A Study of the Yield of Camphor and of Camphor Oil Obtained from the Laurus Camphora of Jamaica

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A STUDY OF THE YIELD OF CAMPHOR AND OF CAMPHOR OIL OBTAINED FROM THE LAURUS CAMPHORA OF JAMAICA

A Thesis

Presented to the Faculty

of the University of Kansas.

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In commerce we have various qualities of camphor, all coming from the Asiatic Orient. The principal quality, and the most common, is the ordinary camphor from Formosa, China and Japan. This has the chemical composition expressed by the formula $C_{10}H_{16}O$, and is extracted from the camphor tree, having the botanical term Laurus Camphora, or the Cinnamomum Camphora, or Camphora Officinalis. This tree is a laurel species coming from the far East and from the islands of Asia. The chemical study of camphor is not within the scope of this article. It is limited to the study of Laurus Camphora, in regard to its yield in commercial camphor(1). We are occupied principally in the examination of its properties and its foliage which grows in Jamaica.

Up until the Japanese-Russian war in 1904 and 1905, the growth of the Laurus Camphora, outside of the control of the Japanese government, for industrial purposes was unknown. At the time the war broke out the United States was importing over two million pounds of camphor from Japan yearly. During the first year the

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(1) This investigation was carried out jointly with Professor H. W. Emerson.

importation was cut down one million pounds and the second year, one and one-half million pounds, while the price was increased from 62 cents to $1.57 per pound. This large decrease in the amount imported resulted from the use of camphor by the Japanese in the manufacture of gun powder. The United States was not the only country to suffer these high prices, as at this time we stood second in the importation of camphor, with Germany first, and England and France a close third and fourth, respectively. A stimulus for the extention of the camphor industry was therefore furnished, and Italy was the first country to take it up. Previous to this time the few camphor trees growing in Italy were used only as ornamental trees. The industry soon spread to other subtropical countries, and at present the Laurus Camphora is being cultivated at Ceylon, Canary Islands, Southern California, Florida, Texas and Jamaica.

These same reasons also brought forth many companies to prepare camphor synthetically, but almost all of these companies failed in the attempt to compete with the natural product. This was a stronger argument in favor of the extracting of camphor from the Laurus Camphora as there seems to be no fear of approaching possible competition on the part of

synthetic camphor. Since synthetic camphor must be prepared from the costly American pinene, or even from ordinary essence of turpentine, it will always be possible to obtain with greater profit camphor from the natural source. Finally, if, as there is reason to believe, the foliage of the Laurus Camphora is less rich in camphor when these trees grow in Formosa, the Japanese will be justified in fearing competition against their camphor obtained from the roots and trunks of century old trees, not only on the part of synthetic camphor derived from the pinene of Germany, England and the United States, but the possible competition of natural camphor ffom Jamaica and the Mediterranean countries, where, just as in the case of Japan, hand labor is relatively cheap, and where the product may be put on the market more easily. The increasing high prices after the war were a stimulus to new cultural innovations in spite of the fact that synthetic camphor was already on the market. It is proposed to cultivate the camphor tree in such a way as to permit the annual harvest of foliage. The object of the investigation which we carried on was, therefore, to determine the quality and amount of camphor and oil that can be obtained from the twigs and leaves of the

trees growing in Jamaica.

The methods for determining the quantity of camphor are not very precise. The camphor in fact, besides containing water, is found mixed with essential oils from the transformation by which it is derived. Furthermore, in the process of drying and distilling it is not possible to separate it from water and the essential oil, except with a loss of some of the camphor. We have thought it best, therefore, to follow a method which may be like the one used in Industrial extraction. The results which we obtained are also obtained on a large scale, when the wood and foliage of the camphor tree are subjected to distillation . The method which proved to be most suitable was the distillation of the leaves and twigs with steam.

The leaves, twigs or any other part of the plant, are cut up, weighed and introduced into a large galvanized iron can, two feet high and one and one-half feet in diameter, holding about six thousand grammes of leaves. This can contains a false bottom made of fine wire gauze, permitting the passing in of steam below the leaves by means of a glass tube, which reaches to the bottom of the can. This glass tube is connected with a steam generator which has a capacity of five gallons

of water. The steam, together with the camphor in the vapor form, is led out through a large condenser, connected with a two-liter balloon flask, which is surrounded by running water and this in turn is connected with a second condenser, leading into a large bottle filled with alcohol. Almost all of the camphor is condensed in the balloon flask, but a very little passes over and is condensed in the second condenser. The camphor together with the oil, is collected on the water, as it is soluble in water to the extent of only one part to one thousand, and is separated from the water by filtration. The camphor in this form is a heavy brownish-white substance containing oil and other impurities.

The steam distillation for the leaves lasted two hours, at the end of which time the camphor of the leaves was generally all removed. Rarely was it necessary to continue the distillation longer. The steam distillation for removing the camphor from the twigs required five hours duration. Long distillations cause a decomposition of some of the constituents of the oil of camphor, giving the distillate a peculiar odor which is not pleasant and is very hard to remove. All of the oil comes over with the camphor and is removed by pressing it out in a small hand press.

There was sent to us a total of 787.5 pounds of leaves, wood and roots, and we received 620.25 pounds, making a loss of 21.2% during transportation.

The following tables contain the results obtained from examining the leaves:

Sack No. I.

TABLE I.

2000	grammes	of	leaves	54.00	grammes	of	camphor	&	oil 2.7 %
5000	"	"	"	119.0	"	"	"	"	" 2.4%
2000	"	"	"	44.5	"	"	"	"	" 2.2%
5000	"	"	"	130.0	"	"	"	"	" 2.6%
2000	"	"	"	42.0	"	"	"	"	" 2.1%
5000	"	"	"	105.0	"	"	"	"	" 2.1%
2000	"	"	"	45.0	"	"	"	"	" 2.3%
5000	"	"	"	132.0	"	"	"	"	" 2.6%
28,000				671.5					2.4%

This was the best sack of green leaves, and gave a little higher percent of camphor.

Sack No. II.

TABLE II.

(1)	2000	grammes	of	leaves	23.0	grammes	of	camphor	& oil 1.2%)
(1)	2000	"	"	"	30.5	"	"	"	" " 1.52%)
(1)	5000	"	"	"	93.5	"	"	"	" " 1.87%)
	2000	"	"	"	38.0	"	"	"	" " 1.90%
	2000	"	"	"	38.0	"	"	"	" " 1.90%
	5330	"	"	"	137.0	"	"	"	" " % 2.57%
	18330				360.0				1.82%

(1) Omitting because of leaks and using only those in which there was no known loss of camphor, gives an average of 2.27%.

This was a sack of wettest leaves and the leaves

were mildewed considerably, and this seems to decrease the percentage of camphor more than would be accounted for by the increased moisture. The mildew possibly causes the breaking up of the cells which contain the camphor and which seems to hold it quite effectively during ordinary drying.

Sacks Nos. III. & IV.

TABLE III.

1500	grammes	of	leaves	30.0	grammes	of	camphor	&	oil	2.0%
2000	"	"	"	39.5	"	"	"	"	"	1.98%
2000	"	"	"	39.0	"	"	"	"	"	1.95%
2000	"	"	"	46.5	"	"	"	"	"	2.33%
2000	"	"	"	40.0	"	"	"	"	"	2.00%
2000	"	"	"	41.8	"	"	"	"	"	2.09%
2000	"	"	"	48.0	"	"	"	"	"	2.4%
140	"	(500	leaves)	3.0	"	"	"	"	"	2.1%
2000	"	"	"	62.0	"	"	"	"	"	3.1%
2000	"	"	"	59.5	"	"	"	"	"	2.98%
2000	"	"	"	60.0	"	"	"	"	"	3.00%
19640				469.3						2.38%

The difference in the percent yield as noted in the leaves was due to the difference in moisture contained by them, which ranged from 25% in the leaves, on the outside of the sack, to 40% in the wet leaves in the center of the sack.

Summary of the camphor and oil in the green leaves.

TABLE IV.

19,640	grammes of leaves	469.3	grammes of camphor & oil	2.38%
9,350	" " "	213.0	" " " " "	2.27%
27,950	" " "	671.5	" " " " "	2.40%
56,940		1353.8		2.35%

On removing the oil from the camphor.

TABLE V.

1.32% pure camphor in green leaves.
.54% oil of camphor.
.387% water.
.043% loss in manipulation
2.350%

Percentage of camphor contained in the dried leaves.

Sack 5.

TABLE VI.

6000	grammes of leaves	160.	grammes of camphor & oil	2.66%
6000	" " "	142.0	" " " " "	2.36%
2500	" " "	70.0	" " " " "	2.80%
6000	" " "	176.0	" " " " "	2.93%
20500		548.0		2.69%

Sack No. VI.

TABLE VII.

	2000	grammes of leaves	38.0	grammes of camphor & oil	1.90%
(1)	5000	" " "	70.0	" " " " "	1.40%
	2000	" " "	48.0	" " " " "	2.40%
	5000	" " "	100.0	" " " " "	2.00%
	5000	" " "	133.0	" " " " "	2.66%
	2000	" " "	54.5	" " " " "	2.73%
	21,000		443.5		2.08%

(1) Omitting because of leak in the still.

16,000 grammes of leaves 373.5 grammes of camphor & oil. 2.33%

Sack No. VII.

TABLE VIII.

5000	grammes	of	leaves	125.0	grammes	of	camphor	&	oil 2.50%
2000	"	"	"	44.0	"	"	"	"	" 2.20%
5000	"	"	"	136.0	"	"	"	"	" 2.72%
2000	"	"	"	48.0	"	"	"	"	" 2.40%
5000	"	"	"	126.0	"	"	"	"	" 2.52%
2000	"	"	"	55.0	"	"	"	"	" 2.75%
5000	"	"	"	140.0	"	"	"	"	" 2.80%
2000	"	"	"	54.0	"	"	"	"	" 2.70%
2500	"	"	"	70.0	"	"	"	"	" 2.80%
30,500				798.0					2.60%

Summary of results of dried leaves.

TABLE IX.

20,500	grammes	of	leaves	548.0	grammes	of	camphor	&	oil 2.69%
16,000	"	"	"	373.5	"	"	"	"	" 2.33%
30,500	"	"	"	798.0	"	"	"	"	" 2.60%
67,000				1719.5					2.54%

Removing the oil from the camphor.

TABLE X.

600 grammes	gave	365	grammes	camphor 60.83%
		110	"	oil 18.33%
		120	"	water 20.00%
		5	"	loss 0.84%

TABLE XI.

500 grammes	gave	305	grammes	camphor 61.00%
		90	"	oil 18.00%
		95	"	water 19.00%
		10	"	loss 2. %

TABLE XII.

600 grammes gave	380 grammes	camphor	63.33%
	105 "	oil	17.5%
	105 "	water	17.5%
	10 "	loss	1.67%

TABLE XIII.

300 grammes gave	190 grammes	camphor	63.33%
	50 "	oil	16.67%
	55 "	water	18.33%
	5 "	loss	1.67%

TABLE XIV.

Yield of camphor is made up of

1.55% camphor in dried leaves
0.445% oil of camphor
0.471% water
0.074% loss in operation
2.540%

Camphor in dead leaves.

TABLE XV.

2730 grammes leaves 38 grammes camphor 1.39%.

The variation of the camphor content depends both on the very tender leaves which contain more water, and also on the proportion of twigs mixed with the leaves.

On comparing Table V. with that of Table XIV., it is evident that the perventage of camphor is much higher in the dried leaves, while the percentage of oil is lower. The transformation of the oil of camphor into

camphor is presumably aided by an oxidizing fungus(1).

From the above determinations we believe we can safely state that the dry leaves left for a long time on the ground are much richer in camphor than the green leaves. This is probably due to an accelerated oxidation of oil of camphor during the last stages of life in the leaves. This also shows that the camphor is so well enclosed in the vegetable cells that it is not easily dispersed. This is further substantiated by the fact, that the dead leaves still contain a considerable quantity of camphor, as indicated in Table XV. In comparing our results with those of Giglioli(2), they appear very favorable. The form, size and conditions of the leaves does not indicate hybridization taking place by the changed conditions to which the plants were subjected. In all cases where hybridization does take place, the percentage of camphor found in the leaves is greatly decreased.

Table XV. shows the amount of camphor contained in the twigs.

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(1) J. of Soc. of Chem. Ind. Vol. 21, p. 1036.
(2) Book La Camfora Italiana by Italo Giglioli, Chap. XIII.

First, twigs from the green leaves.

TABLE XVI.

2,275	grammes of twigs	24.15	grammes of camphor & oil		1.06%
2,800	" " "	22.00	" " " " "		.81%
2,800	" " "	38.00	" " " " "		1.36%
4,000	" " "	51.00	" " " " "		1.27%
10,550	" " "	138.00	" " " " "		1.30%
22,425		273.15			1.16%

From 273.15 grammes of camphor & oil obtained.

TABLE XVII.

136.60	grammes of camphor	50% camphor
60.28	" " oil	22% oil
61.24	" " water	22.4% water
15.03	" " loss	5.0% loss

The green twigs then yielded.

TABLE XVIII.

.580% camphor
.255% oil of camphor
.260% water
.065% loss

Twigs from dried leaves.

TABLE XIX.

8770 grammes of twigs 84 grammes camphor and oil 0.99% yielded 46.2 grammes camphor, 55% of crude gum is camphor, 0.5445% of dried twigs is camphor.

Camphor contained in the wood.

TABLE XX.

1800 grammes of wood 11 grammes camphor and oil 0.61%

The bark from the camphor tree and the roots did not contain any camphor. The rpots, however, yielded 20 cc. of oil, having a strong safrol odor, and a specific gravity of 1.044 at 15°C. The specific gravity of safrol at 15°C. is 1.065.

In comparing the percentage of camphor found in the twigs (Tables XVIII & XIX) with that in the leaves (Tables V. & IX), it will be seen that it runs less than half, and taking this into consideration with the length of time it takes to completely extract the camphor from the leaves and twigs, and the fact that long distillations bring over a disagreeable odor caused by the breaking down of the oil, it appears that for industrial work the camphor must all be obtained from the leaves. However, if all the camphor is to be removed from the twigs, the leaves and twigs will have to be separated before distillation. The amount of camphor contained in the wood and roots of these young trees may be regarded as worthless. However, as the trees become older the indications point to an increased amount of camphor in the wood, but the question at present is not to obtain camphor from the woods but from the leaves and twigs, so as to spare the forests from destruction.

The camphor obtained by the above method is separated from the oil by pressing and subjecting to

further purification by sublimation. This is accomplished as follows: the camphor is mixed with sand, slacked lime and carbon, in an iron dish, covered with a large funnel. It is heated by electricity so as to obtain a more constant temperature. The sand is added to moderate the release of camphor rendering more uniform the process of sublimation, the lime to remove water and any oils present, and the carbon to remove the color. The temperature is gradually raised to the boiling point of camphor, about 204°C. The process of sublimation used was not of the best, and a loss of ten percent was the result. The camphor completely sublimes without leaving a residue.

The product obtained is a white crystalline substance, having a peculiar odor and taste. When first taken from the press and dried, it had a melting point of 175.5°C. and boiling point of 203°C., on subliming, melting point 176°C., boiling point 204°C. and on resubliming and crystallizing from petroleum ether, melting point 177°C., and boiling point 204.5°C. It is dextro rotatory 42.82°. Specific Gravity 0.980. Very readily soluble in alcohol but only slightly soluble in water. One part dissolves in 1000 parts of water.

The properties of camphor required by the U. S. P. are as follows:

(1)
Melting point 174°C.
Boiling point 204°C.
Specific gravity 0.990
Dextro rotatory 41°

The above properties indicate this to be a good pure gum camphor, possessing all of the properties required by the U. S. P.

The oil obtained from the green leaves was fractionated into the portions, as shown in Table XXI.

Boiling point	158° to 165° -----	11.2%	
" "	165° to 173° -----	14.5%	
" "	173° to 178° -----	9.82%	
" "	178° to 190° -----	10.20%	
" "	190° to 195° -----	5.45%	45.2% camphor obtained from these fractions.
" "	195° to 205° -----	12.9%	
" "	205° to 228° -----	21.1%	
" "	228° to 240° -----	5.45%	
" "	240° to 255° -----	3.63%	
Residue	----------	1.82%	

The oil from the dried leaves gave the following results.

TABLE XXII.

Boiling point	158° to 175° ------	7%
" "	175° to 185° ------	19%
" "	185° to 195° ------	20%
" "	195° to 205° ------	20%
" "	205° to 220° ------	10%
" "	220° to 240° ------	10%
" "	240° to 252° ------	5%
Residue	----------	9%

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(1) Pharmacopoeia of the United States, Eighth Decennial Revision, p. 88.

After several fractionations the following fractions were obtained.

TABLE XXIII.

Boiling point	158° to 170°	9.3%	
" "	170° to 175°	10.3%	
" "	175° to 185°	21.7%	
" "	185° to 195°	7. %	
" "	195° to 205°	22.4%	camphor
" "	205° to 215°	6.3%	separated
" "	215° to 230°	7.7%	
" "	230° to 235°	2.7%	
" "	235° to 240°	1.3%	
" "	240° to 260°	2.3%	
" "	260° to 280°	2.3%	
Residue		6.4%	

The oil from the dried leaves gave the following results.

TABLE XXIV.

Boiling point	150° to 195°	41.5%
" "	195° to 220°	41.5%
" "	220° to 245°	8.5%
Residue red oil		8.5%

The oil has a specific gravity 0.915.

Analysis of oil of camphor. Schimmel's Report 1902, Oct.(1)

Six samples. TABLE XXV.

Boiling point	175° to 180°	26 to 38%
" "	180° to 190°	30 to 44%
" "	190° to 195°	11 to 19%
" "	195° to 200°	4 to 7%
" "	200° to	4 to 6%

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(1) J. of Soc. Chem. Ind. Vol. 21, p. 1551.

400 cc. of the oil obtained by combining fractions which boiled at the same temperature were redistilled and the following results were obtained.

TABLE XXVI.

I.	158° to 165° -------	20 cc -- 5%,	Sp. gr.	0.844
II.	165° to 173° -------	76 cc --19%,	" "	0.860
III.	173° to 178° -------	43 cc - 10%	" "	0.870
IV.	178° to 192° -------	56 cc - 14%	" "	0.899
V.	192° to 202° -------	08 cc - 2%	" "	0.915
V-a.	Camphor 101 gms.		25%	
VI.	202° to 215° -------	26 cc - 6.5%	" "	0.924
VII.	215° to 228° -------	20 cc - 5%	" "	0.928
VIII.	228° to 236° -------	18 cc - 4.5%	" "	0.931
IX.	236° to 252° -------	16 cc - 4.0%	" "	0.933
X.	Residue ----------	20 cc - 5.0%		
			100.0%	

V. and VI. are the result of six fractionations. Starting with 120 cc. boiling between 192° and 215°C. we froze out successively,

TABLE XXVII.

55	grammes	of	camphor
26	"	"	"
14	"	"	"
6	"	"	"
Total -- 101			

Thus from 400 cc. of oil 25.25% of camphor was obtained. Comparing the oil of camphor from the Jamaica leaves, Table XXVI, with that of the analysis of camphor oil, Table XXV, we find,

TABLE XXVIII.

		Jamaica.	Schimmels.
Boiling point	175° to 180°	34%	26-38%
" "	180° to 185°	14%	30-44%
" "	185° to 190°	7%	11-19%
" "	190° to 215°	31%	10% camphor,
" "	215°	18.5%	4% safrol & eugenol.

The last three constituents are valuable and for this reason the Jamaica oil is a very good oil.

The constituents of the oil as given by Allen(1).

TABLE XXIX.

Pinene -------	$C_{10}H_{16}$ -------	158° to 167°
Phellandrene -	$C_{10}H_{16}$ -------	170°
Cineol -------	$C_{10}H_{18}O$ ------	176°
Dipentene ----	$C_{10}H_{16}$ -------	180°
Camphor ------	$C_{10}H_{16}O$ ------	204°
Terpineol ----	$C_{10}H_{16}O_2$ -----	232°
Safrol -------	$C_{10}H_{14}O$ ------	236°
Carvacrol ----	$C_{10}H_{14}O_2$ -----	274°
Sesquiterpene-	$C_{15}H_{24}$	
Eugenol ------		

The oil as first obtained in pressing the camphor is a colorless liquid, which soon turns yellow on exposure to the air. On fractionating this a colorless transparent liquid is obtained, specific gravity 870-910. It boils from 150° to 195°. The chief constituents are pinene, phellandrene, cineol, dipentene and some camphor. On further fractionating, a yellow and a

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(1) Schimmel & Co. Allen's Commercial Organic Analysis, Vol. II. Part III, p. 804.

red oil are obtained. The camphor is separated out from the yellow oil and safrol from the red oil.

There are two grades of camphor oil on the market, known as the "light camphor Oil and the "heavy camphor " Oil, respectively. The former oil consists of the lower fractions, obtained in the manufacture of safrol. It has a specific gravity from 895 to 900. The rectified camphor oil has been recommended as a substitute for the oil of turpentine, although it cannot replace the latter in the manufacture of paints. It is useful as a solvent for resins. The "heavy camphor " Oil is of a light green colored liquid, boiling at 240° to 300°, specific gravity from 960 to 970. It is also used as a solvent for resins and for scenting soft soap. The oil of camphor has only come into use within the last few years.

From this report it is evident that the determinations are almost all made from the leaves which in all cases were richer in camphor than the wood: the very opposite being true in Japan, Formosa and China. However, from the percentage of the camphor contained in the leaves, with the cheap labor and the good methods for obtaining the camphor and oil, which has been found to possess equal properties with the best camphor placed on the market, renders possible the industry of camphor in Jamaica.

REFERENCES.

BOOK LA CAMFORA ITALIANA:
By Italo Giglioli: Professor Nella R.; Universita di Pisa, Ministero De Agricoltura E. Commercio. Roma Tepografia Nazonale, 1907.

THE FORMOSAN CAMPHOR INDUSTRY, The Island of Formosa:
By James W. Davidson, pp. 397-444, 1903.

COMMERCIAL ORGANIC ANALYSIS:
By Alfred H. Allen. Vol. II, Part III, pp. 366-377, pp. 345, 1907.

VOLATILE OILS:
By Gildemeister and Hoffman, 1900.
Schimmel's Report, 1902, Jour. of Soc. Chem. Ind., Vol. 21, p. 1551.

PREPARATION OF CAMPHOR:
Ampere Electric Company, Jour. of the Chem. Soc. Abstr. Vol. 83, 1903, I, p. 502.

CAMPHOR OIL:
Jour. Soc. of Chem. Ind., Vol. 21, p. 1244.

CAMPHOR IN FORMOSA:
Jour. Soc. of Chem. Ind., Vol. 20, p. 1262.

MANUFACTURE OF A CAMPHOR SUBSTITUTE?
Jour. Soc. of Chem. Ind., Vol. 21, p. 1411: Vol. 20, p. 604.

NATURAL CAMPHOR OILS:
Jour. of Pharmacy, Vol. 57, 1885: Vol. 58, 1886.

FORMATION OF CAMPHER OIL:
A. Tschirch, & H. Shirasawa: Jour. of Soc. of Chem. Ind., Vol. 21, p. 1036.

CONSTITUTION OF CAMPHOR:
Jour. of the Chem. Soc., Vol. 73, Part II.

CAMPHOR:
United States Dispensatory, Nineteenth Edition; Wood, Remington and Sadtler, pp. 274-280, 1907.

CAMPHOR:
Pharmacopoeia of the United States, Eighth Decennial Revision, p. 88, 1907.