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TERPENOIDS OF CARDAMOM OIL AND THEIR COMPARATIVE DISTRIBUTION AMONG VARIETIES

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Abstract—The cold-pressed essential oils derived from three varieties of cardamom seeds and Oil of Cardamom, N.F. were investigated by a gas chromatographic procedure employing open tubular columns. The presence of α-pinene, sabinene, β-pinene, myrcene, α-terpinene, D-limonene, 1,8-cineole, methyl heptenone, γ-terpinene, *trans*-sabinene hydrate, linalool, β-terpineol, borneol, 4-terpinenol, α-terpineol, nerol, linalyl acetate, geraniol, 4-terpinenyl acetate, α-terpinyl acetate, neryl acetate, and nerolidol reported by earlier investigators was confirmed. In addition, the following previously unreported constituents were tentatively identified: camphene, α-phellandrene, camphor, citronellal, citral, citronellol, ascaridole, geranyl acetate, bisabolene, and farnesol. Identification of isolated fractions was accomplished by gas chromatographic retentions, peak enrichment techniques, and infrared spectroscopy. Qualitative and quantitative data on the composition of the oils examined are presented, and comparisons are drawn between the various oils.

INTRODUCTION

THE CARDAMOM oils are derived from the dried fruit of *Elettaria cardamomum* Maton. (Zingiberaceae) a large perennial herbaceous plant native to India.[1] The fruit consists of a three-celled capsule, each cell containing a number of seeds. A conventional method for the preparation of the essential oil is by steam distillation of the decorticated (i.e. capsule removed) seeds. This procedure produces a near colorless liquid with a spicy odor, reminiscent of eucalyptus.[2] Although an important spice known for centuries, little is known about its chemical composition. In the nineteenth century Dumas and Péligot[2] observed crystals of terpin hydrate in old cardamom oil. This material probably originated from α-terpineol, an alcohol identified some 60 years later. The oil from the variety *minuscula* Burkhill was further investigated by workers at Schimmel & Co.,[3] Parry,[4] Wallach[5] and Moudgil[6] who reported the presence of the following terpenoids: limonene,[4] sabinene,[6] cineole,[3] d-α-terpineol,[3] terpinyl acetate,[3] borneol,[6] and an unknown acid.[6]

The variety β-Major Thwaites was investigated by Weber[7] and Wallach[8] who reported the presence of sabinene,[8] terpinene,[7] 4-terpinenol,[7,8] 4-terpinenyl formate and acetate,[2] and a low melting solid compound.[2]

Considering the methods at hand in this period, these identifications were remarkable feats of chemistry. With the advent of GLC, there was renewed interest in the composition

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[1] J. W. PARRY, *Spices*, Vol. I, pp. 173–175, Chemical Publishing, New York (1969).
[2] E. GUENTHER, *The Essential Oils*, Vol. V, p. 96. Van Nostrand, New York (1952).
[3] BER. SCHIMMEL & Co., 9 October 1897.
[4] E. J. PARRY, *Pharm. J.* **63**, 105 (1899).
[5] O. E. WALLACH, *Liebigs Ann. Chem.* **360**, 90 (1908).
[6] K. L. MOUDGIL, *J. Soc. Chem. Ind.* **43**, 137T (1926).
[7] E. WEBER, *Liebigs Ann. Chem.* **238**, 98 (1887).
[8] O. E. WALLACH, *Liebigs Ann. Chem.* **350**, 168 (1906).

of many essential oils. In 1960 Ikeda *et al.*[9] examined the monoterpene hydrocarbons of 29 essential oils, and reported 20·7% of the oil of cardamom consists of monoterpene hydrocarbons. Using retention data, these workers identified them as: α-pinene, α-thujene, β-pinene, sabinene, α-terpinene, myrcene, D-limonene, γ-terpinene, and *p*-cymene. Nigam *et al.*[10] used serial dilution and i.r. spectra of isolates to reaffirm the presence of the constituents previously reported, with the exceptions of terpin hydrate, α-thujene, β-pinene, α-terpinene, and γ-terpinene. Additionally, the oil was found to contain the following previously unreported constituents: methyl heptenone, linalool, linalyl acetate, β-terpineol, geraniol, nerol, neryl acetate, nerolidol and heptacosane. These investigators tentatively characterized two ketones, 2-undecanone and 2-tridecanone. Unfortunately, this research[10] is marred by the absence of any clear statement as to which components were identified by i.r. spectral methods and which were only tentatively characterized from GLC data.

Lewis *et al.*[11] investigated three varieties of cardamom oils obtained by steam distillation of the seeds and reported their data clearly show that differences among the three oils were mainly quantitative. Their published chromatograms indicated 17 peaks.

More recently, Brennand and Heinz[12] investigating the effects of pH and temperature on the volatile constituents of cardamom confirmed the presence of many of the terpenoids previously indicated, but they were unable to detect α-thujene, β-pinene, α-terpinene, γ-terpinene, borneol, and the two ketones reported by Nigam *et al.*[10] They did isolate and identify the low melting solid previously reported by Guenther[2] as *trans*-sabinene hydrate. Using improved chromatographic procedures, Brennand and Heinz[12] were able to distinguish 31 chromatographic peaks for the cardamom oils they examined.

The present investigation reports the composition of the oils derived from three varieties of cardamom and a commercially produced oil, and presents quantitative and qualitative comparisons of these oils.

RESULTS AND DISCUSSION

Unfortunately, there are a number of designations for each major variety of cardamom. The varieties used in this study and their synonyms are:

I. Malabar type = *Elettaria cardamomum* Maton. var. *minuscula* Burkhill[2] = var. α-minor[2] = 'cv.' Malabar[2] = 'cv.' Malabar-Ceylon[2] = 'cv.' Mysore–Ceylon[2] = *E. repens* (Sonner.) Baill.[13]

II. Mysore type—also classified under the same botanical name(s) as above.[14]

III. Ceylon type = *Elettaria ensal* (Gaertn.) Abeywick.[13] = *E. cardamomum* Maton. var. β-Major Thwaites[2] = *E. major*[2] = 'cv.' Long wild cardamoms.[2]

The Malabar type is cultivated in the state of Kerala, India, in Guatemala and in Ceylon; the Mysore type is grown extensively in the state of Mysore, India. The Ceylon type is native to that country. The three types are quite distinctive and are readily distinguished from each other by the size, shape, and color of their capsules (Fig. 1). In the upper left

[9] R. M. IKEDA, W. L. STANLEY, S. H. VANNIER and E. M. SPITLER, *J. Food Sci.* **27**, 455 (1962).
[10] M. C. NIGAM, I. C. NIGAM, K. L. HANDA and L. LEVI, *J. Pharm. Sci.* **54**, 799 (1965).
[11] Y. S. LEWIS, E. S. NAMBUDIRI, and T. PHILIP, *Perfumery Essent. Oil Record* **57**, 623 (1966).
[12] C. P. BRENNAND and D. E. HEINZ, *J. Food Sci.* (in press).
[13] B. A. ABEYWICKREME, *Ceylon J. Sci. (Biol. Sci.)* **2**, 145 (1959).
[14] B. A. ABEYWICKREME, personal communication.

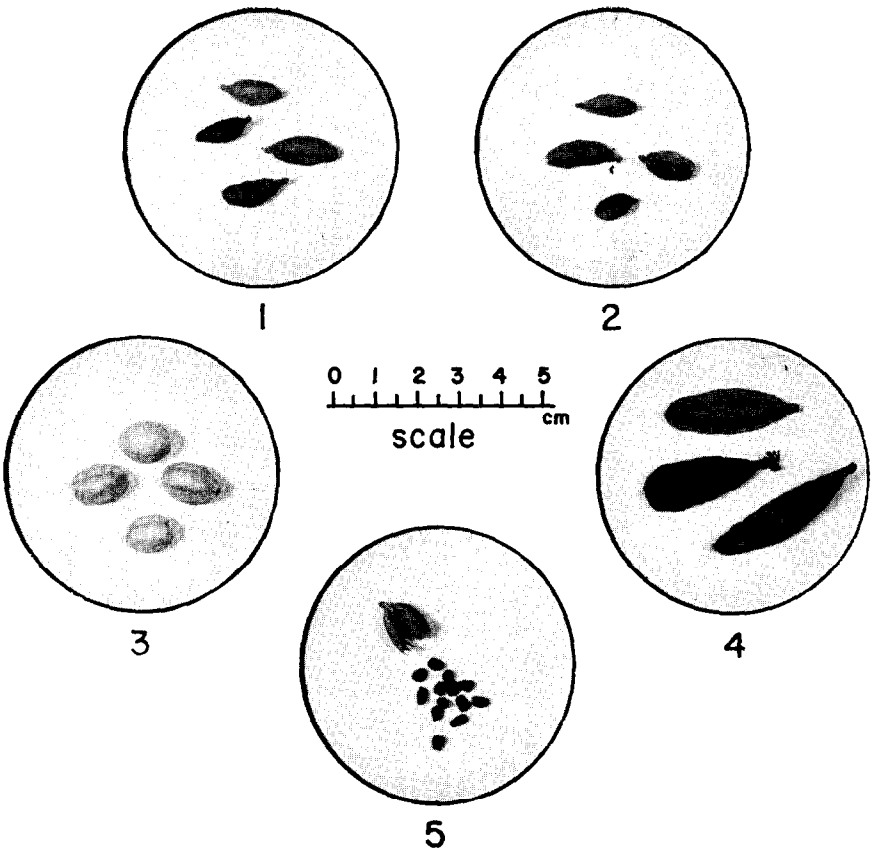

FIG. 1. CARDAMOM CAPSULES.

Circle 1, Malabar type, Ceylon. Circle 2, Malabar type, Guatemala. Circle 3, Mysore type. Circle 4, Ceylon type. Circle 5, Malabar type, Ceylon—decorticated.

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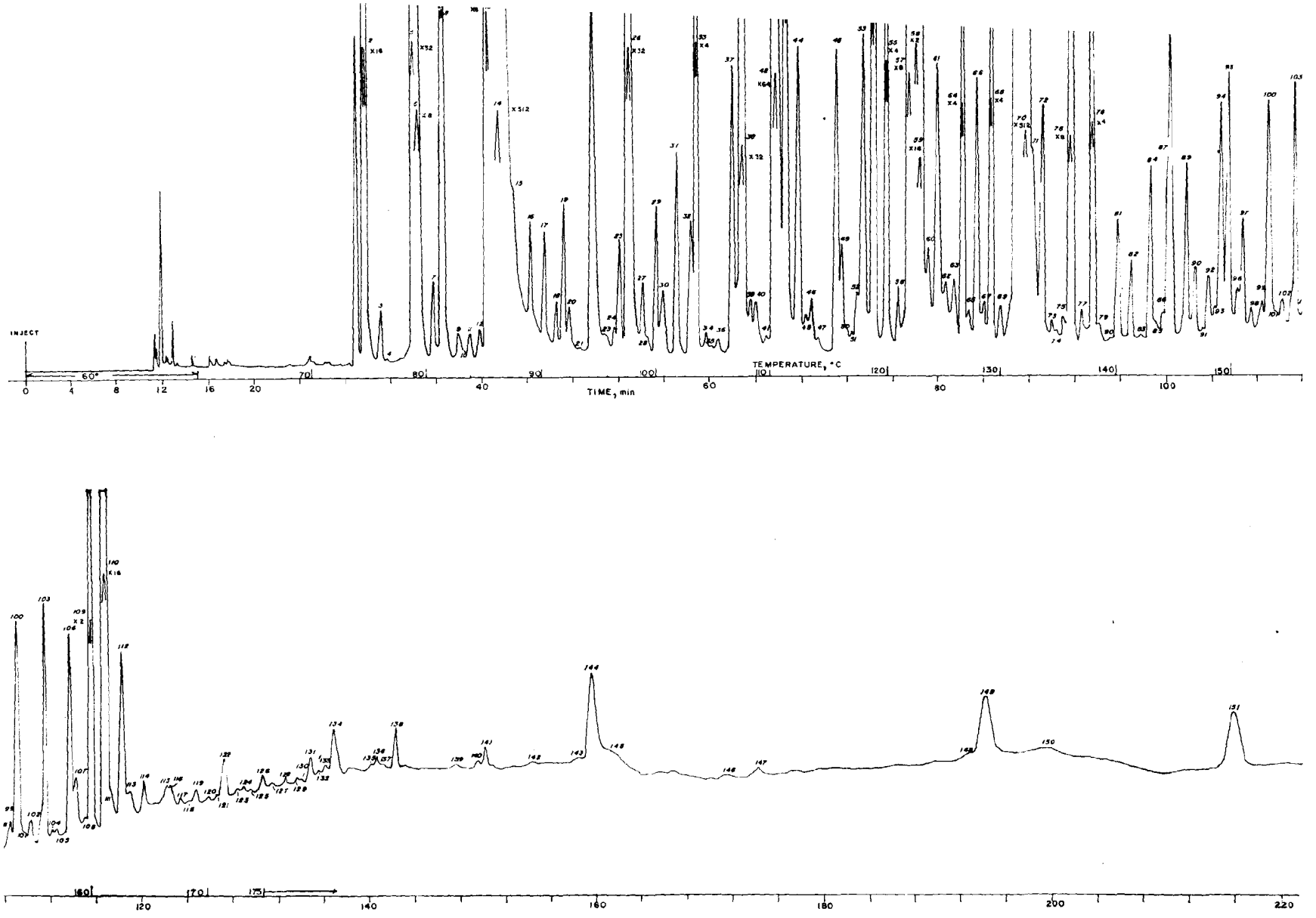

FIG. 2. GAS CHROMATOGRAM OF OIL OF CARDAMOM, N.F.
See Table 1 for peak identification and Table 3 for operating parameters.

(circle 1) are four capsules of the Malabar type. Capsules are 15–20 mm in length, three-sided, and grey-green in color. This sample was cultivated in Ceylon. Capsules of a second sample of this type (circle 2) grown in Guatamala are of similar size and shape, but yellow-brown in color. Capsules of the Mysore type (circle 3) are thicker and shorter (10–15 mm long) than the former, and are light tan. The capsules of the Ceylon type (circle 4) are thicker and larger than the others, being 30–40 mm long. The exterior color is a deep grey-brown. Seeds of the Malabar type (circle 5) are 3–4 mm long and of a rich, brown color.

Preliminary investigations were conducted in order to determine the most suitable stationary liquid phase for the separation of the constituents of cardamom oil and the optimum operational parameters for the chromatographic procedures. For example, an open tubular column coated with OV-101** resolved the commercial oil sample into 75 individual peaks, while SF-96†† gave twice as many peaks. Accordingly, SF-96 was used for all of the comparative determinations. A typical chromatogram is presented in Fig. 2.

Peak identification of many of the major components of cardamom oil is presented in Table 1. The identifications confirmed by i.r. spectral analysis are indicated. Identification

TABLE 1. CONSTITUENTS IDENTIFIED IN CARDAMOM OILS

Peak No. (see Fig. 2)	Constituent	Confirmed Identity*	Reference†
2	α-Pinene	RD, PE, i.r.	9–12
3	Camphene	RD	
5	Sabinene	RD, i.r.	6,8–12
6	β-Pinene	RD	9
8	Myrcene; α-terpinene	RD, PE	9–12
9	α-Phellandrene	RD	
13	D-Limonene	RD, i.r.	4,9–12
14	1,8-Cineole	RD, i.r.	3,10–12
15	Methylheptenone	RD	10,11
17	γ-Terpinene	RD, i.r.	7,9
19	*trans*-Sabinene hydrate	RD	2‡,12
26	Linalool	RD, i.r.	10–12
29	DL-Camphor	RD	
33	β-Terpineol; citronellal	RD, PE	10,11
34	DL-Borneol	RD, PE	6,10,11
38	4-Terpinenol	RD, i.r.	7,8,12
42	α-Terpineol	RD, i.r.	3,10–12
48	*trans*-Citral	RD	
52	Citronellol	RD	
53	Nerol	RD, PE	10–12
57	Geraniol	RD, i.r.	10,11
61	4-Terpinenyl acetate	RD, i.r.	2
62	Ascaridole	RD	
70	α-Terpinyl acetate	RD, i.r.	3,10–12
72	Neryl acetate	RD, i.r.	10–12
76	Geranyl acetate	RD, i.r.	
106	Bisabolene; *trans*-nerolidol	RD	
110	*cis*-Nerolidol	RD	10–12
134	Farnesol	RD	

* Key: RD, GLC retention data; PE, peak enhancement; i.r. infrared spectrum.
† Compounds previously reported in the literature. See references in text.
‡ Reported in Guenther[2] as an unknown solid, m.p. 60–61°.

** OV-101 is a dimethyl silicone of approx. mol. wt. 30,000 and a viscosity of *ca.* 1200 cs.
†† SF-96 is also a dimethyl silicone of unspecified mol. wt. and a viscosity of *ca.* 50 cs.

of the remaining peaks listed in Table 1 can only be considered tentative as these identities are based upon retention data and peak enrichment techniques.[15]

With the exception of the commercially produced cardamom oil which was prepared by steam distillation of seeds, the oils examined in this study were prepared by a cold-pressing procedure. We chose this method so as to avoid subjecting the seeds and oils to prolonged heating which could lead to the possible loss, oxidation and rearrangement of the terpenoids. Capsules were decorticated immediately prior to use to avoid oxidation and loss of volatile constituents.[16] Expression of the seeds gave satisfactory yields of the oils (*ca.* 5%) which were generally clear and free of debris.

Our results are in accord with previous investigations[3,10-12] indicating that the two major constituents of the Oil of Cardamom, N.F. were 1,8-cineole and α-terpinyl acetate. The oils obtained by cold-pressing seeds of both the Malabar and the Mysore types of cardamom also showed this preponderance of these two terpenoids.

TABLE 2. APPROXIMATE % COMPOSITION OF CARDAMOM OILS*

Peak No. (see Fig. 2)	Component	Oil of Cardamom, N.F.	Malabar type (Ceylon)	Malabar type (Guatemala)	Mysore type	Ceylon type
2	α-Pinene	0·59	1·1	0·71	1·4	13·0
3	Camphene	0·01	0·02	0·03	0·04	0·13
5	Sabinene	1·2	2·5	3·4	3·1	4·9
6	β-Pinene	0·19	0·20	0·34	0·26	4·9
8	Myrcene; α-Terpinene	0·37	1·8	1·5	1·1	2·5
9	α-Phellandrene	<0·01	<0·01	0·01	<0·01	0·42
13	D-Limonene	0·28	0·02	0·12	0·14	2·1
14	1,8-Cineole	51·3	31·0	23·4	44·0	3·3
15	Methylheptenone	0·07	0·04	0·09	0·05	4·1
17	γ-Terpinene	0·03	0·04	0·07	0·07	11·2
19	*trans*-Sabinene hydrate	0·03	0·12	0·34	0·10	22·2
26	Linalool	1·4	2·1	4·5	3·0	3·7
29	DL-Camphor	0·03	<0·01	0·01	<0·01	0·35
33	α-Terpineol; citronellal	0·13	<0·01	0·04	0·06	0·13
34	DL-Borneol	<0·01	<0·01	0·03	<0·01	<0·01
38	4-Terpinenol	0·83	0·14	0·28	0·87	15·3
42	α-Terpineol	3·3	1·4	1·9	1·5	0·86
48	*trans*-Citral	0·06	0·09	0·08	0·07	0·48
52	Citronellol	0·02	<0·01	0·04	<0·01	0·01
53	Nerol	0·07	0·02	0·04	0·06	0·78
54	Linalyl acetate	0·74	3·3	6·3	3·1	0·31
55	*cis*-Citral	0·13	0·16	0·15	0·19	1·3
57	Geraniol	0·57	0·27	0·38	0·25	0·34
61	4-Terpinyl acetate	0·09	<0·01	0·02	<0·01	1·7
62	Ascaridole	0·03	0·02	0·02	0·01	0·05
70	α-Terpinyl acetate	34·6	52·5	50·7	37·0	0·14
72	Neryl acetate	0·05	0·09	0·09	0·02	0·03
76	Geranyl acetate	0·18	0·08	0·13	0·15	1·5
106	Bisabolene; *trans*-nerolidol	0·05	0·09	0·83	0·07	0·44
110	*cis*-Nerolidol	0·65	0·23	1·6	0·28	0·37
134	Farnesol	0·01	0·01	0·06	0·02	0·05
	Unidentified	3·00	2·80	2·79	3·09	3·41

* Based upon integration of peak areas. Values are medians of four replicates.

[15] R. A. BERNHARD and B. SCRUBIS, *J. Chromatog.* **5**, 137 (1961).
[16] J. F. CLEVENGER, *J. Assoc. Offic. Agri. Chemists* **17**, 283 (1934).

The cold-pressed oils from the Malabar varieties from Ceylon and Guatemala gave rather similar chromatograms, but quantitative differences in the distribution of constituents existed (Table 2). The content of α-pinene, 1,8-cineole, and α-terpinyl acetate was significantly lower in the oil from Guatemala, while this variety contained greater amounts of sabinene, linalool, α-terpineol, linalyl acetate, and nerolidol than did the sample from Ceylon. Chromatograms of both oil samples differed considerably from Oil of Cardamon, N.F. in that they were lower in 1,8-cineole content, and higher in α-terpinyl acetate content. Additionally, Oil of Cardamom, N.F. contained less sabinene, myrcene, α-terpinene, *trans*-sabinene hydrate, linalool, and linalyl acetate, than did either of the two Malabar type samples examined. The commercial oil had a higher content of D-limonene, β-terpineol, citronellal, 4-terpinenol, α-terpineol, geraniol, and 4-terpinenyl acetate than did either of the Malabar oils.

In the area from peak 27 to peak 38 (Fig. 2), component peaks were generally smaller in the Malabar type oils than in the commercially produced oil examined.

The Mysore type oil was significantly higher in the amounts of α-pinene, sabinene, β-pinene, myrcene, α-terpinene, linalool, and linalyl acetate than were present in the commercial oil. The 1,8-cineole content was lower than in the commercial oil, as was the α-terpineol content. The quantity of α-terpinyl acetate was of the same magnitude in the two oils. The Malabar oils appeared to differ most significantly from the Mysore oil in the content of myrcene, α-terpinene, 4-terpinenol, 1,8-cineole and α-terpinyl acetate (Table 2).

Examination of the Ceylon type oil revealed that both 1,8-cineole and α-terpinyl acetate were present in relatively small amounts (Table 2). The Ceylon type showed major amounts of α-pinene, sabinene, β-pinene, myrcene, α-terpinene, D-limonene, methyl heptenone, γ-terpinene, *trans*-sabinene hydrate, 4-terpinenol, the citrals, 4-terpinenyl acetate, and geranyl acetate. Two terpene alcohols, yet to be identified, (peaks 25 and 46, Fig. 2) also appeared to be present in this oil in significant amounts, i.e. of the order of 1–2% each. All of our attempts to purify these alcohols by preparative GLC were unsuccessful, for they seemed to undergo chemical rearrangement on all of the columns tested. The relatively large amount of *trans*-sabinene hydrate reported for this oil (Table 2) may be due, at least in part, to the presence of a significant quantity of an unknown component, peak 20 (Fig. 2).

The Ceylon type oil differed materially in its quantitative distribution of components from all of the other oils examined in this study (Table 2). Our findings differ from those of Lewis *et al.*[11] who reported a high content of 1,8-cineole (36%) and α-terpinyl acetate (30%) in the Ceylon type oil they examined. In fact, their proximate analysis is similar for all three varieties examined in their study. It is conceivable that the variety of cardamom analyzed by Lewis *et al.*[11] was not truly the giant Ceylon type, but a sample of the regular variety commonly cultivated on the island, for our capsules appear to be larger than those pictured in the publication of Lewis *et al.*[11] Our specimen of the Ceylon type was collected in Ceylon and authenticated by Mr. N. Kumarage, Department of Agrarian Services, Ceylon.

Ikeda *et al.*[9] have reported that the terpene hydrocarbon content of commercial cardamom oil (of unspecified origin) amounts to 20·7 per cent. They found large amounts of α-pinene, sabinene, myrcene, and D-limonene in this fraction. Our results indicate that the terpene hydrocarbon content of the commercial sample, Oil of Cardamom, N.F., and both the Malabar and the Mysore type oils examined were significantly lower than that of Ikeda *et al.* The content of known terpenes in the Oil of Cardamom, N.F. amounts to

2·62%. Assuming some of the unidentified fraction contains additional terpene hydrocarbons, this could reach a maximum of not more than 5·68%. The content of known terpene hydrocarbons in the Malabar type from Ceylon was 5·59% with a maximum of 8·53% possible; while these figures for the Malabar type from Guatemala were 6·18 and 9·04%, and those of the Mysore type were 6·08 and 9·19%. The oil from the Ceylon type presents a vastly different picture. Here the known terpene content adds to 39·04% with a possible maximum of 42·59%. These figures indicate that the sample of commercial oil did contain a significantly lower proportion of terpene hydrocarbons than did the other oils examined in this study. Whether this was due to the method of preparation, *viz.* distillation *vs* cold-pressing, or not remains to be determined.

It seems reasonable to suggest that the differences between quantitative results obtained by earlier investigators and those reported in the present study can be accounted for, at least in part, by our experimental procedures and methods of analysis; our increased resolution of components gave a far greater number of individual peaks than have been previously reported.

We were unable to verify the presence of terpin hydrate,[2] α-thujene,[9] *p*-cymene,[9-11] 2-undecanone,[10] 2-tridecanone,[10] and heptacosane[10] in any of the oils examined. The absence of *p*-cymene may be a consequence of our method of oil preparation. Ikeda *et al.*[17] have shown that the presence of *p*-cymene in lemon oils arises from oxidation of γ-terpinene. They also demonstrated that in a matter of 3–5 days appreciable amounts of *p*-cymene can be formed from pure γ-terpinene. Thus oils analyzed immediately after preparation should be relatively free of *p*-cymene as an artifact. One may further speculate that the sample examined by Ikeda *et al.*[17] could well have been a blend containing a considerable quantity of the Ceylon type oil.

On the basis of our analyses, we conclude, as did Lewis *et al.*,[11] that the application of gas chromatographic procedures to distinguish between different cardamom oils must be conducted with great caution, for the apparent differences are more quantitative than qualitative in nature.

EXPERIMENTAL

Source of Materials

Authentic varieties of cardamom capsules were obtained from the following sources: Malabar and Mysore types grown in Ceylon from Mr. U. K. Percy Silva, Colombo, Ceylon; Malabar type grown in Guatemala from Dr. William H. Stahl, McCormick and Co., Inc., Cockeysville, Md., U.S.A.; Ceylon type from Mr. N. Kumarage, Department of Agrarian Services, Ceylon.

Preparation of Oils

The essential oils used in this study were prepared by a cold-pressing procedure. Capsules were decorticated just prior to use to prevent oxidation and loss of volatiles,[16] and the freshly decorticated seeds were placed in a specially constructed stainless steel press. This press consisted of a cylinder 90 mm dia. and 110 mm in length. A hole 26 × 67 mm was bored in the center of the cylinder. The bottom of the hole was tapered at a 45° angle. A piston was fabricated so as to fit snugly into this hole with an equivalent taper at the bottom. The interior surface of the hole and exterior surface of the piston were highly polished to permit close contact and smooth vertical movement of the piston in the hole. An oil groove was cut at the top of the hole on the upper cylinder surface to permit recovery of the cold-pressed oil.

In a typical experiment, 13·5 g of decorticated seeds yielded 700 mg of oil when a pressure of 10–12 tons (20,000–24,000 psi) was applied to the top of the piston. The expressed oil filled the groove atop the cylinder and could readily be removed by means of a micro-syringe. The oils thus obtained were clear and appeared devoid of any particles. To remove any possible traces of solids, the oil was centrifuged at 4200 rev/min for 5 min, immediately placed in glass vials, sealed, and refrigerated at −20° until examined.

[17] R. M. IKEDA, W. L. STANLEY, S. H. VANNIER and L. A. ROLLE, *Food Tech.* **15**, 379 (1961).

For purposes of comparison, a sample of commercially prepared Oil of Cardamon, N.F. obtained from F. Ritter and Co., Los Angeles, Calif., U.S.A. was also examined. Portions of the oil samples were subsequently deterpenated as described previously[18] for examination of the terpene hydrocarbon and oxygenated fractions.

Gas Chromatographic Procedures

The composition of the volatile components of the oils was determined by a sensitive gas chromatographic procedure. Four replicate analyses were made for each oil examined and the median value of the quantitative data presented in Table 2. An open tubular column (152·4 m × 0·0762 cm) was used for all analytical examinations. Where it was possible to isolate constituents for subsequent spectral analysis, a preparative column was employed. The operating conditions of the two gas chromatographs are presented in Tables 3 and 4.

TABLE 3. OPERATING CONDITIONS FOR AEROGRAPH MODULINE MODEL 1860–4* GAS CHROMATOGRAPH FOR GLC OF TERPENOIDS FROM CARDAMOM OILS

Stationary liquid phase:	SF-96†
Column length:	152·4 m‡
Column, inside dia.	0·0762 cm‡
Column temp.:	
Initial:	60°
Final:	175°
Program rate:	1°/min§
Injection block temp:	175°
Detector oven temp.:	185°
Detector:	Flame ionization
Carrier gas velocity, N_2:	25 cm/sec‖
Make up gas flow rate, N_2:	18 ml/min
Hydrogen flow rate:	25 ml/min
Air flow rate:	250 ml/min
Recorder range	1 mv
Chart speed	15 in/hr
Electrometer range	2×10^{-11} amp full scale
Sample size	0·02–0·03 μl

* Varian Aerograph, Walnut Creek, Calif., U.S.A.
† Methyl silicone fluid, General Electric Company, Schenectady, N.Y., U.S.A.
‡ Open tubular column, stainless steel type 316.
§ An initial delay of 15 min at 60° was used prior to start of temperature program.
‖ Corresponds to a flow rate of 7·5 ml/min.

Component Identification

Various peak identities were assigned by determination of the corrected retention volumes, V°_R,[19] of known terpenoids and comparison with those for the unknown peaks. Peak identity was further confirmed by an enrichment procedure in which known terpenoids were added one at a time to fresh portions of the oils and re-examined by GLC using the open tubular column. For each of the above procedures to be well within the bounds of experimental error, an arbitrary limit of agreement for relative corrected retention volumes $V^\circ_R/V^\circ_{R_n}$ and coincidence in peak enhancement not to exceed 1% was established.[20] Compounds with values of $V^\circ_R/V^\circ_{R_n}$ not agreeing to within 1% are not presented in Table 1.

The third method of peak identification was by i.r. spectrophotometry. Fractions eluted from the preparative gas chromatograph were trapped in thin-walled glass capillary tubes as described by Jennings *et al.*[21] Components extrapped in this fashion were flushed from the tubes using spectro grade CH_2Cl_2 or *n*-hexane and deposited on "Mini-Cells" of AgCl (Wilks Scientific Corp., South Norwalk, Conn., U.S.A.). The solvent was allowed to evaporate and the cover cell placed over the material to form a conventional

[18] J. R. CLARK and R. A. BERNHARD, *Food Res.* **25**, 389 (1960).
[19] D. AMBROSE, A. I. M. KEULEMANS and L. H. PURNELL, *Anal. Chem.* **30**, 1582 (1958).
[20] R. A. BERNHARD, *Anal. Chem.* **34**, 1576 (1962).
[21] W. G. JENNINGS, R. K. CREVELING and D. E. HEINZ, *J. Food Sci.* **29**, 730 (1964).

TABLE 4. OPERATING CONDITIONS FOR AEROGRAPH MODULINE MODEL 1720–10* GAS CHROMATOGRAPH FOR GLC OF TERPENOIDS FROM CARDAMOM OILS

Stationary phase:	30% (w/w) LAC-2-R446*,†
Solid support:	Firebrick (acid washed) 100/120 mesh‡
Column length:	4·88 m (stainless steel)
Column diameter:	outer 0·635 cm; inner 0·476 cm
Column temperature:	150°
Injection block temperature:	180°
Detector oven temperature:	225°
Detector type:	Thermal conductivity
Detector current:	150 ma
Carrier gas flow rate, He:	50 ml/min
Recorder range:	1 mv
Chart speed:	30 in/hr
Sample size:	10–100 μl

* Varian Aerograph, Walnut Creek, Calif., U.S.A.
† The adipate polyester of diethylene glycol partially cross-linked with pentaerythritol. Cambridge Industries Co., Watertown, Mass., U.S.A.
‡ U.S. Standard Screen.

smear of capillary path length. I.r. spectra were obtained using a Perkin-Elmer Model 457 grating spectrophotometer. Identification of the constituents (Table 1) was made by matching i.r. spectra of authenticated reference standards with those of the unknown components.

Quantitative Analysis

Peak areas were determined by multiplying peak height by the width at half-height.[22] To improve precision, all peak measurements were made using a 50·0 cm machine ruled scale (Paragon No. 563278, Keuffel and Esser Co., New York, N.Y., U.S.A.) and observation with a 5X magnifying viewer.

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[22] E. CREMER and R. MÜLLER, *Mikrochem. Acta.* **36/37**, 553 (1951).