magnetic stirring. The vigorous evolution of trimethylamine gas from the reaction mixture was observed during the early part of the reaction. The reaction mixture was then brought to dryness under reduced pressure. The dried residue was triturated with 50 ml. of water and 200 ml. of ether. The aqueous layer was washed with 100 ml. of ether. The ethereal layer was combined with the washing, washed with water, dried over anhydrous sodium sulfate, and evaporated to dryness to leave a 0.75-Gm. yellow oil. The des-*N*-methine crystallized from ether as colorless fine prisms (0.64 Gm.), m.p. 170–172°, $[\alpha]_D^{25} \pm 0°$ (c 2.05, chloroform); $\lambda_{max.}$ 267 mμ (ε 55,000), 317 mμ (ε 31,000); N.M.R. spectrum ($CDCl_3$), τ = 5.76, 5.83, 5.88, 5.91, 6.05, 6.07, 6.19 (21H, O-methyl).

Anal.—Calcd. for $C_{39}H_{38}O_8$: C, 73.80; H, 6.04. Found: C, 73.54; H, 6.20.

PHARMACOLOGICAL RESULTS[2]

Thalicarpine was evaluated in a variety of pharmacologic procedures and found to possess a modicum of biological activity. In acute dose range or toxicity studies, oral doses of 300 mg./Kg. failed to produce discernible gross behavioral changes in the mouse. Intraperitoneal injection of 25 mg./Kg. of thalicarpine to a cat produced sensitivity of the forepaws, rubbing of the neck, and emesis.

The principal action of thalicarpine on blood pressure was depressor in nature. In the cat anesthetized with chloralose, mean arterial blood pressure was lowered transiently following acute intravenous doses ranging from 0.5 to 5 mg./Kg. Lethality, due to respiratory arrest, occurred at a dose of 10 mg./Kg. Bradycardia, respiratory depression, and andrenergic blocking action accounted for the weak hypotensive activity. Anticoagulant, hypoglycemic, and anticonvulsant properties were not observed after oral doses of 100 mg./Kg. in the rat, guinea pig, or mouse, respectively. An oral dose of 50 mg./Kg. caused a slight antidiuretic response in rats hydrated with saline. In the Randall and Selitto test for anti-inflammatory activity oral doses of 50 mg./Kg. of thalicarpine produced a very low order of analgetic activity (25%) and no significant antipyretic action.

In summary, weak hypotensive activity of a transient nature was the principal action of thalicarpine when injected intravenously into the anesthetized cat. Respiratory toxicity and weak adrenolytic activity accompanied this action. Thalicarpine failed to exhibit significant biological activity as an anti-inflammatory, anticoagulant, hypoglycemic, or diuretic agent.

REFERENCES

(1) Chopra, R. N., Nayar, S. L., and Chopra, I. C., "Glossary of Indian Medicinal Plants," Council of Scientific and Industrial Research, New Delhi, India, 1956, p. 243.
(2) Sekiguchi, H., *J. Pharm. Soc. Japan*, **80**, 759(1960).
(3) Ossadcha-Janata, N., "Herbs Used in Ukrainian Folk Medicine," Research Program on the U.S.S.R., New York Botanical Garden, 1952, pp. 14–16.
(4) Train, P., Henrichs, J. R., and Archer, W. A., "Medicinal Uses of Plants by Indian Tribes of Nevada," Contributions Toward a Flora of Nevada, No. 45, U. S. Department of Agriculture, rev. ed., 1957, p. 97.
(5) Watt, J. M., and Breyer-Brandwijk, M. G., "Medicinal and Poisonous Plants of Southern and Eastern Africa," 2nd ed., E. and S. Livingstone, London, 1962, p. 881.
(6) Ousepyan, A. M., *Izv. Akad. Nauk Arm. SSR*, **9**, 57–66 (1956); through *Chem. Abstr.*, **50**, 14,111(1956).
(7) Hogg, R., Beal, J. L., and Cava, M. P., *Lloydia*, **24**, 45(1961).

Gas Chromatographic Analysis of Oil of Nutmeg

By ELWINA A. BEJNAROWICZ and ERNST R. KIRCH

Four samples of commercially available oil of nutmeg were analyzed by gas chromatography. Of a number of stationary liquid phases used, a 20 per cent Reoplex 400 on Dichromite gave the best separation. The composition of the oils was determined on the basis of retention times and enrichment.

ESSENTIAL OILS contain volatile compounds representing many classes of organic substances. One such volatile oil is the well known oil of nutmeg (*Myristica*), an important spice used for the flavoring of numerous food products. It is also used as a component of certain types of perfumes and as a flavoring agent for dentifrices (1). The literature lists two oils of myristica—oil of nutmeg and oil of mace. Both are derived from the fruit of *Myristica fragrans* Houtt. (*fam. Myristicaceae*) (2).

The dried seeds of nutmeg contain from 5 to 15% of the volatile oil, as well as from 25 to 40% of a fixed oil, and from 5 to 15% of ash. The rest consists of moisture, fiber, and starch (3, 4).

There are two principal types of nutmeg which are recognized today, and these depend primarily on geographical origin. "Banda nutmegs" or East Indian variety are the finest; the other variety comes from the West Indies (5).

The West Indian type of oil has a lower specific gravity, lower refractive index, and a lower residue on evaporation, but has a higher

Received November 15, 1962, from the Department of Chemistry, College of Pharmacy, University of Illinois at the Medical Center, Chicago.
Accepted for publication March 29, 1963.
Abstracted in part from a thesis presented by E. A. Bejnarowicz to the Graduate College, University of Illinois at the Medical Center, in partial fulfillment of Master of Science degree requirements.

optical rotation than the oil obtained from the East Indies.

Nutmeg oil consists principally of terpenes, and about 60% of the oil distils below 180° (6). In 1907, Power and Salway (7) examined a "genuine" oil of Ceylon nutmeg having a specific gravity of 0.8690 at 15° and an optical rotation of 38°4′. They found this oil to contain primarily over 80% of terpenes and 6% terpene alcohols.

In reviewing the literature concerning differences between East Indian and West Indian oil, one finds statements primarily referring to the higher content of terpenes and a lower content of phenols in the West Indian oil. Therefore, it was of interest to undertake an analysis of the nutmeg oil obtained from these two sources, using gas chromatography.

EXPERIMENTAL

Apparatus.—The apparatus employed was Beckman GC-2 gas chromatograph with a four-filament thermal conductivity cell connected to Sargent SR recorder equipped with a K-4 disk integrator. Helium was used as the carrier gas, and the flow rate was maintained at 38 ml. per minute. The filament current used was 380 ma., while the chart speed of the recorder was 1.0 in. per minute.

Column Preparation.—Reoplex 400 (polyethylene glycol adipate) was used as the stationary liquid phase and was supported on 80/100-mesh Diachromite. Approximately 20.0 Gm. of the solid support was required to pack a 6-ft. column. The 5-Gm. liquid phase was introduced by deposition gradually and with constant stirring from a 45 ml. solution in dichloromethane. The resulting mixture was first dried in air, then at 90° in the oven for about 3 hours. Columns used were of copper tubing, 1/4 in. diam. and 6 ft. in length.

Operating Conditions.—Temperatures ranging from 100 to 190° were employed. Samples of 0.005 ml. were used, except in cases where the fraction was collected. Then the sample was increased to 0.05 ml.

Oil Samples and Standard Compounds.—The four commercially available samples of nutmeg oil that were analyzed are listed below along with their respective specific gravities and refractive indices.[1] Sample A (East Indian nutmeg oil U.S.P. extra) n_D^{20}, 1.4804; sp. gr.^{24}, 0.884. Sample B (East Indian nutmeg oil U.S.P.) n_D^{20}, 1.4809; sp. gr.^{24}, 0.897. Sample C (East Indian nutmeg oil U.S.P.) n_D^{20}, 1.4793; sp. gr.^{24}, 0.880. Sample D (West Indian nutmeg oil U.S.P.) n_D^{20}, 1.4756; sp. gr.^{24}, 0.870.

For purposes of identification the retention times and relative retention times of the unknown were compared with a known pure standard sample. In this way a tentative identification of most of the compounds could be made. We have confirmed these particular results by enrichment method, in which known compounds were added individually to the sample chromatographed and compared in each instance to the chromatogram obtained by using the oil alone. Infrared spectra were obtained for unknown peaks. Tables I, II, and III list the retention times and relative retention times of standard compounds at 100 and 160°, respectively. Relative retention times are based on *p*-cymene.

[1] The authors express their thanks to Fritzsche Brothers, Inc., New York, N. Y., for supplying Sample C of oil of nutmeg.

TABLE I.—RETENTION TIMES AND RELATIVE RETENTION TIMES OF STANDARD COMPOUNDS ON REOPLEX 400 AT 100°C. (FLOW RATE, 38 ML./MIN.; FILAMENT CURRENT, 380 MA.; *p*-CYMENE = 1.00)

Standard Compd.	Retention Times, min.	Relative Retention Times
dl-α-Pinene	3.36	0.25
d-Camphene	4.56	0.34
β-Pinene	5.56	0.41
Terpinolene	7.56	0.56
d-Limonene	8.80	0.65
p-Cymene	13.5	1.00
dl-Linalool	52.4	3.88
Camphor	54.0	3.99
dl-α-Terpineol	129	9.51

TABLE II.—RETENTION TIMES AND RELATIVE RETENTION TIMES OF STANDARD COMPOUNDS ON REOPLEX 400 AT 130°C. (FLOW RATE, 38 ML./MIN.; FILAMENT CURRENT, 380 MA.; *p*-CYMENE = 1.00)

Standard Compd.	Retention Times, min.	Relative Retention Times
dl-α-Pinene	1.89	0.33
d-Camphene	1.95	0.34
β-Pinene	3.02	0.51
Terpinolene	3.69	0.65
d-Limonene	4.24	0.72
p-Cymene	5.70	1.00
dl-Linalool	16.6	2.90
Camphor	19.2	3.37
dl-Borneol	28.9	5.06
dl-α-Terpineol	34.7	6.09
Geraniol	67.3	11.8
Safrole	80.7	14.2
Eugenol (was not eluted even after 125 min.)		

TABLE III.—RETENTION TIMES AND RELATIVE RETENTION TIMES OF STANDARD COMPOUNDS ON REOPLEX 400 AT 160°C. (FLOW RATE, 38 ML./MIN.; FILAMENT CURRENT = 380 MA.; *p*-CYMENE = 1.00)

Standard Compd.	Retention Times, min.	Relative Retention Times
dl-α-Pinene	1.32	0.42
d-Camphene	1.32	0.42
β-Pinene	1.88	0.59
Terpinolene	2.22	0.70
Limonene	2.44	0.78
p-Cymene	3.18	1.00
dl-Linalool	7.00	2.20
Camphor	9.53	3.00
dl-α-Terpineol	14.6	4.58
Geraniol	24.9	7.55
Safrole	30.5	9.62
Eugenol	83.0	26.1
Isoeugenol	146	46.1

RESULTS AND DISCUSSION

Three commercial samples of an East Indian nutmeg oil and one sample of a West Indian nutmeg oil were analyzed by gas liquid partition chromatography. Of a number of stationary liquid phases that were used, only Reoplex 400 supported on 80/100-mesh Dichromite gave satisfactory results.

That the various components of the nutmeg oils could be separated more efficiently at different temperatures is shown in Figs. 1–3 when Sample A of oil of nutmeg was chromatographed at 100, 130, and 160°, respectively. For example, eight peaks were observed at 100° (Fig. 1), while at 130° the number of peaks increased to ten (Fig. 2). At 160°, two additional peaks were obtained using this same oil (Fig. 3). Similar results were observed with the other samples of the East Indian variety oil.

Attention should be directed to Fig. 2 which shows ten peaks obtained with Sample A at 130°. Comparing this with the chromatograms representing the peaks obtained at 100° (Fig. 1) and 160° (Fig. 3), respectively—and using this same oil—it should be pointed out that in the chromatogram obtained at 100°, peak numbers 8, 9, 10, 11, and 12 (Fig. 3), representing higher retention times, are absent. It was further observed that peak number 3 at 130° (Fig. 2) or 160° (Fig. 3) could be resolved into two components at 100°. One of these peaks (No. 3) we have identified as limonene.

We have used the results obtained at 100 and 160°, respectively, to calculate the percentages of the components of the East Indian oils (Samples A, B, and C). With the West Indian oil (Sample D) two unidentified components which appeared as two distinct small peaks at 130° (Fig. 5) but only as one peak at 160° (Fig. 6) were observed. At temperature of 100° (Fig. 4) these same constituents also appeared as two peaks, but one of them (with the lower retention time) could not be separated distinctly from the preceding one. The results obtained at 130° were also used to calculate the percentages of these two components of the oil. We have based all the percentages as calculated on the total eluted. To keep the peaks within the limits, it is customary to change attenuation at times from component to component. This was done in this investigation also. It is well recognized, however, that a particular area will differ with change in attenuation. Since the percentages are calculated from the integrated signals, and since these signals are proportional to the attenuation, each sample of the oil was re-run at one and the same attenuation.

The samples of the East Indian oil of nutmeg (Samples A, B, and C) that were examined differed in composition not only from the West Indian oil, but they also showed variation in composition from one to the other (Table IV).

All of the oils of the East as well as the West Indian variety were found to contain α-pinene, β-pinene, and limonene. Of the four samples of oil that we have investigated, the West Indian variety contained the highest total percentage (40.1%) of these terpenes, while the East Indian Samples A, B, and C contained 30.9, 30.2, and 33.9-

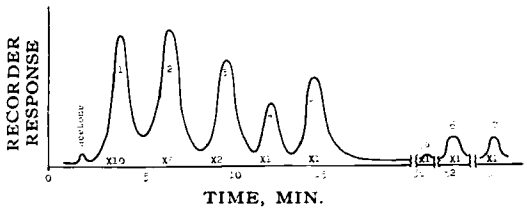

Fig. 1.—Typical chromatogram of Sample A of nutmeg oil on Reoplex 400 at 100°C.

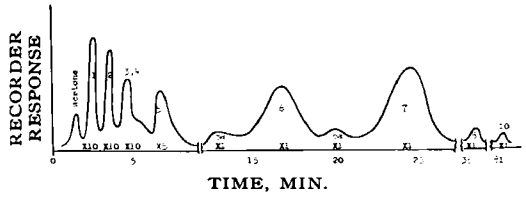

Fig. 2.—Typical chromatogram of Sample A of nutmeg oil on Reoplex 400 at 130°C.

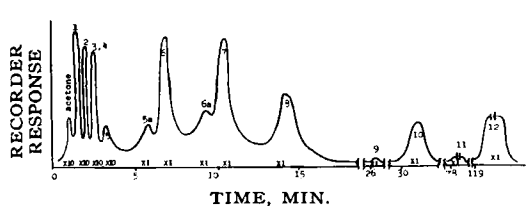

Fig. 3.—Typical chromatogram of Sample A of nutmeg oil on Reoplex 400 at 160°C.

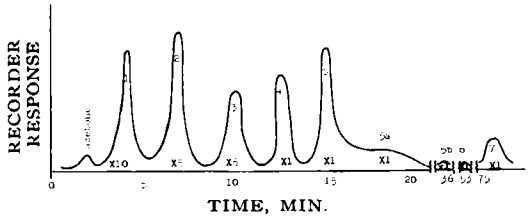

Fig. 4.—Typical chromatogram of Sample D of nutmeg oil on Reoplex 400 at 100°C.

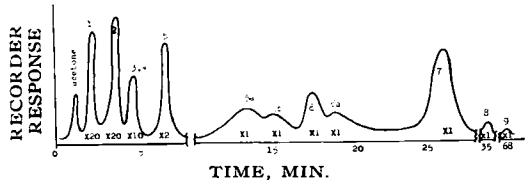

Fig. 5.—Typical chromatogram of Sample D of nutmeg oil on Reoplex 400 at 130°C.

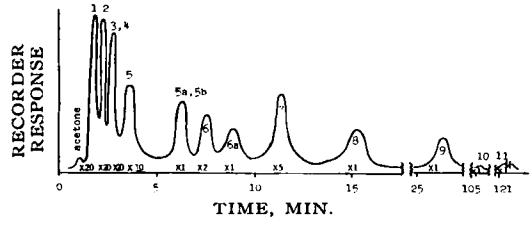

Fig. 6.—Typical chromatogram of Sample D of nutmeg oil on Reoplex 400 at 160°C.

TABLE IV.—COMPOSITION OF NUTMEG OILS, PER CENT

	Sample A	Sample B	Sample C	Sample D
α-Pinene	13.9	12.9	12.5	13.4
β-Pinene	12.3	12.6	19.1	14.8
Terpinolene	...	1.4	0.8	...
Limonene	4.7	4.7	2.3	11.9
p-Cymene	1.6	2.6	2.7	4.3
Linalool	10.6	8.1	...	5.9
Camphor	...	...	...	3.4
Terpinen-4-ol	14.5	14.2	19.5	24.7
α-Terpineol	7.7	4.7	4.0	5.0
Geraniol	0.3	11.9	...	3.6
Safrole	6.0	4.2	5.5	...
Eugenol	1.3	3.8	0.3	...
Unknown A	22.2	13.9	25.9	1.7
Unid. (3 components)	4.9	5.4	...	...
Unid. (4 components)	...	...	7.4	11.4

%, respectively. Table IV lists the percentages of individual components in each of the oils. The presence of terpinolene was observed only in Samples B and C and in 1.4 and 0.8%, respectively. One of the peaks which appeared on each of the chromatograms was identified as p-cymene and ranged from 1.6 to 4.3% (Table IV).

It should be noted that linalool and geraniol are absent in Sample C of the East Indian nutmeg oil. Samples A, B, and D contain linalool in 10.6, 8.1, and 5.9%, respectively. Geraniol was found to be present in two samples of the East Indian oil, Samples A and B. It is interesting that there is a large difference in percentage of geraniol in the two oils—namely, 0.3% in Sample A and 11.9% in Sample B. In the West Indian oil, 3.6% geraniol was found.

A component with relative retention times ranging from 5.48 to 5.58 at 100°, 4.15 to 4.31 at 130°, and 3.22 to 3.26 at 160° (Tables V, VI, VII, and VIII) which was present in all the oils, was collected and an infrared spectrum was obtained. By comparison of I.R. spectra, this compound is tentatively identified as terpinen-4 ol. West Indian nutmeg oil contained the highest amount of terpinen-4 ol (24.7%), while East Indian oils as analyzed contained the following percentages of this alcohol: 14.5, 14.2, and 19.5% for Samples A, B, and C, respectively. As can be seen from Figs. 2 and 5, another component is present with a higher retention time (peak 8). This is present in approximate range of 4–8%, depending upon the oil; it has been tentatively identified as α-terpineol using the enrichment procedure.

While safrole and eugenol were absent in the West Indian oil, both of these were present in all three samples of the East Indian variety. The per cent of safrole ranges from 4.2 to 6.0 and that of eugenol from 0.3 to as high as 3.8% (Table IV).

Another component (Unknown A) found in all samples of oil tested had a relative retention time ranging from 37.3 to 38.6 (Tables V, VI, VII, and VIII) at 160°. It was collected and its infrared

TABLE V.—RELATIVE RETENTION TIMES OF SAMPLE A OF EAST INDIAN NUTMEG OIL. (STATIONARY PHASE-REOPLEX 400; HELIUM FLOW RATE 38 ML./MIN. p-CYMENE = 1.00; FIGS. 1, 2, AND 3)

Peak No., Compd.	100°C. Unknown	100°C. Known	130°C. Unknown	130°C. Known	160°C. Unknown	160°C. Known
1. α-Pinene	0.25	0.25	0.32	0.33	0.42	0.42
2. β-Pinene	0.42	0.41	0.50	0.51	0.60	0.59
3. Limonene	0.66	0.65	0.71	0.72	0.78	0.78
4. Unidentified	0.86	...	...	...	...	...
5. p-Cymene	1.00	1.00	1.02	1.00	0.99	1.00
5a. Unidentified	2.67	...	2.04	...	1.82	...
6. Linalool	3.88	3.88	2.86	2.90	2.16	2.20
6a. Unidentified	...	...	3.49	...	2.74	...
7. Terpinen-4-ol	5.55	...	4.15	...	3.22	...
8. α-Terpineol	...	...	6.10	6.09	4.45	4.58
9. Geraniol	...	...	...	...	8.00	7.55
10. Safrole	...	...	14.2	14.2	9.24	9.62
11. Eugenol	...	...	...	...	22.5	26.1
12. Unknown A	...	...	...	...	37.3	...

TABLE VI.—RELATIVE RETENTION TIMES OF SAMPLE B OF EAST INDIAN NUTMEG OIL. (STATIONARY PHASE-REOPLEX 400; HELIUM FLOW RATE 38 ML./MIN. p-CYMENE = 1.00)

Peak No., Compd.	100°C. Unknown	100°C. Known	130°C. Unknown	130°C. Known	160°C. Unknown	160°C. Known
1. α-Pinene	0.24	0.25	0.33	0.34	0.41	0.42
2. β-Pinene	0.42	0.41	0.53	0.51	0.59	0.59
2a. Terpinolene	0.60	0.56	...	...	...	...
3. Limonene	0.64	0.65	0.75	0.72	0.79	0.78
4. Unidentified	0.84	...	...	...	...	...
5. p-Cymene	1.00	1.00	1.06	1.00	1.05	1.00
5a. Unidentified	2.68	...	2.08	...	1.86	...
6. Linalool	3.79	3.88	2.88	2.90	2.20	2.20
6a. Unidentified	...	...	3.55	...	2.80	...
7. Terpinen-4-ol	5.48	...	4.22	...	3.23	...
8. α-Terpineol	...	...	6.18	6.09	4.41	4.58
9. Geraniol	...	...	11.9	11.8	7.79	7.55
10. Safrole	...	...	14.3	14.2	9.53	9.62
11. Eugenol	...	...	...	...	24.2	26.1
12. Unknown A	...	...	...	...	38.6	...

spectrum was obtained. The spectrum showed absorption bands usually associated with aromatic ring, 1630 cm.$^{-1}$, 1516 cm.$^{-1}$, 1459 cm.$^{-1}$, and a carbonyl group 1718 cm.$^{-1}$ The quantity of this component ranged from a low of 1.7% in the West Indian oil to a high of 25.9% in Samples C of East Indian variety (Table IV).

Results obtained by the authors differ from those reported for the oil of nutmeg not only qualitatively but also quantitatively, with the possible exception of trace amounts of the esters of fatty acids, which we did not observe under the conditions used. Myristic acid and myristicin which have been reported present in a concentration of 2–3% were not eluted even after 105 minutes at a higher temperature (190°) than that used for the final analysis of the other components of the oils.

In most instances Power and Salway (8) listed the combined percentages of a few components. For example, according to these investigators α-pinene and *d*-camphene make up about 80% of the oil. Others have reported the presence of about 70% of terpenes (α-pinene, *d*-camphene, β-pinene, and dipentene). Analysis of the four samples of oil examined by us confirmed the presence of α-pinene. Samples A, B, C, and D were observed to contain 13.9, 12.9, 12.5, and 13.4% of α-pinene, respectively, while *d*-camphene was not present.

Only a small amount of β-pinene was detected in an oil analyzed in another study (9). The percentages of β-pinene observed in the present study differ considerably. As can be seen from Table IV, the percentage of β-pinene varies from a low of 12.3% to a high of 19.1%.

About 8% of a substance called dipentene was reported present in the oil analyzed by Power and Salway. It is important to note that this substance identified as dipentene by them (8) is not a pure compound but rather a mixture composed of $C_{10}H_{16}$ terpenes. Westaway and Williams (10) have analyzed two synthetic and two natural samples labeled as dipentene. Both types of samples differed both qualitatively and quantitatively. One natural sample contained as high as 80% of limonene; in the other three samples, limonene was present in various lower concentrations. Terpinolene was observed in varying percentages in all four samples. Other terpenes which were reported present by Westaway and Williams in some samples and absent in others include α-pinene, *d*-camphene, β-pinene, tricyclene, α- and γ-terpinene.

A peak labeled number three (Fig. 1) in this study present in each of the four oils and identified as limonene is the main component of dipentene referred to by Power and Salway. Furthermore, Samples B and C contained small amounts of terpinolene which were not reported present in the oil of nutmeg.

Power and Salway (9) identified *d*-linalool, *d*-borneol, "isoterpineol," and geraniol in nutmeg oil. They reported these four alcohols present in about a total of 6%. In this investigation borneol was absent in all samples of nutmeg oil, while another

TABLE VII.—RELATIVE RETENTION TIMES OF SAMPLE C OF EAST INDIAN NUTMEG OIL. STATIONARY PHASE-REOPLEX 400; HELIUM FLOW RATE 38 ML./MIN. *p*-CYMENE = 1.00

Peak No., Compd.	100°C.		130°C.		160°C.	
	Unknown	Known	Unknown	Known	Unknown	Known
1. α-Pinene	0.23	0.25	0.35	0.34	0.42	0.42
2. β-Pinene	0.41	0.41	0.54	0.51	0.59	0.59
2*a*. Terpinolene	0.63	0.56	...	...	...	...
3. Limonene	0.70	0.65	0.74	0.72	0.80	0.78
4. Unidentified	0.86	...	...	...	...	...
5. *p*-Cymene	1.02	1.00	1.03	1.00	1.07	1.00
6. Unidentified	...	...	2.12	...	1.30	...
6*a*. Unidentified	...	...	2.61	...	2.14	...
6*b*. Unidentified	...	...	3.04	...	2.57	...
7. Terpinen-4-ol	5.58	...	4.31	...	3.26	...
8. α-Terpineol	...	...	6.45	6.09	4.56	4.58
9. Safrole	...	...	14.7	14.2	9.53	9.68
10. Eugenol	...	...	...	...	24.3	26.1
11. Unknown A	...	...	...	...	38.6	...

TABLE VIII.—RELATIVE RETENTION TIMES OF SAMPLE D OF WEST INDIAN NUTMEG OIL. (STATIONARY PHASE-REOPLEX 400; HELIUM FLOW RATE 38 ML./MIN. *p*-CYMENE = 1.00; FIGS. 4, 5, AND 6)

Peak No., Compd.	100°C.		130°C.		160°C.	
	Unknown	Known	Unknown	Known	Unknown	Known
1. α-Pinene	0.24	0.25	0.32	0.33	0.42	0.42
2. β-Pinene	0.43	0.41	0.53	0.51	0.59	0.59
3. Limonene	0.66	0.65	0.72	0.72	0.78	0.78
4. Unidentified	0.85	...	...	...	...	...
5. *p*-Cymene	1.01	1.00	1.01	1.00	1.05	1.00
5*a*. Unidentified	1.30	...	2.13	...	1.86	...
5*b*. Unidentified	2.69	...	2.53	...	...	...
6. Linalool	3.91	3.88	2.92	2.90	2.20	2.20
6*a*. Camphor	...	...	3.31	3.37	2.67	3.00
7. Terpinen-4-ol	5.53	...	4.26	...	3.25	...
8. α-Terpineol	...	...	6.12	6.09	4.61	4.58
9. Geraniol	...	...	11.9	11.8	7.78	7.55
10. Unidentified	...	...	...	...	33.2	...
11. Unknown A	...	...	...	...	38.2	...

component, camphor, was detected in the West Indian oil. Linalool was present in Samples A (10.6%), B (8.1%), and D (5.9%), while it was not observed in Sample C of East Indian variety. The same observation was made as far as geraniol is concerned. We found 3.6% present in the West Indian sample, while two samples of the East Indian variety contain 0.3 and 11.9%, respectively.

The amount of α-terpineol varies depending on the sample of the oil (Table IV). Westway and Williams (10) did not report the percentage of α-terpineol in the oil they investigated.

In 1907 (9), safrole was reported present in about 0.6% in the oil of Ceylon nutmeg. Our analysis confirmed its presence in the East Indian oils only. The amount ranges from 4.2 to 6.0%. This same report mentions the presence of about a total of 0.2% of eugenol and isoeugenol in the oil from Ceylon. A peak corresponding to the retention time of isoeugenol was not observed in the chromatograms. We have observed eugenol in varying amounts in all three samples of the East Indian variety. Samples A, B, and C contain 1.3, 3.8, and 0.3%, respectively, of eugenol.

One of the constituents of the oil, which was not yet reported in literature, was isolated from the oil, using gas chromatography; an infrared spectrum was run. This constituent, which ranges from as low as 1.7% in the West Indian sample to as high as 25.9% in Sample C of the East Indian variety, was tentatively labeled as "Unknown A."

SUMMARY

Four commercially available samples of nutmeg oil were analyzed by gas-liquid chromatography, using 20% Reoplex 400 column. Relative retention times and enrichment procedure were used to identify the constituents of the oils. In some instances infrared spectra were employed.

The following components were present in the three samples of East Indian variety and in the West Indian sample: α-pinene, β-pinene, limonene, *p*-cymene, terpinen-4 ol, α-terpineol, and Unknown A. Terpinolene was found in small amounts and only in two samples of East Indian oil, while linalool and geraniol are absent in one sample of East Indian nutmeg oil. Safrole and eugenol were present in all three samples of the East Indian variety but were not observed in the West Indian sample. Camphor was found in the West Indian oil only.

Differences are also observed between the percentages of the components present not only compared to those listed in the literature but also between the oils analyzed.

REFERENCES

(1) Gildemeister, E., "The Volatile Oils," Vol. II, John Wiley and Sons, Inc., New York, N. Y. 1948, p. 18.
(2) Guenther, E., "The Essential Oils," Vol. V, D. Van Nostrand Co., Inc., New York, N. Y., p. 64.
(3) *Ibid.*, p. 65.
(4) Swing, J. M., *Spice Mill*, **72**, No. 2, 48(1949).
(5) Whitaker, U. S. Department of Commerce, Foreign Commerce Weekly, Washington, D. C., **25**(November 23, 1946) p. 8; through Guenther, E., "The Essential Oils," Vol. V, D. Van Nostrand Co., Inc., New York, N. Y., 1952, p. 63.
(6) Finnemore, H., "The Essential Oils," D. Van Nostrand Co., Inc., New York, N. Y., 1926, p. 273.
(7) Power, F. B., and Salway, A. H., *J. Chem. Soc.*, **91**, 2040(1907).
(8) *Ibid.*, **91**, 2040(1907) Transactions Pt. 2.
(9) "Semi-Annual Report of Schimmel and Co.," Fritzsche Brothers, New York, N. Y., April 1910, pp. 80–82.
(10) Westaway, H., and Williams, J. F., *J. App. Chem.*, **9**, 440–444(1959).

ERRATUM

In the paper titled "Absorption, Metabolism, and Excretion of the Semisynthetic Penicillin 6 (2-Ethoxy-1-naphthamido)penicillanic Acid (Nafcillin)" (1), a broken line represents intramuscular and a solid line represents oral groups of dogs in Figs. 1–3.

(1) Walkenstein, S. S., Wiser, R., LeBoutillier, E., Gudmundsen, C., and Kimmel, H., THIS JOURNAL, **52**, 763 (1963).