NOTES ON SOME INDIAN ESSENTIAL OILS.

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List of Oils distilled and analysed.

Number	Botanical name of the plant	Common name of the plant	Portion of the plant from which oil was obtained	Page
	PINACEAE			
1	*Callitris rhomboidea*, R. Br. ...	...	Leaves	144
	GRAMINEAE			
2	.....[1] ...	Inchi grass	Flower-heads	146
3	.....[1] ...	Botha grass	,,	,,
4	.....[1] ...	Bode grass	,,	,,
5	*Vetiveria zizanoides*, Stapf. ...	Cus-cus or Veti-vert grass	Roots	147
	AROIDEAE			
6	*Acorus Calamus*, Linn. ...	Calamus	Roots	149
	ZINGIBERACEAE			
7	*Zingiber officinale*, Rosc. ...	Ginger	Rhizome	151
8	*Curcuma Zedoaria*, Rosc. ...	Zedoary	,,	153
9	*Elettaria Cardamomum*, Maton (Var. *a* minor.	Cardamom	Fruits	155
	PIPERACEAE			
10	*Cubeba officinalis*, Miq. ...	Cubeb	,,	159
	LAURACEAE			
11	*Cinnamomum Camphora*, Nees and Eberm.	Japan camphor tree	Leaves, twigs, branches, stump, stem, roots and bark	160
	RUTACEAE			
12	*Zanthoxylum Rhetsa*, D.C. ...	...	Carpels and seeds of the fruit	174
13	*Citrus Bigaradia*, Risso. ...	Bitter orange	Rind and pips	178
	UMBELLIFERAE			
14	*Coriandrum sativum*, Linn. ...	Coriander	Fruits	182
15	*Cuminum Cyminum*, Linn. ...	Cumin	,,	182
16	*Anethum Sowa*, Roxb. ...	Dill	,,	183
17	*Foeniculum Panmorium*, D.C. ...	Fennel	,,	184

[1] The correct specific names for these three grasses have not yet been decided.

FAMILY: PINACEAE.

1. *Callitris rhomboidea*, *R. Br.* (Syn. *Frenela rhomboidea*, Endl.)

This is an Australian pine introduced into the Nilgiris[1] in 1885–1891. It is stated that there are more than a hundred acres under this species in the Nilgiris, where it can reproduce itself from seed. It has been used for hedges, also as a nurse for other trees and is likely to be a useful fuel plant.[2] Many of the Australian species of *Callitris*, are known to yield economic woods, which are very resistant towards termites,[3] and different parts of the trees yield valuable essential oils, when distilled with steam. Baker and Smith[4] consider it a pity that *rhomboidea* and not *Tasmanica*, has been introduced into India, as the yield of oil from the latter is six times as great, while the ester-content of the oil is more than double. They consider the economic possibilities of *C. Tasmanica*, as a perfumery oil-producing plant, greatly superior to those of *rhomboidea*.

The leaves of this tree were obtained from the Collector and distilled the next day, 25-9-1918.

TABLE I.

Steam-distillation of C. rhomboidea leaves.

Weight of leaves	= 144·3 kilos
,, steam	= 163·6 ,,
Duration of distillation	= 7·5 hours.
Weight of oil obtained	= 254 grams.
Ratio, oil : steam	= 0·0016 : 1
Yield of oil on weight of raw material	= 0·17 per cent.

Baker and Smith[5] in Australia distilled the leaves in January and obtained a yield of 0·033 per cent. of essential oil. Puran Singh[6] distilled the fresh leaves in winter in the Nilgiris and obtained a yield of 0·039 per cent. The yield of oil obtained in these laboratories is about five times the value recorded by him. The season during which the leaves are distilled is well known to have an effect on the yield of oil, thus more oil is obtained in summer than in winter. It is doubtful, however, if the season will produce the difference noted

[1] *Perf. and Essent. Oil Rec.*, 1917, 8, 304.
[2] J. S. Gamble, *A Manual of Indian Timbers*, 1922, p. 695.
[3] Baker and Smith, *A Research on the Pines of Australia*, Sydney, 1910, p. 60.
[4] *Perf. and Essent. Oil Rec.*, 1918, 9, 108.
[5] *Loc. cit.*, p. 220.
[6] *Perf. and Essent. Oil Rec.*, 1917, 8, 304.

above. *C. Tasmanica*[1], which Baker and Smith suggest introducing into India, gave 0·14 per cent. essential oil when distilled in March, and 0·208 per cent. in June.

Table II gives the analytical constants of (*a*) the oil obtained in these laboratories, (*b*) the oil obtained by Puran Singh, (*c*) the Australian oil and (*d*) the oil from *C. Tasmanica*. The ester in both these oils consists almost exclusively of geranyl acetate.[1]

The ester percentage is lower in the sample distilled here.

TABLE II.

Analysis of leaf oils of C. rhomboidea.

	Bangalore, 1918	Puran Singh[2]	Baker and Smith[3]	*Callitris Tasmanica*[4]
d_{15}^{15}	0·8715	0·8717	0·8878	0·8976–0·9088
n_{D}^{25}	1·4703	1·4723	1·4747	1·4738–1·4779
a_{D}^{25}	−29·1°	−27·6°	−19·2°	+ 1·0° to − 5·8°
Acid value	...	1·2	...	...
Saponification value	47·1	51·1	...	...
Ester per cent. as $C_{10}H_{17}OCOCH_3$ ...	16·5	17·8	30·43	59·9–62·2

The following are the results of a fractional distillation of 27 gms. of oil at 683 mm. pressure.

TABLE III.

Distillation of C. rhomboidea oil at 683 mm. pressure.

Temperature in degrees centigrade	Weight of oil in grams	Per cent. on whole oil
160–170	11·7	43·3
170–180	3·2	11·9
180–200	4·6	17·0
Residue	7·25	27·0

The oil contains nearly 50 per cent. terpenes.

[1] Baker and Smith, *loc. cit.*, p. 223. [2] *Loc. cit.* [3] *Loc. cit.*, p. 220.
[4] Baker and Smith, *loc. cit.*, p. 223.

FAMILY : GRAMINEAE.

2–4. *Inchi, Botha and Bode grass oils.*

The Botha grass grows just beyond the Mysore border in the North Arcot District of the Madras Presidency. The oil from this grass had an odour very similar to that of the oil from the Inchi grass growing in Travancore. Another oil, which has a similar odour is the Bode grass oil, obtained from a grass growing in Mysore. The analytical constants, obtained from these samples of oils are given below.

TABLE IV.

Analytical data for some Cymbopogon oils.

—	Inchi grass oil from Travancore	Botha grass oil from Madras	Bode grass oil from Mysore
d_{15}^{15}	0·9200	0·9038	0·9231
n_D^{25}	1·4849	1·4748	1·4831
a_D^{25}	− 40·0°	+ 39·3°	− 24·7°
Acid value	...	...	1·3
Saponification value	5·9	44·7	45·5
Saponification value after acetylation ...	98·4	120·7	112·7
Total alcohols as $C_{10}H_{18}O$...	29·2 per cent.	36·5 per cent.	34·4 per cent.
Distillation at 685 mm. pressure	160–170 = 28·5 per cent. 240–275 = 30·0 per cent.	150–180 = 40 per cent. 180–240 = 30 per cent. 240–260 = 20 per cent.	...
Date of examination	1–11–20	10–1–25	24–9–20

The analytical constants of the Botha grass oil indicate that it has a higher ester-content, a lower specific gravity than Inchi grass oil and is dextro-rotatary. The difference may be partly due to the different stages of ripeness of the grasses. Moudgill and Krishna Iyer [1] have found in the Inchi grass oil, *l*-borneol, *l*-terpineol, *l*-limonene, *l*-terpinine, and sesquiterpenes. It is intended to examine the Botha grass oil when more of the material is available.

[1] *Perf. and Essent. Oil Rec.*, 1922, 13, 292.

5. *Vetiveria zizanoides, Stapf.*

It is from the roots of this grass, commonly known as the cus-cus grass, that vetivert oil is distilled. In India it grows mostly wild, but is largely cultivated in Java and Reunion. The oil is much valued commercially, as its uses in perfumery are manifold.

The roots used in the first three distillations were obtained from the Bangalore bazaar. There are two grades of roots offered for sale, the first quality roots being superior and priced about double the second quality. In distillation 4, Table V, 9·1 kilos of roots were obtained from Agra. These, on being freed from adhering mud weighed only 4·1 kilos and were equal to the second quality roots in appearance.

The vetivert oil is only very sparingly volatile and hence a lengthy distillation is necessary. It is found that at the beginning of the distillation 1 to 3 kilos of steam are required to distil 1 gram of oil and towards the end, to distil the same quantity 6 to 20 kilos of steam are required.

The yield varied from 0·62 to 0·79 per cent. for first quality roots and 0·2 to 0·3 for the inferior quality. Puran Singh[1] who some years ago distilled the roots grown in different localities in India obtained yields between 0·37 to 1·14 per cent. The first quality roots yield three times the quantity compared with the second quality roots. Soaking the roots in water before distillation appreciably increased the yield of oil from the roots (cf. 2 and 3, Table V).

TABLE V.

Steam-distillation of cus-cus roots.

No. of distillation	Weight of roots in kilos	Quality of roots	Treatment before distillation	Weight of steam in kilos	Weight of oil in grams	Ratio	Yield per cent. on raw material
1	50·4	II	Disintegrated ...	670·8	102	0·00015	0·20
2	39·2	I	Do. ...	754·6	244	0·00032	0·62
3	1·26	I	Steeped in water for three days.	174·5	10	0·000057	0·79
4	3·6[2]	II	Kept in water for 24 hours in the still.	192·0	10	0·000052	0·28

[1] *Chemist and Druggist*, 1914, 86, 51.

[2] This represents the weight of the dry root after deducting moisture at 10·8 per cent. These roots were from Agra.

The oils were dark brown. 8 grams of oil from distillation 4 on being redistilled at 3–4 mm. pressure, gave 7 grams of a golden yellow liquid boiling at 120–160°.

Table VI gives the analytical constants for the samples of oil obtained in these laboratories and also constants for the two types of commercial oils—the superior Java oil from the dry roots and the inferior Reunion oil from the fresh roots.

TABLE VI.

Analysis of the oil from cus-cus roots.

Distillation No.	2	2 redistilled	3	4 redistilled	Java oil	Reunion oil
d_{15}^{15}	1·0028	1·0008	1·0030	1·0040	1·015 to 1·004	0·990–1·02
n_{D}^{25}	1·5214	1·5210	1·5210	1·5191	1·524 to 1·529	1·517–1·529
a_{D}^{25}	...	+24·2°	+25·5°	−65·0°	+ 25° to + 37°	+22° to +37°
Acid value	21·4	...	...	41·2	27–65	4·5–17
Saponification value ...	31·0	29·7	33·8	...	...	...
Ester value ...	10·4	...	...	...	9·8–23	5–20
Ester value after acetylation.	102·3	...	...	146·8	130–158	124–145
Total alcohols as $C_{15}H_{24}O$.	43·4	...	...	64·8	56·6–70·4	53·7–63·9

On redistillation under 10 mm. pressure of 30 grams of oil, (No. 2), the following fractions were obtained, 145–165 = 16·6 per cent. (−4·3°),[1] 165–175 = 24 per cent. (+16·0°), 175–180 = 16·6 per cent. (+26·6°), 180–195 = 18·3 per cent. (+36·2°), 195–215 = 8·3 per cent.[2] 215–240 = 8·3 per cent., residue 6·6 per cent. The major portion of the residue dissolved in 98 per cent. (by weight) alcohol, leaving only about 15 per cent. undissolved. The specific rotation of this residue in alcoholic solution was + 52°, the concentration of the solution being 0·57 gram per 100 cc. solution.

Semmler [3] obtained the following four fractions, by fractional distillation of the German distilled oil at 12 mm. pressure, 129–175 = 23

[1] The figures within brackets give the optical rotation of the fraction.
[2] The optical rotation of the mixture of this fraction and the next one was + 40°.
[3] *Ber.*, 1912, 45, 2347.

per cent., 175–190 = 34 per cent., 190–250 = 8 per cent., 250–300 = 30 per cent.; the oil distilled in Reunion gave at 10 mm. pressure, 120–150 = 23 per cent., 150–165 = 43·5 per cent. and 165–185 = 31 per cent.

A comparison with the results obtained in these laboratories indicates that the Bangalore oil is midway between the German and the Reunion distilled oils, as regards its proportion of high boiling constituents; the Bangalore oil has no constituents boiling above 240° at 10 mm., while nearly a third of the German distilled oil boils between 250° and 300° at 12 mm. An examination of the literature of vetivert oil shows great variation in the constants and chemical composition of the oils examined; to what extent this is due to the different stages of ripeness of the roots is not yet clear. The oils obtained from the flower-heads of grasses are, however, well known to undergo rapid changes in chemical composition. [1]

Puran Singh[2] steam-distilled, under laboratory conditions, 75 grams of vetivert oil distilled in India from Indian-grown roots and obtained 65 grams of oil, which was lævo-rotatary. The residue was found to be a dark red resin whose specific rotation in alcoholic solution is given as + 488·04°. It has been advanced that the usual dextro-rotation of the oil is due to the small amount of the resin and that the oil free from the resin will be lævo-rotatary.[3] The Bangalore oil gave no similar resin when distilled under reduced pressure; the residue had no such high positive rotation and the redistilled oil had a dextro-rotation of 24·2° and not a lævo-rotation.

FAMILY: AROIDEAE.

6. *Acorus Calamus, Linn.* (Calamus root.)

The dry roots, stated to be grown in the Coimbatore district were bought in the bazaar at Rs. 6-4-0 per 25 lbs. They had been dressed on the surface for appearance and it is likely that some oil was lost during the process of peeling, as some of the essential oil glands are said to be on the surface.

The results of distilling the disintegrated ($\frac{1}{8}$" screen) roots are given in Table VII.

[1] *This Journal*, 1925, 8A, 16.
[2] *Chem. and Drug.*, 1914, 86, 51.
[3] Allen, *Commercial Organic Analysis*, 1917, 9, 348.

TABLE VII.

Steam-distillation of calamus root.

Weight of disintegrated roots = 20·7 kilos.
Moisture = 9·1 per cent. Date. 13-11-23.

	Time in hours	Oil in grams	Steam in kilos
	1·5	127	27·3
	3·75	95	71·0
	3·5	51	76·4
	1·5	Nil.	
Total ...	8·75	273	174·7

Yield on dry roots = 1·5 per cent. Ratio, oil : steam = 0·00156 : 1.

The oil sank in the receiver, being heavier than water. The unpeeled dry root yields 1·5 to 3·5 per cent. of volatile oil while the fresh root which contains 70–75 per cent. water, yields about 0·8 per cent. The Japanese root which is possibly that of *Acorus spurius*, Schott, yields as much as 5 per cent.[1] The results of analysis of calamus oil are given in Table VIII.

TABLE VIII.

Analytical data for calamus root oil.

—	Bangalore	Constants for genuine calamus oil [2]	Japanese oil [2]	Java oil [2]
d_{15}^{15}	1·0694	0·958 – 0·970	0·970 – 0·995	1·0771 – 1·0783
n_D^{25}	1·5030	1·5000 – 1·5080	1·5095 – 1·5175	1·5504 – 1·5507
a_D^{25}	+6·2°	+9° to +35°	−12° to +25°	+1·0°
Acid value	1·4	0 – 3	0 – 2	...
Saponification value ...	5·1	5 – 20	1 – 10	0·12
Saponification value after acetylation	16·6	30 – 55	16 – 28	...
Solubility	Sol. at 8° in 1·5 vol. of 70 per cent. (vol.) alcohol.	Easily soluble in 90 per cent. alcohol.	Soluble in equal volume of 90 per cent. alcohol.	Soluble in 1 to 1·5 volume of 70 per cent. alcohol.

[1] Gildemeister and Hoffmann, *The Volatile Oils* (Eng. Transl.), 1916, **2**, 255.
[2] Parry, *The Chemistry of Essential Oils* (fourth ed.), 1921, **1**, 90.

The results of distillation of 30 grams of calamus root oil at 10 mm. pressure are given in Table IX.

TABLE IX.

Distillation of calamus root oil at 10 mm. pressure.

Temperature in degrees centigrade	Quantity in grams	Per cent. on whole oil
Below 130	Nil.	...
130-140	4	13·3
140-150	4	13·3
150-157	20	66·6
Residue	1	...

Semmler [1] isolated pinene, camphene and camphor in the low-boiling portions of genuine calamus oil. None of these appear to be present in the oil distilled here. The analytical constants also indicate that these do not fall within the limits of the known commercial oils, this being particularly true of the density.

FAMILY : ZINGIBERACEAE.

7. *Zingiber officinale, Roscoe.*

The gingers of commerce are of widely different value and are obtained chiefly from Jamaica, India and China. The plant is cultivated all over the warmer and moister parts of India up to an elevation of 4000 to 5000 ft. in the Himalayas. The best ginger grown in India is on the Malabar coast. The rhizomes are cleaned and sun-dried by the cultivators and are then submitted to an elaborate process of cleaning and bleaching with sulphur fumes and are finally sorted into several grades, as brown rough ginger, rough bleached ginger and white ginger, and then exported.[2] In preparing the best quality white ginger, the bleached and dried ginger is carefully trimmed and scraped in order to remove the outside tissue and make it appear as smooth as possible. The scrapings and the rejected ginger are waste ginger, and would form a valuable source of the volatile oil.

[1] *Ber.*, 1913, 46, 3700.
[2] An excellent account of the process is found in *J. Roy. Soc. Arts.*, 1916-17, 65, 511.

The distillation, as the results will indicate, is a very prolonged one, not so much because of sparing volatality of the oil, but on account of the oil cells not being sufficiently broken. The product required twice recrushing before all the volatile oil was removed. When distilled, dried ginger is stated to give 2–3 per cent. volatile oil.[1] The yield of 3·54 per cent. obtained is high. This is chiefly due to the fact that the essential oil mainly lies just under the epidermis and is most concentrated at the top of the rhizome.[2] As the scrapings principally consist of these portions of the root they contain a high percentage of oil.

The oil was golden yellow and of good aroma, but had none of the pungency of ginger, the pungent principle being non-volatile.[3] The constants are within the limits for ginger oil except for the presence of a larger proportion of alcohols. The results of fractional distillation, compared with the values obtained by Thresh [4] (and quoted by Parry [5]) indicate a slightly lower percentage of terpenes and a higher percentage of the 265–280° fraction, the sesquiterpene alcohol fraction containing zingiberol.[6] The oil undergoes decomposition at ordinary pressure and hence the results of fractionation at this pressure may be misleading.

TABLE X.

Steam-distillation of waste ginger from the West Coast.

The material was crushed between rollers before distillation.
Weight 144·5 kilos.

Time in hours	Oil in grams	Steam in kilos
2·5	2580	56·8
3·7	957	67·7
0·8	156	22·7
1·0	81	20·9
6·0 [7]	736	130·9
3·0	154	65·5
10·0 [7]	451	227·3
27·0	5115	591·8

Ratio, oil : steam = 0·00864 : 1 ; yield = 3·54 per cent.

[1] Gildemeister and Hoffmann, *The Volatile Oils* (English Transl.), 1916, 2, 281.
[2] Gildemeister and Hoffmann, *loc. cit.*
[3] For the constitution of the pungent principle, see Lapworth, *J. Chem. Soc.*, 1917, 111. 777 ; and Namura, *ibid.*, p. 769.
[4] *Year Book of Pharmacy*, 1879, p. 426.
[5] *Loc. cit.*, p. 99.
[6] Brookes, *J. Amer. Chem. Soc.*, 1916, 38, 430.
[7] The material was removed from still, recrushed and again distilled.

TABLE XI.

Analysis of the oil from waste ginger.

	Bangalore	Constants for the pure oil [1]
d_{15}^{15}	0·8822	0·875 - 0·886
n_D^{25}	1·4898 [2]	1·4795 - 1·4855
a_D^{25}	−39·2°	−28° to 50°
Acid value	2·1	0 - 2
Ester value	7·7	0 - 15
Ester value after acetylation ...	49·8	33 - 42

TABLE XI*a*.

Distillation of ginger oil at 685 mm.

Fraction No.	Boiling point	Per cent.	Per cent. given by Thresh
1	to 150	1·4	5·0
2	150–200	5·0	10·0
3	200–240	12·3	8·0
4	240–265	40·0	60·0
5	265–280	29·8	7·0
Residue	.....	9·7	10·0

8. *Curcuma Zedoaria, Roscoe.*

According to Hooker [3], the rhizomes of this plant are cultivated throughout India. The plant has long been cultivated in Ceylon, where the leaves constitute a favourite vegetable.

The roots are locally cultivated and are available in the Bangalore bazaar in the form of dry thin discs about 0·5″ to 1″ in diameter. A sample was disintegrated ($\frac{1}{8}$″ screen) and steam-distilled.

[1] Allen's *Commercial Organic Analysis*, 1917, 9, 362.
[2] According to Parry, *loc. cit.*, p. 99, the range of the refractive index for ginger oils is 1·4885 to 1·4950.
[3] *Flora of India*, 1875, 6, 210.

TABLE XII.

Distillation of dry discs of Curcuma Zedoaria.

Weight of powder = 45 kilos. Moisture = 13·9 per cent.

Time in hours	Oil in grams	Steam in kilos
0·5	190	11
0·5	29	11
1·0	20	22
4·5	85	98
6·5	40 [1]	142
6·0	10	113
.....	15 [2]	...
Total ... 19·0	389	397

Ratio, oil : steam = 0·00097 : 1.

Yield = 1·01 per cent. on dry rhizomes; according to Parry [3], the roots yield between 1 to 1·5 per cent. of an essential oil.

TABLE XIII.

Analytical data of the Curcuma Zedoaria oil.

The oil is greenish brown by transmitted light.

——	Bangalore	Parry [4]
d_{15}^{15}	0·9863	0·982 -1·01
n_D^{25}	1·5024	1·5020-1·5060
a_D^{25}	...	+ 8° to + 18°
Acid value	2·1	0-2·5
Saponification value	9·8	15-25
Saponification value after acetylation.	73·4	55-68
Aldehydes; absorption with sodium sulphite	3·5	...
Solubility in volumes of 80 per cent. alcohol	1·5 at 15°	1·5-2

[1] The oil, at this stage and onwards, was heavier than water
[2] This oil was collected from second and third receivers.
[3] *Loc. cit.*, p. 107.
[4] *Ibid.*

Distillation at 685 mm. pressure, 165–180° = 1.1 per cent.; 180–230° = 23 per cent.; 230–290° = 56 per cent.

The oil has a lower ester-content and a slightly higher content of total alcohols than the oils quoted by Parry.

9. *Elettaria Cardamomum*, *Maton* (var. α- minor).

The cardamoms of commerce are the fruits of the plant *Elettaria Cardamomum*, Maton. There are two distinct forms or varieties of this plant.

1. *E. Cardamomum*, Maton (var. α- minor).—This is the variety that is largely cultivated. There are two chief forms of this variety known in trade, the fruits of which are distinguished by the names of the localities, the Mysore and the Malabar. The Mysore fruits vary in length from 0·25″ to 0·8″; the Malabars are smaller than Mysores, rarely more than 0·5″ long and there is a greater proportion of seed to pericarp in them.[1] The Mysore plant is of a more robust habit and is better suited for growth at higher elevation than the Malabar and is more resistant to exposure and wind.[2] The value of the produce from the Mysore plant is equal to that of the Malabar plant. The oils obtained from the seeds of these forms seem to be similar,[3] and these appear to be the seeds recognised by the British Pharmacopœia. Both the Mysores and the Malabars are largely cultivated in Ceylon and in the moist forests of Malabar, Canara, Coorg, Mysore and Travancore. To produce fruits of a light-straw colour, without black or brown spots, the ripe fruits are gathered, washed and dried in the sun. Occasionally the fruits are washed with soap or soapnut solution or exposed to sulphur fumes. They are then sorted according to their colour, plumpness and maturity before being marketed. The material distilled in these laboratories belonged mainly to the Mysore variety.

2. *E. Cardamomum*, var. β.—This was once considered to be a separate species, but was later shown by Fluckiger[4] to be only a variety of *E. Cardamomum*, Maton. The plant grows wild in Ceylon and the fruits are generally known as 'Ceylon wilds.' This fruit is oblong, 1″ to 2″ long and rather narrower than the Malabar fruit, distinctly three-sided and dark-greyish brown when dry, the inner seeds larger, more numerous and less aromatic.[5] Till 1900, the

[1] Parry, *The Chemistry of Essential Oils*, *loc. cit.*, p. 103.
[2] Ridley, *Spices*, 1912, p. 326.
[3] Parry, *Pharm. J.*, 1899, 63, 105.
[4] *Pharmacographia* (second ed.), p. 644.
[5] Ridley, *loc. cit.*, p. 325.

cardamom oil of commerce was distilled from these fruits. The oil differs from that obtained from the Mysores or the Malabars. The yield of oil is 3 to 6 per cent.[1]

Other varieties in addition to these two are occasionally met with, e.g., Siam cardamoms or fruits of *Amomum Cardamomum*, Linn., from Siam; Bengal or Nepal cardamoms (*Amomum aromaticum*, Roxb.) growing in the lower valleys of Bhutan and Sikkim.[2] These two and many others are derived from quite different plants, yield different oils and are of no importance.

The results of numerous distillations are given in Table XIV.

In Nos. 1, 2 and 8 the seeds only were used, but in the remaining distillations, both seeds and pericarp were utilised. Nos. 4, 5, 6 and 7 were carried out in a flask, under laboratory conditions, blowing steam at ordinary pressure.

In No. 2, the uncrushed seeds were employed. In Nos. 4, 5, 6, 7, 10, 11 and 14, the material was completely crushed between rollers and distilled. In Nos. 8, 9, 12 and 13 uncrushed or partly crushed material was first distilled, then removed from the still, recrushed and redistilled.

Crushing the material before distillation, as may be expected, shortens the time of distillation and effects a reduction in the steam used. Thus the ratios of oil to steam indicate that in Nos. 10 and 11, 1·7 times the quantity of oil is obtained per unit of steam, as when uncrushed material is distilled (Nos. 9, 12 and 13). Crushing does not affect the yield of the oil, as is shown by the yields in Nos. 10, 11, 12 and 13. It is not necessary to distil seed and pericarp separately, as the yield is not appreciably altered by distilling crushed seed and pericarp together (cf. 8 and 9).

The yield of the oil in distillation No. 1 is very low, but the oil, as indicated by its analytical constants in Table XV, is quite normal and hence the low yield is not due to adulteration with low quality seeds, as Ceylon wilds. There are instances of genuine Mysore and Malabar cardamoms giving low yields, but the reason for the low yields is not well known. Thus Parry[3] got 2·6 per cent. of oil from Mysores and 1·3 per cent. from Malabars. The Malabar cardamoms are stated to yield 3·5 to 7 per cent. of oil.[4] The yield of oil obtained in the

[1] Parry, *The Chemistry of Essential Oils*, *loc. cit.*, p. 103.
[2] Mukherji, *Handbook of Indian Agriculture*, 1923, p. 321.
[3] *Pharm. J.*, 1899, **63**, 105.
[4] Gildemeister and Hoffmann, *loc. cit.*, p. 284.

TABLE XIV.

Steam-distillation of cardamoms.

No of distillation	Date of distillation	Material employed	Weight of raw material in kilos	Weight of steam in kilos	Grams of oil	Ratio oil : steam	Yield per cent. on raw material
1	...	Seeds ...	44·8	84·5	1061	0·0130	2·42
2	Dec. 1917	,, ...	36·8	120·0	1900	0·0158	5·16
3	...	Seeds with pericarp ...	44·1	94·5	2620	0·0277	5·94
4	...	...	0·515	...	28	...	5·44
5	Dec. 1917	Green dried crushed between rollers	1·661	...	89	...	5·36
6	...	,,	1·265	...	65	...	5·10
7	...	,,	1·105	...	54	...	4·90
8 (*a*)	May, 1918	Seeds partially crushed ...	198·98	210	12362	0·059	6·20
8 (*b*)	,,	Fully crushed ...	,,	...	490	...	0·25
8 (*a*+*b*)	,,	...	,,	...	12852	...	6·45
9 (*a*)	...	Put in whole ...	201·4	201	11835	0·059	5·87
9 (*b*)	...	Crushed 9 (*a*) ...	,,	54·5	980	0·010	0·49
9 (*a*+*b*)	...	...	,,	255·5	12815	0·0502	6·36
10	...	Crushed in plain rollers ...	198·4	145·0	12218	0·0843	6·10
11	...	Crushed seed in plain rollers...	197·7	167·3	12024	0·0718	6·0
12 (*a*)	...	Uncrushed ...	202·7	119·5	9597	...	4·73
12 (*b*)	...	Crushed ...	,,	118·2	2830	...	1·40
12 (*a*+*b*)	...	...	,,	237·7	12427	0·0513	6·13
13 (*a*)	...	Uncrushed ...	202·3	137·2	9706	0·0714	4·80
13 (*b*)	...	Crushed ...	,,	121·4	2700	0·0222	1·33
13 (*a*+*b*)	...	...	,,	258·6	12406	0·048	6·13
14	Sept. 27, 18	Cardamom with pericarp ...	113·07	184·0	6997	0·038	6·22

distillations in these laboratories has been, on an average, above 6 per cent. The pericarp of the fruit also contains some essential oil. Nothing is known as regards the yield and composition of this oil in the case of Mysore or Malabar cardamoms. In the case of Ceylon wilds, Schimmel & Co. obtained a yield of 0·2 per cent. oil from the pericarp and the analytical constants of the oil were found to be

identical with those of the seed oil.[1] The various methods of curing and bleaching are bound to affect the yield of oil from the pericarp, and it appears that the quantity was very small in the material we have distilled as there was no appreciable difference in the yields with and without the pericarp for the same batch of seeds. We have not distilled pericarps alone.

The analysis of the oils obtained is given in Table XV. Nos. 1 and 13 are within the limits stated by Gildemeister and Hoffmann to be usual for genuine oils. Nos. 5, 6 and 7 seem also to be normal as indicated by their optical rotation. Nos. 2, 3 and 4 are not within the limits. The yield of the oils, in these cases, varied between 5 and 6 per cent. and is thus quite normal. The specific gravity of the oils is high, thus eliminating the possibility of the material being the Ceylon wilds, but the optical rotation is low. It is possible that this low rotation is due to the fruits being immature. No data bearing upon the relation between age of fruit and properties of the oil are available.

TABLE XV.

Analytical constants for cardamom oil.

Number of distillation	d_{15}^{15}	n_D^{25}	a_D^{25}	Acid value	Saponification value
1	0·9326	1·4613	+ 44·0°	0·6	120·6
2	0·9283	1·4603	+ 17·6°	...	...
3	0·9264	1·4620	+ 15·1°	0·36	96·5
4	0·9315	1·4608	+ 17·5°	...	...
5	...	...	+ 28·1°	...	...
6	...	...	+ 29·6°	...	...
7	...	...	+ 30·2°	...	...
13	0·9349	1·4610	+ 31·4°	1·3	156·4
Malabar Oil[2]	0·923-0·944	1·460-1·465	+ 24° to + 41°	Up to 4	94-150[3]
Oil from Ceylon wilds[4]	0·895-0·906	...	+ 12° to + 15°	...	25-70

[1] Gildemeister and Hoffmann, *loc. cit.*, p. 287.
[2] *Ibid.*, p. 286.
[3] The limits are for ester value, not saponification value.
[4] Gildemeister and Hoffmann, *loc. cit.*, p. 287.

10. *Cubeba officinalis*, Miq. (Syn. *Piper Cubeba*, Linn.).

A sample of cubebs stated to be grown in the Mysore Province, was obtained from the local bazaar. The fruits were crushed and distilled. From 4·5 kilos of fruits, using 41 kilos of steam during six hours, a total of 539 grams of oil was obtained corresponding with a 11·85 per cent. yield and an oil : steam ratio of 0·0132 : 1.

TABLE XVI.

Analytical constants of oil of cubebs.

—	Bangalore	British Pharmacopœia[1]
d_{15}^{15}	0·9167	0·910-0·930
n_D^{25}	1·4894	1·486-1·500
a_D^{25}	−29·9°	−25° to −40°
Saponification value ...	0·5	...
Saponification value after acetylation	24·1	...
Solubility in volumes of 90 per cent. alcohol ...	5 at 16°	Up to 10[2]
Colour	Light green	Pale green or greenish

TABLE XVII.

Fractionation of oil of cubebs at 685 mm. pressure.

Temperature in degrees centigrade	Oil in grams	Per cent.
140—170	1	5
170—225	4	20
225—245	3	15
245—265	9	45
265—280	2	10
Residue and loss	1	5

[1] 1914, p. 265.

[2] Parry, *loc. cit.*, p. 109.

Parry[1] quotes the results of distillation of four samples of pure oil by Messrs. Orange.[2] Three of the samples gave no fraction boiling below 230°. The fourth had, below 220° = nil, 220–250° = 12·8 per cent., 250–280° = 79·0 per cent., residue = 8·2 per cent. These results are not in agreement with the identification of pinene, camphene and dipentene in the pure oil.[3]

The sample of oil distilled in these laboratories has 25 per cent. boiling below 225° at 685 mm. This quantity of a low boiling fraction is high for a normal oil of cubebs. The fruits used in the distillation were possibly slightly immature. The oil just passes the standard of the British Pharmacopœia.

FAMILY: LAURACEAE.

11. *Cinnamomum Camphora*, Nees and Eberm.

(syn. *Camphora officinarum*, Bauh., *Laurus Camphora*, Linn.).

The natural habitat of this tree is Formosa and parts of Japan and China. It is now well known that the tree is capable of growing in a wide range of climates throughout the tropical and warmer temperate regions, and that the proportion of camphor and other constituents formed depends upon the climatic conditions and locality. Thus the trees grown in Mauritius, and known to be genuine by a botanical examination at Kew, do not yield camphor.[4]

The tree has been grown successfully in a number of localities in India. At one time, it is said to have flourished in Nepal and Tipperah, between Bengal and the Upper Irrawady.[5] In Ceylon, the tree grows well at elevations of 5000 ft. and less. There is a fine avenue of trees in Dehra Dun and also in the botanic gardens at Saharanpur and Calcutta. The last is said to have been planted as early as 1802. In the Nilgiris, it does well even up to 7000 ft. It has been grown in many parts of Burma, among others Maymyo and the Bhamo district and recently[6] 650 acres have been planted in the Southern Shan States at Yakutsk to produce camphor on a commercial scale; but no yields of camphor and camphor oil, from different parts of the tree growing in different parts of India, are available except for those growing in Dehra Dun.

[1] *Loc. cit.*, p. 109.
[2] *Perf. and Essent. Oil Rec.*, 1914, 5, 372.
[3] Oglialoro, *Gazzetta*, 1875, 5, 467.
[4] *Bull. Imp. Inst.*, 1916, 14, 585.
[5] Hooper, *Pharm. J.*, 1896, 56, 21.
[6] *J. Soc. Chem. Ind.*, 1920, 39, 279R.

If it is intended to manufacture camphor on a commercial scale, it is first to be decided whether the camphor is to be obtained from the wood or from the leaves. To grow trees and obtain it from the wood necessitates a long delay before any yield is obtained ; so whenever an Indian camphor industry has been contemplated, attention has been directed to the possibility of obtaining the camphor mainly from the leaves, as in the inquiry at Dehra Dun. Howard, Robertson and Simonsen[1] have worked out a rational method of planting, showing the total yield of leaf and camphor per acre, the number of flushes which might be taken per annum and other points of importance. They found for Dehra Dun conditions, with a spacing of 7 ft. × 7 ft., roughly 900 bushes per acre can be obtained, yielding 43·9 lbs. camphor per acre per annum. They drew attention to the high content of camphor from leaves grown in Southern India and suggest that the climate in South India should be more suitable for camphor bushes than that of Dehra Dun. They came to the conclusion that camphor may be easily cultivated in all parts of India with an annual rainfall of 40 inches and over, but as a commercial enterprise its cultivation should not be attempted outside the tropical areas and that even in the latter, the financial returns are likely to be small.

The material used in this investigation was obtained from a large tree, growing in the Mysore Government botanical gardens at Bangalore. The tree was the oldest in the gardens, its age being about 40 years. It was only about 25 ft. high and had a maximum circumference of 10 to 12 ft., and was thus rather stunted in comparison with the height of 100 ft. it is stated to attain in its natural habitat Formosa and Japan. In Ceylon [2] under favourable conditions the tree attains a height of 18 to 20 ft. and a diameter of 6 to 7 ft. in five years. In Dehra Dun, some of the 17–19 year old trees were 53–54 ft. in height. The Bangalore tree was quite healthy and no serious diseases of insect or fungoid origin seem to have been observed. As it came in the way of certain garden improvements, it was transplanted to another site, where it started making fresh growth, but died within a short time.

The leaves, twigs, bark, branches of various sizes, trunk and the roots were distilled (for results see Table XVIII), the oils from the various parts analysed and the camphor-content in each estimated. (See Tables XVIII and XXII). A few experiments were made to find the proper method of preparing the wood before distillation. The camphor-content in parts of trees grown in other places are given in Table XX for comparison.

[1] *Ind. Forest Rec.*, 1923, 9, 307.
[2] Troup, *The Sylviculture of Indian Trees*, 1921, 3, 791.

The distillations were carried out in a still which could hold about 60 kilos of green leaves and 120 kilos of chips. The material for the first seven distillations in Table XVIII was obtained from prunings before the tree was transplanted and for the later distillations, the material was from the whole tree after it died.

Leaves were carefully separated from stems and twigs and distilled in three lots. The first lot was quite green, half of the second lot had dried, and the third had completely dried in the air. The twigs No. 4*a* in Table XVIII were cut into small pieces 5–8 cm. long and distilled. The spent twigs from 4*a* were air-dried, disintegrated and again distilled in 4*b*. In No. 5, twigs of about the same size as in 4*a* were disintegrated and distilled. There was a very intense smell of camphor during disintegration, making it probable that an appreciable amount of camphor was lost during the process. This was confirmed by estimation of camphor in oils from Nos. 4 and 5, and the lower yield in 5 is due to camphor having volatilised during disintegration. The trunk of the tree distilled in Nos. 14 and 15, Table XVIII, was cut into chips ($3'' \times 0{\cdot}3'' \times 1''$) and well mixed, to make it as homogeneous as possible. It was divided into four lots; two of them were distilled in 14*a* and the other two first disintegrated and distilled in 15. The exhausted chips in 14*a* were disintegrated and again distilled in 14*b*. The camphor-content was 1·44 per cent. in 14(*a*+*b*), and 1·06 per cent. in 15, indicating loss of camphor during disintegration.

The results of several individual distillations are given in Table XIX.

It will be noted in Table XIX, that 80 per cent. of the total distillate comes over during the first hour of the distillation of the disintegrated twigs, while during the distillation of the twigs in small pieces only 35 per cent. is obtained during the same time. Similarly 90 per cent. comes over in the first hour with disintegrated stump and only 31·7 per cent. with stump in chips. In each case slightly more steam was passed through the material which gave the lower yield. Although distilling chips takes twice as much steam as distilling the disintegrated wood, it has the advantage that it causes no loss of camphor, as has been proved to occur during disintegration.

The bark in No. 6, Table XVIII, was obtained from branches 5–12 cm. diameter and these branches without bark were distilled in No. 7. In all other distillations up to No. 14, the material was cut into chips and distilled. The spent chips were air-dried, disintegrated and again distilled except in Nos. 10 and 11. In No. 15, as has

TABLE XVIII.

Steam-distillations of different portions of camphor tree.

No.	Date of distillation	Description of material	Weight of material in kilos.	Moisture per cent.	Weight of dry material in kilos.	Total oil in grams.	Camphor per cent. on total oil.	Camphor per cent. on dry material.	Camphor oil per cent. on dry material.	Total volatile oil percentage yield calculated on dry material.
1	4-10-23	Green leaves	18·2	28·1	13·0	160	40·6	0·50	0·32	0·82
2	8-10-23	Leaves	43·6	20·0	34·0	710	43·0	0·90	1·20	2·10
3	12-10-23	Dry leaves	10·5	8·0	9·5	136	30·5	0·39	1·04	1·43
4 (*a*)[1]	16-10-23	Twigs (0·2-1 cm.) diameter	50·9	25·6	37·7	383	35·5	0·33	0·69	1·02
4 (*b*)[2]	24-10-23	Do.	...	...	...	94	nil.	nil.	0·25	0·25
4 (*a*+*b*)	...	Do.	...	...	...	477	25·2	0·33	0·94	1·27
5	22-10-23	Twigs (0·2-2·5 cm.)	53·6	19·4	43·2	379	20·0	0·17	0·71	0·88
6 (*a*)	29-10-23	Bark (from branches)	42·3	21·0	33·4	137	...	...	...	0·41
6 (*b*)	30-10-23	Do.	...	...	...	29	...	...	...	0·09
6 (*a*+*b*)	...	Do.	...	...	...	166	5·0	0·02	0·48	0·50
7 (*a*)	3-11-23	Branches (5-12 cm.) diameter	64·0	13·6	55·4	600	6·5	0·06	1·03	1·09
7 (*b*)	8-11-23	Do.	...	...	...	55	nil.	nil.	0·10	0·10
7 (*a*+*b*)	...	Do.	...	...	...	655	5·9	0·06	1·13	1·19
8 (*a*)	20-6-24	Branches (below 10 cm.) diameter	83·2	17·2	68·9	726	12·0	0·13	0·93	1·06
8 (*b*)	30-6-24	Do.	...	...	...	76	nil.	nil.	0·11	0·11
8 (*a*+*b*)	...	Do.	...	...	...	802	10·9	0·13	1·04	1·17
9 (*a*)	25-6-24	Branches (10-15 cm.)	83·2	15·5	70·5	1051	...	...	...	1·50
9 (*b*)	3-7-24	Do.	...	...	...	40	nil.	nil.	...	0·06
9 (*a*+*b*)	...	Do.	...	...	...	1091	...	...	...	1·56
10	1-7-24	Main stem and branches (10-18 cm.)	83·6	16·4	70·0	1285	...	...	...	1·84
11	4-7-24	Do.	68·2	16·4	56·8	1303	12·0	0·30	2·0	2·30
12 (*a*)	7-7-24	Roots	86·3	32·0	58·6	3830	28·7	1·87	4·68	6·55
12 (*b*)	11-7-24	Do.	...	...	...	830	3·2	0·04	1·36	1·40
12 (*a*+*b*)	...	Do.	...	...	...	4660	24·2	1·91	6·04	7·95
13 (*a*)	15-7-24	Stump	115·5	35·0	75·0	2975	...	...	...	4·00
13 (*b*)	18-7-24	Do.	...	...	...	805	...	...	...	1·40
13 (*a*+*b*)	...	Do.	...	...	...	3780	...	...	...	5·40
14 (*a*)	25-7-24	Do.	80·9	30·6	56·1	2350	...	...	...	4·20
14 (*b*)	1-8-24	Do.	...	...	...	878	...	...	...	1·60
14 (*a*+*b*)	...	Do.	...	...	...	3228	25·0	1·44	4·36	5·80
15	29-7-24	Do.	75·5	30·6	52·3	3158	17·5	1·06	5·04	6·10

[1] The low yield is due to loss of camphor; the condensing water was allowed to become too hot.

[2] In the table (*a*) always refers to the original distillation of the chips and (*b*) to the second distillation, after the exhausted chips have been dried and disintegrated.

TABLE XIX.

Details of individual distillations.

Distillation No. 2. Leaves.

Time in hours	Oil in grams	Steam in kilos
1·0	281	9·0
1·5	166	16·0
2·5	146	27·0
1·5	35	16·0
2·0	35	22·7
1·5	15	16·0
1·5	5	11·3
2·0	20	22·7
2·0	7	22·7
Total. 15·5	710	163·4

Ratio, oil : steam = 0·0044 : 1·0.

Distillation No. 4*a*. Twigs.

Time in hours	Oil in grams	Steam in kilos
1·25	170	16·0
1·0	100	14·0
1·0	30	13·0
2·75	35	36·0
3·0	30	32·0
2·0	10	22·7
2·0	5	28·0
1·0	3	14·0
14·0	383	175·7

Ratio, oil : steam = 0·0022 : 1·0.

Distillation No. 4*b*. Spent chips from 4*a* disintegrated.

1	50	11·0
2	30	28·0
2	10	22·7
2	4	22·7
Total. 7	94	84·4

Ratio, oil : steam = 0·0011 : 1·0.

Distillation No. 5. Disintegrated twigs.

1	300	11·0
1	35	11·0
1	9	13·0
2	10	22·7
2	13	24·0
1	5	13·0
2	7	22·0
Total. 10	379	116·7

Ratio, oil : steam = 0·3032 : 1·0.

Distillation No. 14*a*. Stump in chips.

0·5	525	16·4
2·5	1000	54·5
2·5	440	54·5
0·75	60	16·4
1·5	227	32·8
1·0	45	21·8
3·0	33	65·6
...	20 (from pots)	...
Total. 11·75	2350	262·0

Ratio, oil : steam = 0·0090 : 1·0.

Distillation No. 15. Stump disintegrated.

0·25	1167	5·5
0·25	1093	5·5
0·50	633	11·0
1·0	125	22·0
2·5	110	49·5
2·25	30	46·4
6·75	3158	139·9

Ratio, oil : steam = 0·0225 : 1·0.

been mentioned above, the material was disintegrated before distillation.

Moisture was estimated in the different samples by heating 50 to 80 grams of the material to constant weight at 110°.

Estimation of camphor in the oils.—The method employed was to separate any solid camphor deposited at the laboratory temperature by means of the suction pump. The residual liquid was then cooled in a mixture of ice and salt and the solid camphor removed and the operations repeated until no more solid was obtainable on cooling. The camphor still dissolved in the liquid was recovered by distillation at 40 mm. pressure and collecting three fractions (1) below 90°, (2) 90–107°, (3) residue. The middle fraction was frozen repeatedly and solid camphor separated. The liquid was then further fractionated at 5–6 mm. pressure, in most cases with a five-pear column and divided into convenient 5-degrees or 10-degrees fractions. These fractions were repeatedly cooled and the camphor separated; those still containing camphor were each separately fractionated and all camphor obtainable by fractionation and freezing was recovered. No attempt was made to recover the small quantity of camphor still dissolved in the liquid. Some of the samples had to be distilled more than five times to separate all the camphor.

The method [1] usually employed to estimate camphor is mainly the same as that adopted above. Lohr recommends distilling at atmospheric pressure but as the oil was found to undergo considerable decomposition, the fractionations were all conducted at reduced pressure.

Most of the camphor oil adhering to camphor was removed at the filter pump. The camphor was then pressed between folds of filter paper. Camphor in column 8, Table XVIII, is the weight of camphor thus obtained. It usually melted at 173–174°, the correct melting point of camphor being 179°.[2]

Leaf-oil 2, Table XVIII, alone deposited camphor at the ordinary temperature, the amount being 16·2 per cent., Nos. 1, 3, 4*a*, and the liquid portion from 2 deposited 12·5, 11·0, 8·0, and 14·4 per cent. respectively of solid camphor on cooling the oil in a mixture of ice and salt. The remaining samples gave no camphor by this process and their whole camphor-content was obtained by fractionation and freezing.

[1] H. Lohr, *Chem. Zeit.*, 1901, 25, 292.
[2] *Analyst*, 1923, 48, 536.

Comparison of camphor-content with values obtained elsewhere.— The maximum yield of camphor is from the roots, 1·91 per cent. and then follow the trunk with 1·44, the half-dry leaves with 0·9, the twigs with 0·33, stem and thick branches with 0·30; last come the medium-sized branches, 5 to 12 cm. diameter, and the bark from those branches. Hood [1] in Florida, who examined the camphor-content in the different parts of a thirteen year-old camphor tree found the highest camphor 1·33 per cent. in wood, then the leaves with 0·88 per cent., thick branches 0·60 per cent., twigs 0·48 per cent., and lastly the medium-sized branches, 4 to 10 cm. diameter, with 0·37 per cent. He did not examine the roots. Although the Bangalore tree was forty years old the order in which camphor-content decreases is the same.

Table XX gives the camphor-content in different parts of the tree at various ages grown in different parts of the world. No reliable data about the camphor-content in the various parts of the Formosa tree, especially the leaves could be found in the available literature. Many factors are known to affect the camphor-content. Foremost come perhaps the climatic conditions and the soil. Eaton[2] says that the camphor tree thrives on poor laterite soil provided it is well-drained and not swampy. Hood,[3] however, working with ten different soils in Florida, found that the camphor-content in the leaves varied between 1·61 per cent. on a heavy black clay soil and 1·12 per cent. on a very light sandy soil. The use of commercial fertilisers gave increased yields of camphor in leaves. Camphor leaves from Cochin No. 4, Table XX, show a higher camphor-content than those from any other part of India. It has been concluded that a tropical climate should be more suitable for camphor bushes. Hood [3] found that leaves from trees grown in shade have a lower camphor-content than leaves from trees grown in the open (17 and 18, Table XX). Tender leaves yield more camphor than leaves allowed to remain on the tree through another growing season (15 and 16, Table XX), and lastly the season of cutting the leaves is known appreciably to affect the camphor-content. The camphor-content of leaves from trees at different ages seems to be as yet uncertain. Comparing the data given in Tables XX and XXI, it is probable that other conditions being the same, the leaves from trees aged 2 to 40 years will yield the same amount of camphor (cf. 9, 10, 12, 13 and 17, Table XX; 2 and 4, Table XXI). No. 3, Table XVIII, shows a lower yield as the leaves were lying in the room for over a week after stripping from the tree, and had completely dried. Eaton (12, 13 and 14, Table XX) claims that green leaves, mouldy leaves and air-dried leaves give practically the same yield of camphor.

[1] *J. Ind. Eng. Chem.*, 1917, 9, 552.
[2] *Dept., of Agri., Federated Malay States Bulletin*, 1912, 15.
[3] *Loc. cit.*

The yield of camphor from the twigs seems appreciably to increase up to a certain age of the tree. Thus twigs from 2 to 4 year-old trees yield 0·1 per cent. (20, 21 and 22, Table XX), from trees of 13 to 40 years about 0·3 per cent. (4, Table XVIII; 19 and 24, Table XX; 1, Table XXI).[1]

In smaller branches, up to 10 cm. diameter, the Bangalore tree indicates 0·06 to 0·13 per cent. yield of camphor. This is poor compared with 0·37 per cent. from branches of a thirteen year-old tree and 0·61 per cent. mainly camphor from a five year-old tree (6 and 7, Table XVIII; 28 and 30, Table XX).

The camphor-content in the thicker branches 10 to 18 cm. in diameter from the Bangalore tree was only 0·30 per cent., rather low compared with 0·60, 0·51 and 0·50 per cent. in the larger branches from trees, 13, 17 and 22 years old respectively (29, Table XX; 1 and 2, Table XXI).

The stump of the Bangalore tree contained 1·44 per cent. In Dehra Dun, 0·97 per cent. and 0·69 per cent. camphor were obtained, from stumps of trees 17 and 22 years old.[2] The Formosa trees which are stated to be at least a hundred years old [3] when distilled appear to yield a much larger amount of camphor, viz., 3–4 per cent. This high yield has not so far been obtained anywhere else in the world. It is probable that the older the tree is, the higher the camphor-content in its wood. Hood [4] has shown that camphor is not uniformly distributed in the wood, the outer rings having appreciably higher camphor-content than the inner rings (34 and 35, Table XX).

[1] *Loc. cit.*, p. 450.
[2] *J. Soc. Chem. Ind.*, 1920, 39, 296T.
[3] Hooper, *loc. cit.*
[4] *Loc. cit.*

TABLE XX.

Camphor-content of different parts of the camphor tree grown in India and other countries.

No.	Distillation conducted		Description of material	Place of growth	Age of the tree in years	Camphor per cent.	Camphor oil per cent.	Total volatile oil percentage yield
	in	by						
1	1896	Hooper [1] ...	Green leaves ...	Nilgiris ...	...	0·1-0·7	0·9-0·3	1·0
2	1914	Forest Chemist India [2] ...	,, ...	Madras ...	...	1·99	0·63	2·62
3	Oct., 1916	,, ...	,, ...	Burma ...	...	1·03	0·48	1·51
4	March, 1918	,, ...	,, ...	Cochin ...	...	2·01	0·32	2·33
5	Oct., 1918	,, ...	,, ...	Dehra Dun ...	17–22	0·38	3·66	4·04
6	Aug., 1921	,, ...	,, ...	,, ...	,,	1·33	2·27	3·60
7	July, 1921	,, ...	,, ...	,, ...	,,	1·87	2·55	4·42
8	Sept., 1922	,, ...	,, ...	,, ...	,,	1·87	2·21	4·08
9	1911	Department of agriculture [3] ...	,, ...	Malay ...	2	...	...	1·4-1·6 [4]
10	,,	,, ...	,, ...	,, ...	4	...	...	1·3-1·7 [4]
11	1910	Lommel [5] ...	,, ...	German East Africa.	3·5-4·5	1·0	0·20	1·20
12	1909	Eaton [6] ...	,, ...	Malay ...	5	...	...	1·17-1·22 [4]
13	,,	,, ...	Mouldy leaves ...	,, ...	,,	...	...	1·25-1·47 [4]
14	,,	,, ...	Air dried leaves ...	,, ...	,,	...	...	1·10-1·16 [4]
15	April, 1917	Forest Chemist, India [2] ...	Young leaves ...	Dehra Dun ...	17	0·59	4·24	4·83
16	,,	,, ...	Old leaves ...	,, ...	,,	0·25	1·63	1·88
17	1917	Hood [7] ...	Green leaves ...	Florida ...	12	1·44 [8]	0·51	1·95
18	,,	,, ...	,, ...	,, ...	,,	1·04 [9]	0·35	1·39
19	Oct., 1917	Forest Chemist, India [10] ...	Twigs ...	Dehra Dun ...	17–22	...	...	0·34
20	Aug., 1919	,, ...	,, ...	,, ...	,,	...	...	0·30
21	1911	...	,, ...	Malay ...	2	...	...	0·10-0·23
22	,,	...	,, ...	,, ...	4	...	...	0·13-0·22
23	1910	Lommel [5] ...	,, ...	German East Africa.	3·5-4·5	...	...	0·06-0·10
24	1917	Hood [11] ...	,, ...	Florida ...	13	0·48	0·11	0·59
25	,,	,, ...	Branches 2-4 cm. ...	,, ...	,,	0·38	0·14	0·52
26	,,	,, ...	Bark from branches ...	,, ...	,,	0·50	0·06	0·56
27	,,	,, ...	Bark from trunk ...	,, ...	,,	0·07	0·04	0·11
28	,,	,, ...	Branches 10 cm. ...	,, ...	...	0·37	0·16	0·53

29	,,	,, ...	,, 18 cm. ...	,, ...	...	0·60	0·32	0·92
30	1909	Eaton[12] ...	,, more than 1·3 cm. ...	Malay ...	5	...	...	0·61
31	...	Muriya[13] ...	Branches ...	Formosa ...	...	...	...	3·70
32	...	,, ...	Upper part of stem ...	,, ...	...	...	...	3·84
33	...	,, ...	Lower part of stem ...	,, ...	...	...	...	4·23
34	1917	Hood[11] ...	Wood (outer 4 rings) ...	Florida ...	8	1·33	0·54	1·87
35	,,	,, ...	,, (inner 4 rings) ...	,, ...	,,	0·82	0·39	1·21
36	...	Muriya[13] ...	Upper part of stump ...	Formosa ...	...	...	...	5·49
37	...	,, ...	Lower ,, ,, ...	,, ...	...	...	...	5·74
38	...	,, ...	Roots ...	,, ...	...	...	...	4·46
39	...	Schimmel & Co.[14]	,, ...	...	...	...	...	4·0
40	1909	Eaton[12] ...	,, ...	Malay ...	5	...	...	1·10

[1] *Pharm. J.*, 1896, **56**, 21.
[2] *Ind. Forest Rec.*, 1923, **9**, 307.
[3] *Bull. Imp. Inst.*, 1916, **14**, 578.
[4] This consisted mainly of camphor with a little camphor oil.
[5] *Schimmel's Report*, 1910, **2**, 27.
[6] Gildemeister and Hoffmann, *loc. cit.*, p. 452.
[7] *J. Ind. Eng. Chem.*, 1917, **9**, 552.
[8] This tree was grown in the open.
[9] This tree was grown in the shade.
[10] *J. Soc. Chem. Ind.*, 1920, **39**, 296T.
[11] *Loc. cit.*
[12] *Loc. cit.*
[13] Gildemeister and Hoffmann, *loc. cit.*, p. 450.
[14] *Schimmel's Report*, 1892, **2**, 11.

The roots distilled in Bangalore have given a higher camphor-content than any other portion of the tree, viz. 1·91 per cent. An estimation of camphor in roots, made in Dehra Dun from a tree of unknown age grown in the Botanical Gardens, Calcutta, gave negative results (3, Table XXI). Eaton [1] in Malay obtained 1·1 per cent. yield of total distillate, mainly camphor, from roots of a five-year old tree. Sawyer [2] quotes as follows from a consular report from Formosa: 'the roots contain a much larger proportion of camphor than the trees, 10 lbs. of crude camphor out of 200 lbs. of wood chips being thought satisfactory.' Schimmel and Co.[3] obtained a total yield of 4 per cent. from the roots. The bark from the medium-sized branches 2–10 cm. diameter contained the lowest percentage of camphor. Hood (26 and 27, Table XX), from bark of branches of unknown size, got a much higher yield of 0·5 per cent. and a low yield of 0·07 per cent. from the bark of the stump.

Examining column 8, Table XVIII, which gives the camphor-content on total oil, it is found that the distillate from the leaves is the richest in camphor, next comes the distillate from the twigs, and then follow those from the roots, the stump, the stem, the thicker branches, and lastly the medium-sized branches (2 to 10 cm.) and their bark.

The percentage of camphor oil from which camphor is removed is given in column 10, Table XVIII. The roots yield the maximum quantity, the stump comes next and then the stem. The leaves and the branches of all sizes yield about the same percentage of oil but less than the stem, and are followed by the twigs and then the bark which yields the least. There is a great variation in the percentage of camphor oil yielded by the leaves and its proportion in the total distillate (cf. 4, 5, 8, 11, 15 and 17, Table XX).

The yield of total distillate from different portions of the camphor tree.—Gildemeister and Hoffmann [4] state that camphor 'occurs dissolved in a volatile oil that permeates all the parts of the plant. This oil occurs most abundantly in the underground roots, less abundantly in the trunk, and still less so in the branches, twigs, and leaves. Moreover the camphor oil content varies with the height of the tree, diminishing upwards.' Examining the last column in Table XVIII with this statement in view, there is found to be good agreement. The leaves, however, yield more oil than either twigs or branches or nearly as much as the main stem. The exceptionally high yield from the roots is to be noted. Schimmel and Co.[5] put the average yield

[1] *Loc. cit.*
[2] *Odorographia*, 1894, 2, 442
[3] *Schimmel's Report*, 1892, 2, 11.
[4] *Loc. cit.*, p. 450.
[5] *Ibid.*, p. 451.

from the roots as 4·22 per cent. The yield obtained here is nearly twice as much, viz., 7·95 per cent., and of this 24 per cent. is camphor.

Table XXI gives the yield of camphor and camphor oil from three trees examined at Dehra Dun,[1] side by side with the yields obtained from the tree at Bangalore. The results indicate the difficulty of correlating the camphor-content in different portions with the age of the tree, because of the interference of other factors. Thus the twigs from the 22 year-old tree contain no camphor while twigs from trees 17 and 40 years old contain the same amount of camphor. The portions of the Bangalore tree are on the whole richer in camphor than the trees examined at Dehra Dun.

TABLE XXI.

Camphor-content in different portions of some camphor trees at different ages.

Number	1		2		3		4	
Locality	Dehra Dun[2]		Dehra Dun[2]		Calcutta[2]		Bangalore	
Age in years	17		22		Unknown		40	
—	Camphor per cent.	Camphor oil per cent.	Camphor per cent.	Camphor oil per cent.	Camphor per cent.	Camphor oil per cent.	Camphor per cent.	Camphor oil per cent.
Leaves	0·42	0·74	1·32	0·69	0·54	1·5	0·90	1·2
Twigs	0·31	0·26	nil	0·12	...	...	0·33	0·69
Small branches ...	0·25	0·15	,,	0·13	nil	0·07	0·06	1·03
Larger branches...	0·51	0·43	0·50	0·25	,,	0·17	0·13–0·3	0·93–2·0
Stump	0·97	0·86	0·69	0·29	,,	0·95	1·06–1·44	4·36–5·04
Roots	...	...	...	...	,,	2·03	1·91	6·04

Analysis of the oils.—No attempt has been made to make a detailed identification of the constituents, in any of the oils, as the camphor oils have been more than once examined by different chemists.

[1] *J. Soc. Chem. Ind.*, 1920, 39, 296T.
[2] *Ibid.*

The camphor oil of commerce is the oil from which camphor has been removed. It is of varying quality depending upon the degree of fractionation undergone, and the constituents removed from it. The value of the oil, after the removal of camphor, depends upon its content of safrole, which is used in the manufacture of heliotropin and as a cheap perfume. After the removal of camphor and safrole the oil is fractionated and put on the market, as (1) light camphor oil, containing chiefly terpenes and boiling below 200°, (2) heavy camphor oil boiling between 270–300°, and (3) blue camphor oil boiling about 300°. These fractions are used as a cheap perfume, for polishes, etc., and in the varnish and printing industry. The oil is only a bye-product and being of varying quality has no recognised constants.

Table XXII gives the analytical constants of the oils distilled at Bangalore, as well as some collected from literature for comparison. Samples 2–7 were analysed after camphor had been removed, and 8–15 before. It may be noted that the nearer the part of the tree used is to the roots, the heavier is the oil obtained from it. The twig oil from Dehra Dun has a very low specific gravity. In all cases where the material has been distilled twice, first as chips and then as powder, it is found that the oil from the powder is always lighter, due to all the camphor, which has a high specific gravity ($d_{18}^{18} = 0{\cdot}985$), passing over with the distillate from the chips. Thus the oils from distillations 4*b*, 7*b* and 8*b* were all free from camphor, boiled mainly between 230–270°, only about 15 per cent. boiling between 200–230°, and consisted of sesquiterpenes and sesquiterpene alcohols. An exception is however met with in distillation 12, where the oil from the distillation of the disintegrated powder (12*b*), contained 3·5 per cent. camphor and had an appreciable quantity of terpenes, as nearly 20 per cent. of the oil boiled below 180°. The percentage of acetylisable alcohols in the several oils varied between 10 and 20 per cent. as $C_{15}H_{24}O$. The constants for the oils are normal and agree with the constants usually given for camphor oils.

Examination of the oils for safrole.—Safrole is stated to have been found only in wood oils and so far has not been found in leaf oils anywhere.[1] The oils distilled at Bangalore when examined for safrole, showed its presence in a small quantity. The fraction boiling between 90 and 100° at 5 mm. pressure, which formed about 10 per cent. of the whole oil, in most cases was further fractionated into four or five fractions and none of the fractions had physical constants approaching those of safrole. The fraction 90–100° from the stump oil 14 (*a* + *b*), Table XVIII, was treated with 25 per cent. sodium hydroxide to remove the phenols and acids boiling at about the

[1] Gildemeister and Hoffmann, *loc. cit.*, p. 451.

TABLE XXII.

Physical constants of the camphor oils obtained.

No.	Specific gravity $d^{15\cdot5}_{15\cdot5}$	Refractive index at 25°	Optical rotation at 25°	Acid value	Saponification value	Saponification value after acetylation	Colour of the oil
2	0·9106	1·4758	+ 26·0	...	3·0	25·0	Pale green.
3	0·9253	1·4772	+ 21·5	2·3	6·5	49·2	,,
4(*a*)	0·9175	1·4681	+ 13·6	0·6	2·5	28·8	Yellow.
4(*b*)	0·9248	...	...	...	...	...	Green.
5	0·9284	1·4793	+ 10·4	1·5	7·1	49·3	,,
6(*a*+*b*)	0·9290	1·4748	+ 8·5	...	4·2	42·3	Greenish yellow
7(*a*)	0·9244	1·4678	+ 6·4	0·8	0·6	42·2	Pale yellow.
8(*a*)	0·9316	1·4692	+ 8·2	...	...	...	Greenish yellow.
8(*b*)	0·9276	...	+ 5·8	...	...	...	Yellow.
9	0·9322	1·4685	+ 9·4	...	1·5	35·5	Pale yellow.
10	0·9320	1·4680	+ 9·0	...	4·0	40·0	,,
11	0·9356	1·4690	+ 13·0	...	1·9	38·5	,,
12(*a*)	0·9518	1·4756	+ 24·5	...	2·4	26·8	,,
12(*b*)	0·9288	1·4833	+ 18·5	...	2·7	28·1	,,
13(*a*)	0·9429	1·4709	+ 19·5	...	...	...	,,
13(*b*)	0·9326	1·4802	+ 18·5	...	...	...	,,
14(*a*+*b*)	0·9459	1·4761	+ 20·4	...	1·3	35·4	,,
15	0·9432	1·4749	+ 23·5	...	1·0	27·8	,,
	0·9196[1]	...	+ 38·4	1·1	3·6	25·7	...
	0·9126[2]	...	+ 41·0	...	...	...	...
	d^{30}_{30} = 0·9313[3]	1·4787	+ 34·44	...	...	...	...
	d^{30}_{30} = 0·9165[4]	1·47	+ 32·74	...	...	...	...
	d^{30}_{30} = 0·8777[5]	1·473	...	...	...	...	...

[1] Camphor leaf oil from Malay. The analytical data are for the oil from which camphor had been removed. *Bull. Imp. Inst.*, 1916, 14, 585.
[2] Camphor leaf oil from Malay, *ibid.*
[3] Camphor wood oil from Dehra Dun. *J. Soc. Chem. Ind.*, 1920, 39, 2967.
[4] Camphor leaf oil from Dehra Dun, *ibid.*
[5] Camphor twig oil from Dehra Dun ; *Ind. Forest Rec.*, 1923, 9, 332.

same temperature as safrole, then washed and dried. This must now contain sesquiterpenes, chiefly cadinene, alcohols if any and safrole. The preparation of the nitrosite was attempted, in this case, according to the method of Goulding, [1] but no nitrosite was obtained.

Fifteen grams of the fraction boiling between 90° and 100° at 5 mm. from each of the oils obtained from roots, stump, thick branches, twigs [2] and leaves [2] (Nos. 12 ($a+b$), 14 ($a+b$), 11, 4 ($a+b$) and 2, Table XXVIII) on oxidation according to the method of Tiemann,[3] with potassium permanganate, yielded piperonylic acid weighing 0·4, 0·4 and 0·2 gram respectively, only in the first three cases. It melted in each case at 227°–228° and the equivalents were found to be 167, 169 and 167 by analysis of the silver salts, the correct melting point of piperonylic acid being 228°[4] and its calculated equivalent being 166. The proportion of safrole in the oils is however small, being only 0·2 per cent., calculated from the weight of piperonylic acid obtained.

FAMILY: RUTACEAE.

12. *Zanthoxylum Rhetsa, D. C.*

The fruits of this tree were obtained from the Malnad district of the Mysore Province, where it is only sparsely distributed. Hooker [5] in his description of *Z. Rhetsa* says, 'A tree with corky bark and spreading leafy branches, prickles straight or incurved, the old ones with a solid conic base . . . ripe carpels—solitary, the size of a pea tubercled, seed—subglobose, blue-black. The unripe carpels taste of orange-peel, the seeds like black pepper.' It is distributed on the Western Peninsula, from the Coromandel and the Concan southward. Recently, however, Simonsen and Gopal Rao [6] have drawn attention to the fact that the plant in Canara on the other side of the Western Ghats has been identified as *Z. Budrunga*, Wall. and doubt is raised as regards *Rhetsa* and *Budrunga* being two different species. Hooker [5] also had doubts about their being different and states under *Z. Budrunga* that except in the fewer leaflets, there is no differential character given by Roxburgh, between the two species. *Z. Budrunga* is stated to be distributed in tropical Himalayas, Kumaon, forests of Sylhet, Khasia Mountains, etc.

[1] *J. Chem Soc.*, 1903, **83**, 1099.
[2] The fraction in this case formed only 3 per cent. on the whole oil.
[3] *Ber.*, 1891, **24**, 2489.
[4] *Ber.*, 1892, **25**, 1128.
[5] *Flora of India*, 1875, **1**, 495.
[6] *Ind. Forest Rec.*, 1922, **9**, 141

Schimmel and Co.[1] first distilled fruits which they obtained from the north of Bengal and they described them as seeds of *Z. alatum*. Simonsen [2] pointed out that the oil from the seeds of *Z. alatum* has a different composition and that in all probability the fruits distilled by Schimmel and Co., were from *Z. Budrunga*. Semmler [2] found, in the oil distilled by Schimmel and Co., a terpene xanthoxylene (in all probability sabinene), a trace of an aldehyde and a solid ketone dimethoxyphloracetophenone. Simonsen [3] found *l*-sabinene, a small quantity of terpinene, and an unidentified alcohol. The oil distilled in these laboratories closely resembles the oil obtained by Simonsen ; hence both are probably derived from the same species.

The oils examined by Semmler and Simonsen are stated to be derived in both cases from the seeds of the plant. The fruits of *Z. Rhetsa* consist of aromatic carpels and the inner seeds which are pungent like black pepper. In our examination the carpels and the inner seeds were treated separately. The essential oil was found to be present only in the carpels while the seeds contain a fixed oil and no essential oil.

13·6 kilos of mature fruits, which had been dried in the sun, were purchased. The material was separated into 6·1 kilos carpels, 6·1 kilos seeds, 0·8 kilos twigs and 0·6 kilos dirt and dust. The results of steam-distillation of crushed carpels (moisture = 6·4 per cent.) are given in Table XXIII.

TABLE XXIII.

Steam-distillation of carpels of Z. Rhetsa.

Time in hours	Oil in grams	Steam in kilos
0·5	307	4
1·0	18	9·5
0·75	Nil.	...
Total 1·5	325	13·5

Ratio, oil : steam = 0·024 : 1 ; yield = 5·8 per cent. on dry carpels.

[1] *Schimmel's Report*, 1910, **2**, 147.
[2] *Ber.*, 1911, **44**, 2885.
[3] *Loc. cit.*

The distillation water gave no appreciable amount of oil on extraction with ether.

Simonsen [1] obtained 3 per cent. of volatile oil on roughly ground ripe seeds and 0·6 per cent. from the immature seeds. Schimmel and Co.[2] obtained 3·7 per cent. of a lemon-yellow oil and on prolonging the distillation 0·9 per cent. of a crystalline substance which could not be dissolved in the oil and was later identified by Semmler as dimethoxyphloracetophenone. There is no mention of the carpels and the seeds being separated for distillation in either case. Hence the lower yields obtained by them are probably due to their having distilled the carpels and the inner seeds together, and calculating the yield on their total weight. The weight of the carpels is about the same as that of the seeds from them and as the seeds contain no volatile oil, the yield is about half of that obtained in these laboratories.

The results of analysis of the oil are given in Table XXIV side by side with the analysis of oils examined by Semmler and Simonsen.

TABLE XXIV.

Analysis of the oil from carpels of Z. Rhetsa.

—	Bangalore	Young seeds[3]	Ripe seeds [3]	Semmler [4]
d^{15}_{15}	0·8562	d^{30}_{30}=0·8532	d^{30}_{30}=0·8426	d^{20}_{20}=0·8653
n^{25}_{D}	1·4663	1·4710	1·4676	1·4775
a^{25}_{D}	−32·6°	−37·7°	−29·55°	−23·6°
Acid value	1·2	0·18	0·3	9·9
Saponification value	5·9	3·05	8·5	10·3
Saponification value after acetylation.	49·3	24·21	56·5	33·6

Table XXV gives the results of a fractional distillation of 100 grams of the oil at 7 mm. pressure with a five-pear column.

[1] *Loc. cit.*
[2] *Loc. cit.*
[3] Simonsen, *loc. cit.*
[4] *Loc. cit.*

TABLE XXV.

Distillation of 100 grams of Z. Rhetsa at 7 mm. pressure.

Number of fraction.	Temperature in degrees centigrade	Per cent.	n_D^{25}	a_D^{25}
1	48–52	72·3	1·4660	−35·5
2	52–54	16·0	1·4660	−32·8
3	54–90	5·0	1·4693	−14·7
...	Residue	5·5	1·4835	...

The results indicate that nearly 90 per cent. of the oil consists of terpenes. Simonsen obtained at 100 mm. pressure 98–110° = 84 per cent., 110–120° = 8·8 per cent., and 120–180° = 4·6 per cent. The analysis and results of distillation show that the Bangalore oil closely resembles the oil from ripe seeds examined by Simonsen. The terpene which forms the main constituent of the oil was shown to be sabinene by oxidising 10 grams of fraction 1, Table XXV with potassium permanganate according to directions given by Wallach.[1] The sabinenic acid obtained was recrystallised from hot water and melted at 55–57°.

The fruits distilled by Schimmel differ from those examined at Dehra Dun and Bangalore in yielding dimethoxyphloracetophenone.

Pepper-like seeds of Z. Rhetsa.—The seeds had a very fleeting smell, resembling that of black pepper. They were crushed into a meal but the peppery smell disappeared. The crushed seeds when extracted with light petroleum (b.p. 40–60°) gave 29·7 per cent. of oil, calculated on the weight of the dry seeds (moisture = 12·9 per cent.).

The extract consisted of a small quantity of a solid which formed 2·7 per cent. of the dry seeds, and 27·0 per cent. of a light brown liquid which was not volatile with steam.

It was thought probable that the solid from the extract might prove to be dimethoxyphloracetophenone which Schimmel & Co. obtained on prolonged steam-distillation of the fruits. The solid melted at 53° and had a saponification value of 227·2 showing that it was a glyceride and not the solid ketone which melts at 85°.

The small quantity of twigs when distilled with steam, did not give any volatile oil.

[1] *Annalen*, 1908, 359, 266.

13. *Citrus Bigaradia, Risso.*

The fruit of this plant is called the bitter orange or the Seville orange. Two lots of these fruits were bought in the local bazaar. The second lot was divided into two portions, the yellowish green ripe fruits and the green unripe fruits.

Method of extracting the oil.—The method employed was the ordinary sponge method. The fruits were kept overnight in water. Three longitudinal incisions were made in the fruits and the rind separated from the pulp. The convex side of the peel was pressed against a sponge, with a vessel underneath. The oil in the peel was absorbed in the sponge which was then periodically pressed into the vessel. The pressed liquid was extracted with ether, the ether distilled off and the oil separated.

The yield of the oil and its analysis from three lots of fruits are given in Table XXVI.

TABLE XXVI.

Yield and analysis of bitter orange oils.

—	Bangalore I	Bangalore II	Bangalore III	Bitter orange oil (Italy) [1]
Number of fruits ...	50	23	25	...
Colour of fruits ...	Yellowish green to green	Yellowish green	Green	...
Average weight of fruit in grams .	...	163	153	...
Weight of total peel in grams ...	2420	1256	1173	...
Total oil in grams ...	6·7	4·0	2·9	...
Yield per cent. on weight of peel.	0·28	0·32	0·25	...
$d_{15\cdot5}^{15\cdot5}$	0·8629	0·8649	0·8658	0·853-0·855
n_D^{25}	1·4737	1·4746	1·4749	...
a_D^{25}	+ 84·7°	+ 88·2°	+ 86·3°	+ 92° to + 94°
Per cent. non-volatile residue in the oil	12·0	9·9	12·1	2·5-3·5

[1] *Perf. and Essent. Oil Rec.*, 1922, 13, 239.

Bitter orange-pip oil.

The bitter orange-pips are so far a waste product. D. G. Hewer[1] drew attention to the large quantity of these pips separated by centrifugal means in marmalade factories. It was suggested that the seeds may be dried and the oil removed either by hydraulic pressure or by extraction with volatile solvents. The 158 grams of seeds (moisture, 14·4 per cent.) from 50 fruits (sample 1, Table XXVI) and 106 grams (moisture 19·2 per cent.) from 48 fruits (samples 2 and 3, Table XXVI) were each separately extracted with ether; 30·5 per cent. and 21·4 per cent. of fixed oil, calculated on the dry seeds, were obtained. The extract consisted of 0·7 per cent. and 0·5 per cent. of a solid substance and 29·8 per cent. and 20·9 per cent. respectively of a light yellow liquid. Hewer and R. Meyer [2] obtained 37·5 and 57·3 per cent. respectively of fixed oil from the seeds.

The analytical constants of the liquid portion of the oil, along with the values obtained by Hewer and Meyer for their samples are given in Table XXVII.

TABLE XXVII.

Analytical constants of liquid portion of the bitter orange-pip oil.

——	Bangalore	Bangalore	Hewer	R. Meyer
d_{15}^{15}	0·9243	0·9257	0·9208	0·9251
n_D^{40}	1·4650	1·4652	1·4758	1·4757
Acid value ...	3·1	2·9	...	0·53
Saponification value ...	198·5	198·1	193·7	196·37
Iodine value ...	94·0	94·1	100·3	97·26

FAMILY: UMBELLIFERAE.

Coriander, cumin, dill and fennel have been distilled and their analytical constants given. These are cultivated mainly in the tropics and in the warmer temperate regions. The following Table XXVIII gives the yields of essential oil from fruits grown in India, side by side with those from fruits grown in temperate regions.[3]

[1] *Analyst*, 1917, **42**, 271.
[2] *Chem. Zeit.*, 1903, **27**, 958.
[3] The figures have been quoted from Gildemeister and Hoffmann, *loc. cit.*, **3**, 307, 314, 351 and 373.

TABLE XXVIII.

Yields of essential oils from Umbelliferae fruits grown in India and the temperate regions.

——	India	Temperate region	Country cultivating the best seed
Coriander	0·15—0·2	0·8—1·0	Russia and Thuringia.
Dill	2—3	2·5—4·0	England, Holland and Bavaria.
Cumin	3·0—3·5	3·5—4·5	Malta.
Fennel	0·7	4—6	Roumania, Galicia.

The yield of the oil from *Umbelliferae* fruits grown in temperate regions is generally stated to be higher than those grown in the tropics. The above table shows that this is specially true in the case of coriander and fennel, while in the case of cumin the yield is nearly the same as in the temperate regions. As such, unless the oils obtained from the Indian fruits are superior in some respects and fetch higher prices, they do not come into consideration for factory production. Recently[1] such a claim has been made for Indian coriander oil. It is stated that it has a higher percentage of ester and a superior odour and hence may be given a higher price.

It is not definitely known why the yield from Indian coriander fruits is small. One of the reasons frequently put forward is that a portion of the volatile oil is lost when the fruits are dried in the sun, as is usually done in India, before the fruits are put on the market. This statement is in need of experimental verification. There is no evidence of the plant being botanically distinct from the European plant, as in the case of fennel and some other Indian *Umbelliferae* fruits, which give lower yields. The botanical examination of the plants growing in various parts of India may give useful information. If the Indian plant is botanically identical with the European plant, the lower yield of essential oil must be due to imperfect cultivation or exhausted soil. Manuring has been known to give a greatly improved yield of fruits in the West, but the effects of manures on the oils in those fruits have not been studied. It might be worthwhile to import Russian or Thuringian fruits and plant them to see if as good yields of oil are obtained from them when grown in India. The oil from Indian cumin seems to be very similar to that from European grown cumin. We have also seen above that the yield of volatile oil is only

[1] *Perf. and Essent. Oil Rec.*, 1923, 14, 121.

just a little below the highest. The yield from Indian fennel is even worse than from coriander, being only a sixth of that obtained from the European variety. The Indian plant,[1] however, is not botanically identical with the European plant. The Indian plant, *Foeniculum Panmorium*, D.C., is stated to be only a variety of *Foeniculum vulgare*, the European plant. An experimental crop of Galician or Roumanian fennel grown in India would be of interest.

As regards dill, the oils obtained from the Indian and the European plant are not similar, the chief difference being in the presence of dill-apiol in the Indian oil. The Indian plant has been often described as a distinct species, but the botanists are by no means unanimous on this point, though slight botanical differences between the Indian and the European plant are admitted by all. African dill oil produced from plants grown from English imported seed was very similar to the English dill oil. This is encouraging to those who may think of importing better *Umbelliferae* seeds and trying them on Indian soil. It is also probable that if dill-apiol is removed, the Indian oil will be somewhat similar to the European. It is well known that large quantities of these fruits are being annually exported from India,[2] yet, in spite of their importance, practically no attention seems to have been paid to improving the methods of cultivation.

In European countries, the distillation residues of these *Umbelliferae* fruits have been used as a valuable fodder for animals. Table XXIX gives the protein and fat-content of the distillation residues of European grown fruits.[3]

TABLE XXIX.

Protein and fat-content of Umbelliferae fruits.

—	Per cent. protein	Per cent. fat
Coriander ...	11-17	11-20
Fennel ...	14-22	12-18·5
Dill ...	14·5-15·6	15·5-18

Lewkowitsch [4] quotes from C. Grimm [5] the constants for fatty oils from *Umbelliferae* fruits.

[1] Umney, *Pharm. J.*, 1897, 58, 226.
[2] *This Journal*, 1918-19, 2, 15.
[3] Uhlitzsch, *Die landwirtschaftlichen Versuchsstationen*, 1893, 42, 60.
[4] *The Chemical Technology and Analysis of Oils, Fats and Waxes*, 1922, 2, Table facing p. 246.
[5] *Pharm. Zentr.*, 1911, p. 661.

14. *Coriandrum sativum, Linn.*

The fruits were purchased locally and crushed in a ball-mill and steam distilled. From 60·5 kilos of fruits using 105·5 kilos steam during five hours, 156 grams of oil were obtained corresponding with 0·25 per cent. of the raw material. The analytical constants for the oil are given in Table XXX.

TABLE XXX.

Analytical data for coriander oil.— 29-4-20.

—	Bangalore	Constants for genuine coriander Oil [1]	Indian Coriander Oil [2]
d_{15}^{15}	0·8721	0·870–0·885	0·8715–0·876
n_D^{25}	1·4576	1·4635–1·4760	1·4569–1·4612
a_D^{25}	+13·7°	+7° to +14°	+10° to +13°
Acid value	0·26	1–5	...
Saponification value	31·0	3–22	30–54·3
Saponification value after acetylation.[3]	45·4	...	...
Solubility in volumes of 70 per cent. alcohol.	3 at 15°	3	...

The Indian oil has constants within the limits given for genuine European oils with the exception of the saponification value, which is higher than that found for European oils. This indicates a higher percentage of linalyl acetate and so should make the oil more valuable. It has been stated that the Indian oil is of superior odour.[2]

15. *Cuminum Cyminum, Linn.*

The fruits of white cumin which were bought in the local bazaar were crushed and steam-distilled. From 54·8 kilos fruits using 228·6 kilos steam during ten hours 1·268 kilos of oil were obtained, corresponding with a yield of 2·35 per cent. and ratio, oil : steam of 0·0055 : 1·0. The analytical constants for the oil are given in Table XXXI.

[1] Parry, *loc. cit.*, p. 307.
[2] *Perf. and Essent. Oil Rec.*, 1923, 14, 121.
[3] The oil was acetylated using turpentine as a diluent, according to the method of Boulez, *Bull. Soc. chim.*, 1907, IV(1), 117.

TABLE XXXI.

Analytical data for oil of cumin.—2-7-20.

—	Bangalore	Constants for genuine cumin oil [1]
d_{15}^{15}	0·8945	0·900–0·930
n_D^{25}	1·4910	1·494–1·507
a_D^{25}	+ 3·6°	+ 3·3° to + 8·0°
Aldehydes per cent. by absorption with sodium bisulphite.	16	23–35 35–42 [2]
Solubility in volumes of 80 per cent. alcohol.	11 at 20°	8–10

The oil is not up to the standard. It is lighter, indicating a larger percentage of the terpenes, has a lower percentage of aldehydes and is less soluble in alcohol.

16. *Anethum Sowa, Roxb.*

The fruits purchased locally were crushed and steam-distilled. From 54·5 kilos fruits, using 356·3 kilos steam during 15·5 hours, 1186 grams of oil lighter than water and 552 grams oil heavier than water were obtained, corresponding with a yield of 2·17 per cent. of light oil and 1·02 per cent. of heavy oil and a total yield of 3·19 per cent. on the raw material.

The analysis of the light and heavy oils and also of the two mixed together in the proportion they were present in the fruits have been made. These results, along with the analysis of a sample of Baroda oil from which dill-apiol had been removed, have been given in Table XXXII.

[1] Parry, *loc. cit.*, p. 311.
[2] This is by the phenylhydrazine method ; Gildemeister and Hoffmann, *loc. cit.*, p. 315.

TABLE XXXII.

Analytical data for the dill oils.

—	Bangalore light oil	Bangalore heavy oil	Bangalore whole oil	Oil from Baroda	European dill oil [1]	Indian dill oil [1]
d_{15}^{15}	0·9313	1·0935	0·9785	0·9030	0·895-0·918	0·945-0·970
n_D^{25}	1·4853	1·5154	1·4943	1·4792	1·4830-1·4900	...
a_D^{25}	+58·2°	...	+47·6°	+63·6°	+70° to +82°	+40° to +50°
Carvone per cent. by absorption with sodium bisulphite.	17	...	19·5	18[2]	30-60	...

The dill oil examined has a high specific gravity due to the presence of a large percentage of dill-apiol. It may be noted that if the dill-apiol is removed, the constants of the Indian oil approximate to those of the European oil. The Bangalore light oil was soluble at 13·5° in 5 volumes and the whole oil at 13·5° in 3 volumes of 80 per cent. alcohol.

17. *Foeniculum Panmorium, D.C.*

The fruits were purchased in the local bazaar in two lots, crushed and steam-distilled. The results of steam-distillation are given in Table XXXIII.

TABLE XXXIII.

Steam-distillation of Indian fennel.

Number of distillation	Date of distillation	Weight of raw material in kilos	Moisture per cent.	Weight of dry material in kilos	Weight of steam in kilos	Oil in grams	Ratio. oil : steam	Yield per cent. on dry material
1	7-6-1920	40·5	...	...	207·3	213	0·0010	0·53[3]
2	7-6-1923	11·1	5·9	10·4	26·0	92	0·0036	0·82
3	8-6-1923	10·7	5·9	10·0	22·7	73	0·0032	0·70

[1] Parry, *loc. cit.*, p. 308.
[2] By fractional distillation, carvone was found to be 27 per cent.
[3] The yield in this case has been calculated on the weight of the raw material, as the moisture-content was not known.

Table XXXIV gives the analytical constants for the Indian fennel oils.

TABLE XXXIV.

Analytical data for Indian fennel oils.

Number of distillation	1	2	3	Sweet fennel oil [1]	Indian fennel oil [2]
d_{15}^{15}	0·9744	0·9752	0·9767	0·964-0·976	0·968-0·973
n_D^{25}	1·5355	1·5375	1·5383	1·5280-1·5380	...
a_D^{25}	+ 16·9°	+ 12·1°	+ 11·7°	+ 6° to + 20°	+ 21°
Congealing point ...	+ 5·5°	+ 9·0°	+ 9·0°	+ 3° to + 10°	...
Solubility in volumes of 90 per cent. alcohol.	1 at 6°	1 at 10°	1 at 8°	I	...
Taste	Sweet	Sweet	Sweet	...	...

The results of a fractional distillation of 38 grams of fennel oil at 7 mm. pressure are given in Table XXXV.

TABLE XXXV.

Distillation of 38 grams of fennel oil (sample 3, Table XXXIII) at 7 mm pressure.

Number of fraction	Temperature in degrees centigrade	Weight of fraction as per cent. on oil taken	n_D^{25}	a_D^{25}	Melting point
1	54-75	9·7	1·4740	+58·1°	...
2	75-90	9·7	1·5025	+34·6°	...
3	90-100	7·0	1·5031	+9·5°	...
4	100-101	66·6	1·5560	Nil.	21°
	Residue and loss	7·0	...	...	...

[1] Parry, *loc. cit.*, p. 309.
[2] Umney, *Pharm. J.*, 1897, 58, 226.

Umney[1] gives the yield of oil from Indian fennel as 0·7 to 1·2 per cent. The constants for the Indian oil are within the limits for commercial sweet fennel oil. The results of fractional distillation in Table XXXV indicate that the oil contains over 70 per cent. of anethole. According to Parry [2] a good quality fennel oil contains as much as 60 per cent. anethole.

Fenchone.—This is the characteristic constituent of the oil of fennel. 3 grams of fraction 1, Table XXXV was mixed with 10 cc. of strong nitric acid and after twenty minutes two grams of unattacked oil (corresponding to 6 per cent. on the whole oil) separated. On being washed and dried, it had a camphoraceous odour and solidified at 5–6° when kept in ice. It was confirmed to be fenchone by the preparation of the oxime according to the method of Mahla and Tiemann.[3] The oxime on recrystallisation from alcohol melted at 164°.

Borneol as an ester.—To see if methylchavicol was present, fraction 2, Table XXXV was treated with alcoholic potash and the oil recovered was distilled. Anethole could not be found, making the presence of methylchavicol improbable in any large quantity. The fraction which boiled at 90–93° at 6 mm. pressure solidified, had a camphoraceous odour and on recrystallisation from petroleum ether melted at 203–204°. Borneol has not so far been identified in Fennel oil.

Table XXXVI gives the yields of essential oil, and the oil obtained per unit of steam for the materials that have been distilled. It shows the facility with which some of the oils such as cardamom and cubebs come over and the comparative difficulty of distilling oils like that from vetivert roots.

[1] *Loc. cit.*
[2] *Loc cit.*
[3] *Ber.*, 1896 **29**, 2818.

TABLE XXXVI.

Ratio, oil : steam, and yields of essential oils.

Number	Botanical name of the plant	Portion of the plant from which oil was distilled	Weight of oil in kilos	Weight of steam in kilos	Ratio, oil : steam	Yield of oil per cent.	Preparation of material
9	*Elettaria Cardamomum* ...	Fruits	12·024	167·3	0·0718	6·0	Whole fruits crushed
9	*Do.* ...	Do.	6·997	184·0	0·038	6·22	Whole fruits
12	*Zanthoxylum Rhetsa* ...	Carpels	0·325	13·5	0·024	5·80	Crushed
10	*Cubeba officinalis* ...	Fruits	0·539	41·0	0·0132	11·85	Do.
11	*Cinnamomum Camphora* ...	Stump	2·350	262·0	0·0090	5·80	Chips
7	*Zingiber officinale* ...	Rhizome	5·115	591·8	0·0086	3·54	Twice re-crushed during distillation
15	*Cuminum Cyminum* ...	Fruits	1·268	228·6	0·0055	2·35	Crushed
16	*Anethum Sowa* ...	Do.	1·738	356·3	0·0049	3·19	Do.
11	*Cinnamomum Camphora* ...	Leaves	0·710	163·4	0·0044	2·10	Nil
17	*Foeniculum Panmorium* ...	Fruits	0·092	26·0	0·0036	0·82	Crushed
1	*Callitris rhomboidea* ...	Leaves	0·254	163·6	0·0016	0·17	Nil
6	*Acorus Calamus* ...	Roots	0·273	174·7	0·0015	1·5	Dis-integrated
14	*Coriandrum sativum* ...	Fruits	0·156	105·5	0·0015	0·25	Crushed
8	*Curcuma Zedoaria* ...	Rhizome	0·389	397·0	0·00097	1·01	Dis-integrated
5	*Vetiveria zizanoides* ...	Roots	0·244	756·6	0·00032	0·62	Dis-integrated

SUMMARY.

Among the interesting points revealed by a study of the distillations and analyses recorded in this paper are the following:—

1. The leaves of *Callitris rhomboidea*, R. Br., grown on the Nilgiris give a comparatively high percentage of oil, approximating to that obtained from the allied species *C. Tasmanica.*

2. By distilling the oil from *Vetiveria zizanoides*, Stapf, under reduced pressure the distillate may be dext ro or laevo-rotatory, and is not necessarily laevo-rotatory as stated by Allen.

3. The oil from the calamus roots distilled in Bangalore is devoid of low-boiling constituents and differs from the commercial oils, more especially as regards density and solubility in alcohol and in these respects resembles the oil from Java.

4. The waste ginger from the West Coast gives exceptionally high yields of oil, but the distillation is slow.

5. Mysore cardamom fruits—pericarps and seeds—of good quality gave a 6 per cent. yield of high grade oil.

6. Although camphor wood gives up its oil much more readily when disintegrated than when in the form of chips, this method of treatment is not recommended as during disintegration appreciable amounts of camphor are lost.

The roots of the Bangalore tree gave a very high percentage of oil, viz., 7·9 and of this one-fourth was camphor.

The proportion of safrole in the oils, from the roots and the stump was small.

7. *Zanthoxylum Rhetsa* contains the volatile oil in the carpels only and not in the seeds. The yield is 5·8 per cent. of the weight of the dry carpels.

8. The yields of oil from the fruits of certain species of *Umbelliferae* grown in India, e.g., coriander and fennel are low when compared with the yield from European fruits. Whether this is due to climatic causes, to insufficient manuring or to variations in the plants is not certain.

9. The East Indian dill oil when freed from dill-apiol closely resembles the European oil.

10. The East Indian coriander oil has a higher percentage of esters and an odour superior to that of the European oil.

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