gray color was obtained, the latter being the result of a mixture of yellow and violet, or perhaps red and green, or even blue and orange.

Flueckiger observed that peppermint oil acquires a blue-green color with nitric acid, specific gravity 1.2. (147)

Fifty to seventy drops of peppermint shaken with one drop of nitric acid, sp.gr. 1.2 turns faintly yellow, brownish, and after an hour or two exhibits a most beautiful blue violet, or greensih-blue color, when examined in transmitted light, and when observed in reflected light, the liquid is of a copper color and not transparant. If the misture is warmed the gree

or blue coloration takes place rapidly. It may also be hastened by adding a greater amount of nitric acid.

LITMUS PAPER. (29, 192)

Litmus paper has no reaction, or rather is not colored by peppermint oil.

PICRIC ACID. (140)

If oil of peppermint be agitated with picric acid, nothing is observed at first further than that the picric acid dissolves, and communicates its yellow color to the oil; but in half an hour the mixture is colored manifestly green, and in twenty-fours hours this coloration acquires a great intensity. If a slight heat be applied to the mixture the green color appears more rapidly.

This green product, exposed to the air upon water during four or five days, acquires a reddish-yellow color of dead leaves. Introduced floating on water, into a test-tube containing nitrogen, the color is retained for some time; but in oxygen it disapears more quickly. Treated several times with cold water, the washings remove each time some of the picric acid together with a red coloring matter, and finally the essential oil remains of a reddish-yellow color.

The green product has a strong red flourescence, and in an alcoholic or ethreeal solution, this phenomenon is still

more marked. Treated with solution of caustic potash or ammonia a picrate of the base employed is formed, and the essential oil remains of a reddish-yellow color. When the green product is distilled from caustic potash, a colorless liquid passes over into the receiver, and there remains in the retort a black mass analogous to that obtained by treating oil of peppermint with chromate of potash. The distillate no longer yields the green reaction with picric acid. Nascent hydrogen reduces the green product and transforms it into a brown substance. If, instead of operating in the cold or with a gentle heat, the solution of picric acid in oil of peppermint is boiled for a few moments, it passes from the green state to a yellow-brown, and then to a reddish-brown. Upon the addition of ammonia it quickly forms red crystals, which are probably picramate of ammonia, and some crystals of picrate, whilst in the midst of them is disseminated an amorphous powder of a beautiful red color. This red powder is soluble in water, insoluble in benzin, and oil of turpentine, and very slightly soluble in ether and alcohol.

The reaction between picric acid and oil of peppermint is so clear, that the author thought that picric acid might be used in testing for the presence of peppermint in a mixture of essential oils, and vica versa that oil of peppermint would be a suitable test for the presence of picric acid. For this

purpose he made a mixture of several essences, about two grams to which he added two drops of oil of peppermint. This was shaken with a solution of ten centigrams of picric acid in about fifty grams of water. At the end of twenty-four hours the oils collected on the surface, and presented a very perceptible green tint.

SULPHURIC ACID. (140)

Sulphuric acid produced at first a rose color, then reddish-yellow, passing rapidly to reddish-brown. When the ether was added it acquired a beautiful yellow color, whilst the lower portion of the mixture was colored red. When water was added and the mixture shaken, the mixture separated into two layers of which the lower aqueous layer was rose color, and the uppermost layer took a greenish-blue tint and had a strong red flourescence.

CONSTITUTENTS.

Power and Kleber.	(31)
Halsey.	(169,170)
acetaldehyde	$C H_3 COH$
isevaleraldehyde	$(CH_3)_2CH. CH_2 . COH.$
acetic acid	$CH_3 COOH$
isevalerianic acid	$(CH_3)_2 CH. CH_2 .COOH$
pinene	$C_{10}H_{16}$
phellandrene	$C_{10}H_{16}$
cineol	$C_{10}H_{18}O$
l-limonene	$C_{10}H_{16}$
menthone	$C_{10}H_{18}O$
menthol	$C_{10}H_{20}O$
menthyl acetate	$C_{10}H_{19}O. C_2H_3O$
menthyl isovalerianate	$C_{10}H_{19}O. C_5H_9O$
menthyl esters of	$C_8H_{12}O_2$, $C_{10}H_{19}O$, $C_8H_{11}O$.
lactone	$C_{10}H_{16}O$
cadinene	$C_{15}H_{24}$
anyl alcohol	$C_5H_{12}O$
dimethyl sulphide	$S(CH_3)_2$

PHYSICAL PROPERTIES.

Angle of Polarization.

American Oils:		
Michigan	-18° to -29°	(194)
St.Joseph Co. 1893.	-9°45'	(31)
St.Joseph Co. 1893.	-19°30'	(31)
Wayne Co., N.Y.	-32°	(31)
Wayne Co., N.Y. 1892.	-26°45'	(31)
Wayne Co., N.Y. 1893.	-32°30'	(31)
F.S. & Co.	-32°45'	(31)
V.B.C.M. 1893.	-29°20'	(31)
W.C.M. 1893.	-28°30'	(31)
Mississippi	-13°40'	(31)
Other Amer. Oils	-25° to -33°	(194)
	-24.5° to -32.3°	(90)
	-64°	(164)
	-66°	(159)
	-30.2°	(38)
	-29°	(34)
Fritzsche Bros.	-21°40'	(196)
Bohemian Oil	-27°22'	(194)
English Oils	-23.5°	(166)

		-23.25°	(166)
		-22° to -33°	(194)
		-21°23'	(166)
		-27°70'	(38)
		-23°35'	(31)
		-23°55'	(31)
	redistilled	-23°30'	(31)
		-27°55'	(31)
	Black Oil	-25.5°	(33)
	White Oil	-33°	(33,34)
	Black Oil	-18.5°	(34)
French Oils,			
Buignet		-14.30° to -34.29°	(86)
Other French Oils		-5.54° to -8.20°	(31,194)
	fresh leaves	-30°	(31)
	dry leaves	-32°	(31)
German Oils,			
Lubolt	fresh leaves	-10.5°	(166)
	dry herbs	-20°	(166)
Other German Oils		-24.59°	(38)
Italian Oils		-13° to -18°	(194)

Japanese Oils		-30° to -42°	(194)
	normal	-34°45'	(31)
		-31°20'	(31)
(Soleil-Ventzke polariscope)		-105° to -106°	(150)
Reunion		-6°9'	(194)
Russian Oils		-17° to -22°	(194)
	fresh leaves	-17.13°	(38)
	dry herbs	-18.26°	(38)
Saxon Oils		-25° to -33°	(194)
		-26°	(31)
		-26.25°	(34)

Stahre gives the following angle for twenty-two samples: (168)

-20°18'	-28°36'
-22°50'	-30°11'
-25°37'	-30°26'
-24°	-30°35'
-24°36'	-30°36'
-25°40'	-30°40'
-26°38'	-30°40'
-27°4'	-31°2'

-27°19'	-31°5'
-27°48'	-33°11'
-28°26'	-33°11'

The great differences which can readily be noticed in the above tabulation, are only to be accounted for by the use of polariscope tubes of different lenghts; by adulterations, or possibly by the oil being obtained from plants gathered at various times during the peppermint season.

BOILING POINT.

Andres & Andreff	160 - 220°C. 160 - 232°C. 165 - 225°C. 165 - 225°C. 175 - 233°C.	(38)
Moss	210 - 218°C. 339 - 402°F.	(79) (171)
duffield	414°F.	(159)
Todd	363 - 401.5°F.	(95)

COLOR AND ODOR.

Color changes on long standing, becoming gradually thicker and darker. It changes from a light color to a dark-brown color (102, 131, 177). The color changes also on account of oxidation, but mostly from some adulteration. (102,123,133,130)

Color of oil of plants distilled, lighter than color of oil distilled without steam. (85)

The English black oil of peppermint is lighter than the white oil. (33)

One oil was colored pale greensih-yellow, it was found however to be adulterated with alcohol. (34)

The anhydrous oil has a better odor than the ordinary. (179)

Stahre examined twenty-two samples and found color ranging from pale yellow to brown. (168)

Oils are colored from the rust of boilers and pipes and from other causes. (103)

One oil obtained from Fritzsche Bros. was of a pale yellow color, while one from Lehn & Fink was nearly colorless. (196)

INDEX OF REFRACTION.

Stahre obtained the following indexes from twenty-two samples. (168)

1.458	1.463
1.458	1.463
1.458	1.463
1.459	1.465
1.461	1.465
1.462	1.465
1.462	1.466
1.462	1.467
1.462	1.467
1.462	1.478
1.462	1.479

SOLUBILITY.

German oil soluble in 78% alcohol and in 95% alcohol. American oil the same. (127)

Soluble in 5-6 parts of alcohol. (131)

Soluble in alcohol of all proportions. (95)

This statement is refuted by the following statement which says that 70% alcohol did not yield a clear mixture. (168)

Soluble in two parts of alcohol of Sp. gr. 0.85 (150)

Dissolves four parts menthol. (8)

Iodine Absorption. (173)

English Oils absorb	51.2%
	49.6%
	57.7%
Mitcham Oil	81.9%
American Oil	121.8%
	132.2%
	143.9%
Japanese Oil	43.9%

It was found by experimentation in the labratory on allowing ten grams of tartaric acid to dissolve in fifteen grams of Fritzsche Bros. peppermint oil, and then after two or three days, filtering the solution, and washing the crystals of

tartaric acid with benzol and then weighing the crystals, that the oil had dissolved 1.28% in one case, and 1.27% inthe other case. (196)

RECTIFICATION.

To overcome and leave behind adulterations, the oil should be distilled. It is however very hard to dispose of some adulterants in this way, as their boiling points may be near that of peppermint oil. (86, 103.)

By rectification John Umney obtained the following percentages of oil. (33)

Temperature.	B. Mint.	W. Mint.
---- 200°C.	5%	24%
200 - 205°C.	27%	15%
205 - 210°C.	31%	15%
210 - 215°C.	22%	15%
215 - 220°C.	7%	13%
220°C. ----	8%	18%

Power and Kleber obtained (31)

---- 200°C.	2.6%
200 - 205°C.	2.4%
205 - 210°C.	8.6%
210 - 215°C.	18.8%
215 - 220°C.	24%
220 - 225°C.	19.6%
225 - 230°C.	9%
230 - 235°C.	3.6%
Residue	12.2%

SPECIFIC GRAVITY.

American Oil:

	0.84	(127)
	0.888	(188)
	0.855 - 0.859	(131)
	0.870	(133)
	0.855	(116(
rectified	0.844 - 0.880	(131)
	0.9024	(129)
rectified	0.9098	(129)
	0.915	(30)
	0.9074	(187)
	0.9112	(187)
	0.894	(87)
	0.916	(162)
	0.905 - 0.920	(189)
rectified	0.902	(102)
	0.916	(95)
	0.910 - 0.920	(182,194)
f. leaves	0.9130	(31)
dry herbs	0.914	(31)
	0.912	(38)

Wayne Co. N.Y.	0.958	(31)
Wayne Co. N.Y.	0.9110	(31)
F. S. & Co.	0.9110	(31)
V.B.C.M.	0.9067	(31)
V.C.M.	0.9135	(31)
St.Joseph Co. Michigan	0.9135	(31)
St.Joseph Co. Michigan	0.9083	(31)
Mississippi	0.9250	(31)
English Oil	0.88	(188)
	0.910 - 0.920	(131)
	0.9107	(171)
	0.9085	(95)
	0.9088	(95)
	0.901	(38)
	0.905	(31)
	0.907	(31)
	0.900 - 0.910	(194)
	1.20	(26)
rectified	0.9117	(171)
rectified	0.859	(115)
rectified	0.856	(115)
rectified	0.9105	(31)

	white	0.9058	(33,34)
	black	0.9036	(33)
	black	0.9072	(34)
French		0.906 - 0.914	(188)
		0.900 - 0.930	(86)
		0.920	(182)
Bohemian		0.905	(194)
Chilian		0.916	(194)
German		0.860	(127)
		0.895	(178)
		0.895 - 0.920	(182)
		0.908 - 0.912	(188)
		0.902 - 0.920	(188)
		0.909	(188)
		0.910 - 0.926	(85)
		0.9013	(129)
		0.90	(130)
		0.910	(31, 38)
		0.9057	(34)
		0.930	(194)
Italian		0.911 - 0.926	(194)
		0.915 - 0.921	(182)

Japanese		0.895 - 0.905	(194)
		0.890 - 0.910	(182)
		0.850	(150)
		0.910	(31)
		0.899 - 0.902	(189)
Russian		0.905 - 0.910	(194)
		0.908	(182)
		0.915	(18)
	f.leaves	0.915	(38)
	dry herbs	0.912	(38)
Reunion		0.887	(194)

Stahre gives the following for twenty-two samples. (168)

From 0.9045 - 0.913.

Brandes & Reich obtained 0.955 as the Sp.Gr. by using an accurate araemeter for some oils, and a Sp.Gr. bottle for others. (186)

The specific gravity of oil obtained bys steam distillation is lighter than that obtained by simple distillation. (85)

Dry peppermint plants yield two oils of different specific gravity. (85)

The great differences in the above specific gravities may

be caused by the manner of distillation or by rectification; but in all probability some adulterant was present.

MENTHOL.

SYNONYMY.

Menthol is synonymous with and has been variously termed:

Hakka-no-sei (in Japan)	(2)
Menthol	(7,80)
Peppermint Camphor	(3,4,6)
Peppermint Stearescence	(3,4)
Peppermint Stearoptene	(3,4)
Pip-menthol	(1)

HISTORY.

Menthol was known to the Japanese 200 years ago. The gentlemen wore it in a medicine box suspended from their waist by means of a girdle, in which menthol is found to have been one of the constitutents, of a certain compound called "Hotam". (2)

Menthol was first observed and investigated by chemists in 1771, in oil of peppermint by Flueckiger and Hanbury. (5)

As early as 1829 menthol was investigated by chemists such as Dumas, Walter, Blanchette, and Sell. (1,2,3,4,5)

In 1861 and 62 menthol was obtained in coarse earthen jars from Japan, protected simply by paper covers, and consisting of

a mass of small fragrant prismatic crystals resembling magnesium sulphate. (3,4)

Oppenheim recognized the alcoholic character of menthol in 1861. (5)

In 1864 John Mackay notes the arrival in London of peppermint oil, which became solid when cold. (2)

In 1874 John Moss made some investigations, followed in 1876 by Beckett and Wright. (1,2,3,4,5)

Since then menthol has steadily gained in importance, especially on account of its medicinal properties.

In 1879 Cristy of Lomdon forwarded to Parke, Davis & Co. an important consignment of menthol, soon followed by other shipments. Earl in 1880 Fritzsche Bros. of New York received a consignment. (2)

Dundas Dick in 1881 introduced the now famous menthol cone which has been sucessful in this country. (2,3)

Moriya oxidized menthol $C_{10}H_{20}O$ to $C_{10}H_{18}O$ in 1881. (5)

Atkinson and Yoshida prepared the same substance in 1882. (5)

The formula of menthol is now generally recognized as $C_{10}H_{20}O$. (2)

STATISTICS.

The consumption of menthol has steadily increased, as can be seen by the following figures:

4000 pounds in 1884

5000 pounds in 1885

6000 pounds in 1886

with a proportionate increase in later years. (61)

Although the demand increased greatly in those years, the plantations of Japan had been planted with an inferior grade of peppermint plants, which yielded under unfavorable conditions an oil having a turpentiny and camphoraceous smell. (62)

Menthol was worth about $ 5.50 in 1887. (63)

ADULTERATIONS.

Menthol has been found adulterated with the following substances:

Borneol	(13)
Magnesium Sulphate 10% - 20% (refuted by J.Mackay.)	(7) (8)
Oily matter	(9)
Paraffin	(12, 80)
Salicin	(10)
Salicylic acid	(13)

Phenol (13)

Thymol (11,13,80)

TESTS.

Eyckmann gives the following test for thymol adulteration Dissolve a little of the suspected menthol in one cc. of glacial acetic acid, and add five to six drops of concentrated sulphuric acid. On now adding one drop of nitric acid a fine blue color will make its appearance, (if thymol is present) in lower part of test-tube, which will spread throughout the contents of the latter on shaking. If only a small amount of thymol is present the color will be dichroic red by transmitted, and blue by reflected light. (13, 80)

If phenol is present, a pure violet color only is produced. Salicylic acid, camphol, and borneol yield no color reaction under these circumstances. (13)

Bernbeck also notes the presence of thymol and tests for it according to the following methods:

1. Suspected menthol is mixed with four times its weight of sulphuric acid, and if it contains thymol, a deep coloration is produced upon warming.

2. If the solution is mixed with ten times its weight of water, and digested with an excess of lead oxide, the filtrate

gives a fine violet color on the addition of some drops of ferric chloride solution.

3. A solution of the suspected substance in chloroform and alcohol give a reddish-violet color when heated with caustic potash solution. (11)

Bedford tests for paraffin simply by comaprision of melting points, that of paraffin being much higher. (12)

A.B. Lyons gives a method for the detection of salicin, by means of its solubility in water, its bitterness, and its red color when touched with strong sulphuric acid. (10)

Atkinson and Yoshida note that menthol is usually contaminated with oily matters which accompany it in the plant. (9)

More Menthol Tests.

a. Menthol should be completely and readily soluble in twice its weight of chloroform. On the addition of a small quantity of iodine to the solution, a rich indigo blue color (not dark green) ought to be imparted, and this color should be completely discharged on shaking the mixture with a solution of caustic potash or soda.

b. The melting point ought to be 42.2°C. (108°F.) and the boiling point not lower than 212°C. (413.6°F.) and there should be not residue upon evaporation.

c. Menthol shaken with an oxidizing agent such as sulphuric acid and potassium dichromate, ought to be entirely inverted after prolonged heating into a dark-green flocculous substance (menthene).

d. Strong sulphuric acid ought not (in the cold) to blacken menthol to any extent.

e. When menthol is heated with a small quantity of zinc chloride, the mixture ought to give off the odor of menthene.

f. Strong caustic potash has no action on pure menthol.(16)

Melting point from 40 to 45°C., insoluble in water, soluble in alcohol, ether, chloroform, and acetic acid anhydride. Completely volatilized on a water-bath. To detect presence of thymol a mixture is prepared of one cc. of acetic acid anhydride three drops of sulphuric acid, and one drop of nitric acid. A crystal dissolved in this mixture, if pure menthol, the solution remains colorless; if it contains thymol, it assumes a yellowand afterward an emerald green color. (59)

PREPARATION.

Menthol obtained from oil of peppermint by cooling the the oil. American oil of peppermint yields it at zero degrees centigrade. (42)

Menthol obtained by cooling oil of peppermint to 15°C. (2)

Menthol obtained by freezing the oil. (6)

Peppermint oil subjected to low temperatures at which menthol crystallizes out. The mother liquid is removed by the aid of centrifugal machines, and the menthol recrystallized. (5)

Mix thoroughly about one pint of snow finely crushed with a like quantity of finely powdered salt, and put this in any convenient jar or recepticle which will hold it; in a test tube place the oil of peppermint nearly filling the tube, then cork it and immerse it in the freezing mixture. In ten to fifteen minutes, the oil if pure, will have become cloudy, translucent, thick or of a jelly like consistency, then add four or five crystals of pure menthol, recork and shake thoroughly, and at once replace the tube in the freezing mixture. After a few minutes the oil if pure will present the appearance of a solid frozen mass of crystals. (64)

OCCURRENCE.

Schrenk found menthol in the glandular hairs of mentha piperita in great quantities. They are there singly, but mostly in large conglomerations. The crystals are doubly refractive and can be seen very readily with a polariscope.

It is remarkable how long these crystals remain in the dry leaves. Fragments from an herbarium specimen gathered in 1827, contained them in as perfect condition as leaves of plants collected quite recently.

The tops, the bracts and calyx of mentha piperita are beset with numerous glands containing oil and menthol crystals.

Of other species examined (rotundifolia, aquatica, arvensis sativa, canadensis) the only one which exhibited menthol crystals was Mentha aquatica L., both the type and variety crispa. A specimen of the former gathered in Germany about 1840, gave the same results as the leaves of its variety collected on Long Island collected in 1878, and those of an interesting hybrid of mentha piperita × aquatica, obtained from C. Mueller, and W. Retzdorff of Berlin Agricultural Institute. (60)

Menthol occurs in mentha piperita, and in mentha arvensis var. piperascens, and in var. glabrata. (5, 80)

ESTIMATION.

The following method of estimation is given by Power and Kleber:

Twenty grams of the peppermint oil are heated to boiling with twenty cc. of normal NAOH alcoholic solution for about an hour, in a flask provided with a reflux condenser, in order to decompose the menthol esters. After cooling, the unconsumed alkalie is titrated back with normal sulphuric acid, using phenolphthalein as an indicator. The saponified oil is washed repeatedly with water, and then heated with an equal volume of glacial acetic acid and two grams of anhydrous sodium acetate, in a flask provided with a glass ground tube condenser. After cooling, the oil is washed several times with water and dilute soda solution, dried with calcium chloridexand filtered. Eight to ten grams of this oil are then saponified, as described above, with fifty cc. of normal alcoholic NAOH solution and the excess of alkalie titrated back.

Each cc. of the normal sodium solution corresponds to 0.156 of the menthol, or 0.198 of menthyl acetate. In order therefore, to obtain the percentage of menthol in the original oil(not acetylized but free from ester) it is necessary to deduct 0.042 grams (the difference between 0.156 and 0.198) for

every cc. of normal alkalie consumed. If e.g. s grams of acetyliced oil requires a cc. of normal soda solution, the total menthol content P (free and ester) can be calculated according to the following formula:

$$P = \frac{a \times 15.6}{s - (a \times 0.042)}$$

The result thus obtained does not express exactly the menthol content, in as much as it is assumed for purposes of calculation that all the menthol is present as acetic ester, whereas some of it is combined with isovalerianic acid. The resultin error, however, is so small that it can be disregarded. (31)

Umney obtained the percentage of menthol by saponification, and by the acetylization method. (33,34)

Kleber C. gives the following method for the quickest determination:

About five grams of peppermint oil,are mixed in a flask with a glass ground condensing tube, withnabout five cc of acetic anhydride accurately measured, and boiled for thirty minutes. In the mean time an equal quantity of the same acetic anhydride is titrated with normal NAOH and phenolphthalein. After cooling the boiling liquid somewhat, the condenser is

taken off and washed with water, which is added to the acetylized mixture, and the latter is titrated with normal NAOH. The difference in the number of ccs in both titrations multiplied by 0.156 gives the menthol in oil used. (35)

L. Kleber gives a slight modification to the method used by Power and Kleber. In this method he uses fresh oil for the estimation of the free menthol, and another amount for the combined menthol. This estimation can be made in three hours. (36)

F. Sieker makes the estimation by using Power and Kleber's method for the combined menthol, and Kleber's shorter method for the total menthol. (75)

PROPERTIES.

This heading can be divided into three sub-heads:

a. chemical

b. physical

c. medicinal

CHEMICAL PROPERTIES.

Action toward chemicals.

acetic acid bromine chloroform	(21)
bromine	(20)
camphor	(19)
chloral	(19,78)
hydrochloric acid	(17)
formaldehyde sulphuric acid	(23,74)
iodine	(17)
nitric acid	(19,20,21)
potassium dichromate sulphuric acid	(19)
potassium hydroxide (strong)	(16)
sulphuric acid	(20, 21)
sulphuric acid potassium dichromate	(19, 23)
Thymol	(19)

Bromine dissolves in acetic acid and chloroform forms a compound of the formula $C_{10}H_{20}Br$. (21)

Bromine acts violently on menthol. (20)

Camphor and chloral liquify menthol. (19)

Hydrochloric acid and iodine have no action on menthol. (17)

A brownish-red liquid is produced with 120 drops of sulphuric acid, becoming green upon adding a little potassium dichromate. (19)

Menthol heated with nitric acid becomes thick and wine colored. (19)

Menthol is decomposed upon heating with nitric acid. (20)

Menthol heated in presence of HNO_3, an explosive liquid is formed $C_{10}H_{19}NO_2$. Heated a long time with twenty volumes of HNO_3, a product is formed of the formula $(C_5H_8O_4).H_2O$. (21)

Brochet obtained the following compound by treating menthol with formaldehyde in presence of sulphuric or hydrochloric acid. $CH_2(OC_{10}H_{19})_2$. (74)

Strong KOH has no action. (16)

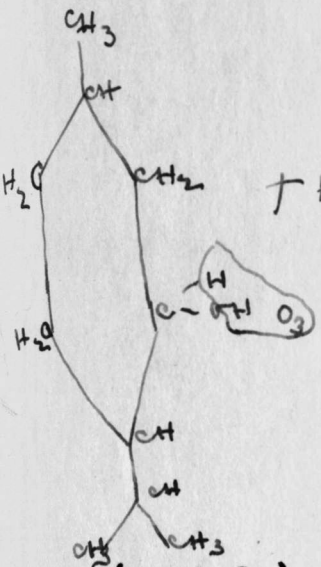
Menthol not affected by any alkalies. (20)

Menthol changed into a dark-brown liquid with H_2SO_4. (20)

Sulphuric acid transforms menthol into dimenthene. ($C_{10}H_{18}$) (21)

Thymol liquifies menthol. (19)

The following reaction takes place when menthol is oxidized. (23)



(Menthol.)

(Menthone.)

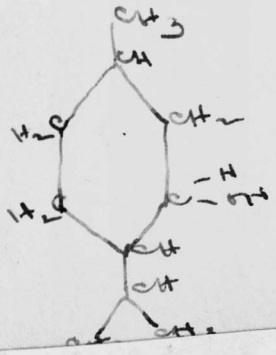
Formula.

Andres. $C_{10}H_{20}O$. (18)

Andres & Andreef. $C_{10}H_{20}O$. (38)

Atkinson. $C_{10}H_{20}O$. (9)

Beckett & Wright. $C_{10}H_{20}O$. (31)



Beckmann. $C_{10}H_{20}O$. (72)

Berkenheim. $C_{10}H_{19}OH.$ (24)

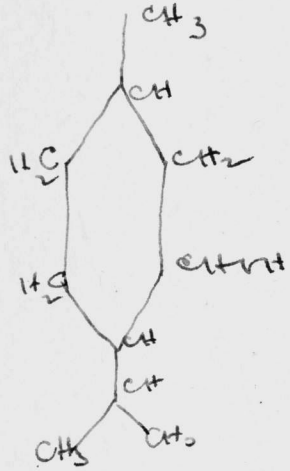
Bruehl. $C_{10}H_{20}O.$ (23)

Dumas. $C_{10}H_{20}O.$ (8)

Hanbury. $C_{10}H_{20}O.$ (8)

Juenger & Klager. $C_{10}H_{20}O.$ (73)

Juenger & Klager

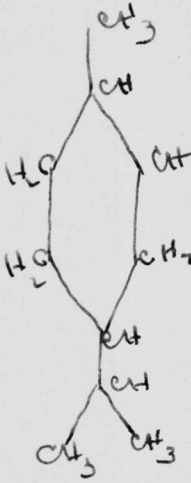


Kremers & Urban. (same as above) $C_{10}H_{20}O.$ (25)

Langgaard. $C_{10}H_{20}O.$ (6)

Mackay. $C_{10}H_{18} + H_2O.$ (8)

Oppenheim. $C_{10}H_{20}O.$ (31)



Richetr. $C_{10}H_{20}O.$ (71)

Roscoe & Schorlemmer. (Incorrect.) $C_{10}H_{20}O.$ (70)

Trimble. $C_{10}H_{20}O.$ (1)

Waldie. $C_{10}H_{20}O.$ (2)

Walter. $C_{10}H_{20}O.$ (15)

PHYSICAL PROPERTIES.

Boiling Point.

Andres.	211°C.	(18)
Arth. (735 mm. pressure)	211.5°C.	(67)
Atkinson & Yoshida.	212°C.	(9)
Beckett & Wright.	212°C.	(1)
Dumas.	208°C.	(7)
Gladstone. (menthol from spearmint)	22[illegible]°C.	(7)
Kremers.	212°C.	(5)
Langgaard.	212°C.	(6)
Long. (742 mm. pressure)	212.5°C.	(68)
Mackay.	210°C.	(8)
Moss. (began at 210°C.)	215°C.	(7, 79)
Oppenheim.	210°C.	(7)
Power & Kleber. (758 mm. pressure)	215.5°C.	(31)
Twirchell.	210-215°C.	(17)
Waldie.	212°C.	(16)
Walter.	213.5°C.	(15)
U.S.Pharmacopoeia. (413.6°F.)	212°C.	(80)

Melting Point.

Atkinson.	42.2°C.	(9)
Beckett & Wright.	42°C.	(37)
Bedford. (100°F. 37.8°C.)	37.8°C.	(12)
Dumas.	25°C.	(7)
Kremers.	42.2°C.	(5)
Langgaard.	43°C.	(6)
Laval. (Pipmenthol)	40°C.	(20)
Laval. (two samples Japanese.)	35-36.5°C.	(20)
Lyons. (107-110°F.)	41.7-43.3°C.	(10)
Mackay. (100°F.)	37.8°C.	(8)
Moriya.	35-37°C.	(14)
Moss.	39°C.	(7, 79)
Oppenheim.	36°C.	(7)
Schrenck.	40°C.	(60)
Trimble. (Japanese.)	41°C.	(1)
Trimble. (Pipmenthol.)	42°C.	(1)
Twitchell.	36-42°C.	(17)
U.S.Pharmacopoeia.	43°C.	(80)
Waldie. (108°F.)	42.2°C.	(2,16)
Walter.	34°C.	(15)

Angle of Polarization.

Atkinson & Woshida. (Menthol was obtained from menthone.)	39°	(9)
Backmann.(In 10% alcoholic solution.)	-57.7°	(69)
Beckmann.(In 20% alcoholic solution.)	-49.3°	(69)
Long.	-49.86°	(68)
Kremers.	-48.05°	(5)
Moriya. (Menthol from Yonezawa.)	-59.3°	(14)

Crystallizatien of Menthol.

Menthol crystallizes in colorless needles or prisms of the hexagonal system. (5,6,14,66,80.)

Oil cooled to 4°C. yields colorless hexagonal crystalsof menthol. (8, 27.)

Peppermint oil exposed on glass slide yields an abundance of crystals of menthol, after some hours of exposure. (26)

When oil is reduced to zero, the yield of menthol is about twenty percent. A mixture of ice and salt is used to make the freezing mixture. A temperature of -8°F. being reached. (30)

Menthol crystallizes in snow-white acicular crystals, and sometimes in white needles with a satiny lustre, forming stellate groups. (28)

Menthol obtained by cooling oil to 15°C., when it crystaaaaaa

lizes out in large trimetric crystals. (2)

Menthol crystallizes in prismatic crystals. (6)

Schrenck finds them to be prismatic and needle shaped in the plant. (60)

Distillation.

Atkinson & Yoshida purified menthol by distillation. (9)

Solubility.

Menthol is soluble in the following substances:

alcohol	(6,7,43,59,60)
bisulphide of carbon	(7,80,16)
ether	7,42,43,16,6,59,80)
chloroform	(16,80,6,59)
fatty oils	(7,42)
volatile oils	(7,42,6,80)
cuastic alkalies(alcoholic)	(7)

Slightly soluble in

warm acetic acid	(20,59)
glycerine	(20,43,6)

Insoluble in

hydrochloric acid	(20)
water	(7,42,43,59,80)

Specific Gravity.

Moriya (15°C.)	0.89	(14)
Laval (Pipmenthol.)	0.9003	(20)
Laval (jap. menthol.)	0.9002	(20)
Laval (Jap. menthol.)	0.9004	(20)

MEDICINAL PROPERTIES.

Menthol is used in the following diseases:

Anaesthesia of larynx	(46)
Anaesthesia of nose	(46)
Anaesthesia of pharynx	(46)
Anodyne	(55)
Antiseptic	(39,41)
Asthma	(57)
Bites (mosquito)	(44)
Bracheolgia	(40)
Burns	(44)
Catarrh (stomach)	(6)
Catarrh (intestinal)	(6)
Cholera	(6)
Coryza	(6)
Diptheria	(6)

Eczema	(58)
Headache	(4,12,54)
Influenza (epidemic)	(6)
Migraine	(54)
Neuralgia	(40)
Neuralgia (intercostal)	(40)
Neuralgia (supraorbital)	(54)
Ointment (formula)	(52)
Phthisis	(6,50)
Piles	(48)
Plaster (formula)	(45)
Pregnancy	(55,56)
Pruritus	(47,58)
Pruritus ani	(58)
Sciatica	(40)
Snuff (formula)	(5153)
Toothache	(20,40)
Ulcus ventriculi	(6)
Urticaria	(44,47,58)

Yield from Oil.

Colcord obtained eight ounces of menthol from sixteen ounces of oil, or 50 percent. (63)

Twitchell says the yield varies from 45 to 60 percent. Oil produced in dry seasons yields as much as 75 percent. (17)

English	(194)	
	Total menthol	58 to 66 percent.
	Free menthol	50 to 60 percent.
	As ester	3 to 14 percent.
French	(194)	
	Total menthol	43.7 to 46 percent.
	Free menthol	35.7 to 39.4 percent.
	As ester	7.1 to 10 percent.
Italian	(194)	
	Total menthol	44.1 to 46.6 percent.
	Free menthol	36.7 to 41 percent.
	As ester	5.6 to 7.4 percent.
Japanese	(194)	
	Total menthol	70 to 91 percent.
	Free menthol	65 to 85 percent.
	As ester	3 to 6 percent

Russian	(194)	
	Total menthol	50.2 percent.
	Free menthol	46.8 percent.
	As ester	3.4 percent.
Saxon	(194)	
	Total menthol	54.7 to 67.6 percent.
	Free menthol	46.5 to 61.2 percent.
	As ester	5.7 to 8.2 percent.

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