Identification of the Characteristic Odorants in Fresh Rhizomes of Ginger (*Zingiber officinale* Roscoe) Using Aroma Extract Dilution Analysis and Modified Multidimensional Gas Chromatography–Mass Spectroscopy

Osamu Nishimura

Ogawa & Company, Ltd., 1-2 Taiheidai Shoocho Katsuta-gun, Okayama 709-43, Japan

An oxygenated hydrocarbon fraction of the extract from the fresh rhizomes of ginger was analyzed by capillary gas chromatography (GC) and eluate sniffing. The application of this technique to stepwise diluted extracts of the volatile compounds allowed the determination of the flavor dilution (FD) factor, which reveals the most intense flavor compounds of the extract. Twenty-two positions with a high FD factor were detected and further analyzed by GC–mass spectroscopy (MS) and/or a modified multidimensional GC-MS system. 2-Pinen-5-ol was tentatively identified as a new compound. Compounds with high FD factor were linalool, geraniol, geranial, neral, isoborneol, borneol, 1,8-cineol, 2-pinen-5-ol, geranyl acetate, (*E*)-2-octenal, (*E*)-2-decenal, and (*E*)-2-dodecenal. In addition, (*E*)-2-alkenals, 2-octyl acetate, 2-pinen-5-ol, 2-(2′,3′-epoxy-3′-methylbutyl)-3-methylfuran, and (*E*)- and (*Z*)-3,7-dimethyl-3,6-octadienal were newly identified compounds in ginger.

Keywords: *Fresh ginger; aroma extract dilution analysis; multidimensional GC-MS; sniffing GC; odorant*

INTRODUCTION

The composition of ginger oil has been the subject of many research studies (Connell and Jordan, 1971; Bednarczyk and Kramer, 1975; Sakamura and Hayashi, 1978; Lawrence, 1983; MacLeod and Pieris, 1984; Sakamura, 1987; van Beek et al., 1987; Ekundayo et al., 1988; Erler et al., 1988). More than 200 different volatiles have been identified so far in ginger oil. The odor of ginger is not characterized by one particular compound, but rather by a mixture of various terpenoids as well as some non-terpenoids. It has been considered unlikely that the typical aroma of ginger would ever be completely unraveled due to the enormous complexity of the oil, the many problems connected with the subjective interpretation of the odor of the individual components, and the existence of many ginger varieties (van Beek et al., 1987).

Bertsch (1978) described the use multidimensional gas chromatography (GC) for separating the components from complex mixtures, such as flavors and diesel oil. Moreover, the multidimensional GC system combined with a mass spectrometer (MDGC-MS) is a very powerful tool for the identification of compounds in a complex mixture (Henderickx and Ramaekers, 1994).

As Grosch (1993) recently reviewed, the potent odorants of a food are located in the capillary gas chromatogram by gas chromatography–olfactometry (GCO) of serial dilutions of an extract. This procedure is called aroma extract dilution analysis (AEDA), and the results are expressed as flavor dilution (FD) factors. The FD factor for a compound is the ratio of its concentration in the initial extract to its concentration in the most dilute extract in which the odor was detected by GCO. Odorants with a high FD factor are important contributors to the characteristic flavors and are suitable as indicator substances for the objective determination of flavor differences in food (Milo and Grosch, 1993).

The objective of the present study was to identify the characteristic odorants in Japanese fresh ginger rhizomes (Shinshoga in Japanese) by AEDA and a modified MDGC-MS.

EXPERIMENTAL PROCEDURES

Materials. Fresh rhizomes of ginger were purchased at a local vegetable and fruit market in Japan.

Extraction and Separation. The fresh rhizomes of ginger were washed to remove soil, peeled, and sliced. Sliced rhizomes of fresh ginger (3 kg) were further grated with a food cutter along with 300 mL of distilled water. The slurry was extracted with 3 L of *n*-hexane with stirring at 500 rpm for 1 h. The solvent was removed with a rotary vacuum evaporator at 40 °C (30 mmHg) after the extract was filtered and dried over anhydrous sodium sulfate. Approximately 6 g of the oily material was obtained (yield; 0.2%). A glass column (18 × 2 cm i.d.) packed with silica gel (30 g Wakogel C-200; Wako Pure Chemical Industries, Ltd.) was used for fractionation. The oily material (~6 g) was dissolved in 15 mL of hexane, and the resultant solution was applied to the column. The column was eluted consecutively with 200 mL each of hexane (hexane fraction) and CH_2Cl_2 (O-part). Each fraction was dried over anhydrous sodium sulfate and concentrated with a rotary vacuum evaporator as just described. Approximately 2 and 3.6 g of the hexane fraction and the O-part were obtained, respectively.

Isolation of 2-Pinen-5-ol. Two hundred grams of ginger oil (purchased from Nishikawaseiyu Company, Ltd.) was distilled under reduced pressure (60–90 °C, 10 mmHg). The fraction containing 2-pinen-5-ol was fractionated by the previously described column chromatography method and then further fractionated by preparative TLC (silica gel; eluting with 1:5 ethyl acetate:hexane). 2-Pinen-5-ol was finally purified by preparative GC, and 3 mg was obtained as white needle-like crystals.

Preparation of 2-(2′,3′-Epoxy-3′-methylbutyl)-3-methylfuran. This compound was synthesized by the same previously described method (Kaiser, 1984).

Isolation of (*E*)- and (*Z*)-3,7-Dimethyl-3,6-octadienal. Thirty grams of cubeba oil (*Litsea cubeba* Pers.) was distilled under reduced pressure (70–100 °C, 18–13 mmHg). The fraction containing (*E*)- and (*Z*)-3,7-dimethyl-3,6-octadienal was further fractionated and isolated by the same methods

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used to prepare 2-pinen-5-ol. One milligram and 0.5 mg of the (*E*)- and (*Z*)-isomers were obtained, respectively.

Gas Chromatography–Olfactometry (GC-O). A Hewlett-Packard (HP) model 5890A gas chromatograph equipped with a thermal conductive detector (TCD) was used. A fused silica column (30 m × 0.53 mm i.d.; coated with a 1-μm film of Supelco wax 10; Sigma Aldrich Japan K.K.) was used without splitting. The column temperature was programmed from 80 to 210 °C at a rate of 3 °C/min in all runs. Both the temperature of the injector and detector were set at 250 °C. Helium was used as the carrier at a flow rate of 4.4 mL/min. A glass sniffing port was connected to the outlet of the TCD and was heated at 60 °C by a ribbon heater. Moist air was pumped into the sniffing port at 100 mL/min to quickly remove the odorant eluted from the TCD out of the sniffing port.

Aroma Extract Dilution Analysis (AEDA). The O-part of the extract of ginger was analyzed by capillary GC on the Supelcowax 10 column. The odor active positions were detected by GC eluate sniffing (GC-O). The FD factors of the odorants were determined by AEDA (Grosch, 1993), and a FD chromatogram (plot of the FD factor of each odorant versus its retention index) was drawn (Grosch, 1993).

Modified MDGC-MS System. The gas chromatograph used for pre-separation of MDGC was a Hitachi 163 gas chromatograph (GC1) equipped with a split injector and a flame ionization detector (FID). The temperatures of both the injector and detector were 250 °C. Helium was used as the carrier gas, and the split ratio was 1:6. A fused silica column (30 m × 0.53 mm i.d.; coated with a 1-μm film of Supelcowax 10) was used as the preparative column. The column end was divided into a two-way passage by a outlet splitter system (GL Sciences, Inc.) in the oven. The divided ratio was 1:4. One (1 part) end led to an FID for monitoring and the other (4 parts) was transferred out of the GC1 oven; the outlet was connected to a 6-mm o.d. glass tube that was packed with Porapak type Q adsorbent (50–80 mesh) obtained from the Millipore Corp. (Milford, MA). The column temperature was programmed in the same way as the GC-O conditions. The GC-MS used was an HP5890 Series II gas chromatograph coupled with a Hitachi Model M-2500 mass spectrometer. The thermal desorption cold trap injector (TCT; supplied from Chrompack) was mounted on the gas chromatograph. Helium was used as the carrier gas. The analytical column used was a 60 m × 0.25 mm i.d. fused silica capillary coated with a 0.25-μm film of DB-1 (J&W Scientific). The column temperature was programmed in the same way as the GC-O conditions. The mass spectrometer was used under the following conditions: ionization voltage, 70 eV; accelerating voltage, 3100 V; ion source temperature, 200 °C. The TCT was operated under the following conditions in all runs: pre-cooling, the cold trap was cooled down to −100 °C; thermal desorption, 180 °C, 5 min, cold trap was maintained at −100 °C; and injection, the cold trap was heated to 220 °C (heating rate, 15 °C/s). The purge gas flow rate in the TCT was 10 mL/min. The split ratio of injector was 1:1.

The analysis was carried out as follows: the volatile heart-cut compounds after separation by the GC1 column were adsorbed onto the Porapak Q column directly out of the GC1 oven. This Porapak Q column was then fitted to the TCT. The volatile compounds adsorbed on the Porapak Q were thermally desorbed, cold trapped, and injected onto the analytical column in the GC-MS.

Preparative GC. The same GC system described in the GC-O was used for the preparation or isolation of a compound.

IR and NMR Spectral Analysis. The IR spectra were recorded on a HP 5965A GC/FT-IR system. NMR spectra were measured in $CDCl_3$ with a Bruker AM-400 with tetramethylsilane as the internal standard.

Identification of Components. The identification of components was made by comparison of their Kovats GC retention indices and MS spectra to those of authentic compounds.

RESULTS AND DISCUSSION

Japanese fresh ginger rhizomes (Shinshoga in Japanese) have a strong citrus-like, camphoraceous, floral, musty, and fatty green odor. The extract of ginger used in this experiment (yield, 0.2%) possessed this fresh ginger odor. This characteristic odor was concentrated in the O-part prepared by column chromatography. AEDA was used for the objective determination of the components that contribute to the characteristic odor of ginger. AEDA of the O-part of the chromatogram revealed 22 positions with FD factors of 10 or higher (Figure 1). These positions seem to make a significant contribution to the characteristic odor of the fresh rhizomes of Japanese ginger. Components in these positions were identified by comparison of their Kovats indices and mass spectra to those of authentic compounds. The odor of these positions were smelled by the GC-O, and the results are summarized in Table 1. Connell and Jordan (1971) reported that freshly harvested Queensland-grown ginger rhizomes possess a "citrus-like" aroma, and compounds with this citrus-like odor were identified as geranial and neral. In this study, the no. 16 and no. 13 positions expressed a strong citrus-like odor and were identified as geranial and neral, respectively, on the basis of their Kovats indices and mass spectra and agreement of their odor quality. This study also confirmed that geranial and neral contribute to the citrus-like odor of fresh ginger. In addition, geranial and neral have high FD factors of 17 and 15, respectively.

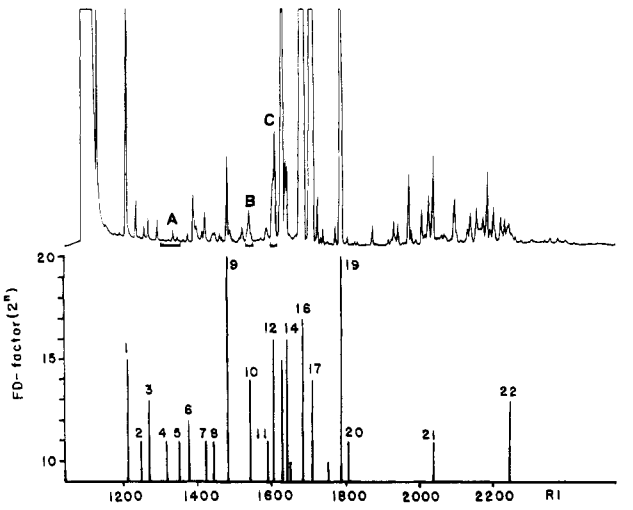

Figure 1. Gas chromatogram monitored by TCD (top) and FD chromatogram (bottom) of the oxygenated hydrocarbon fraction (O-part) of the extract from Japanese fresh ginger rhizomes. The numbers indicate the positions at which an odor was perceived at the sniffing port. The letters **A**, **B**, and **C** are the heart-cut fractions by the modified MDGC system.

A compound in position 17, which has a floral and rosy odor with a high FD factor of 14, was identified as geranyl acetate on the basis of its Kovats index and mass spectra and agreement of its odor quality. Geranyl acetate was previously reported as a unique compound in Japanese fresh ginger rhizomes (Sakamura and Suga, 1987) and seems to be one of the characteristic odorants of Japanese fresh ginger.

1,8-Cineol, linalool, borneol, and geraniol were previously reported as constituents of ginger cultivated in Japan (Sakamura and Hayashi, 1978), in Fiji (Smith and Robinson, 1981), in Sri Lanka (MacLeod and Pieris, 1984), in Vietnam (van Beek et al., 1987), and in Nigeria (Ekundaya et al., 1988), but the degree of contribution of these compounds to the ginger odor has not been discussed. 1,8-Cineol and borneol have camphoraceous and dry-camphoraceous odors, respectively, and have FD factors of 15 and 16, respectively. Both linalool and

Table 1. Potent Odor Compounds in the O-Part of the Extract from the Fresh Ginger Rhizomes

no.	compound	RI (DB-WAX)	odor description[a]	FD factor
1	1,8-cineole	1214	camphoraceous	15
2	bornyl methyl ether[b]	1252	earthy, musty	11
3	2-heptanol	1273	mushroom-like, herbaceous	13
4	unknown	1320	green, fruity, melon-like	11
5	unknown	1352	mushroom-like	11
6	(*E*)-2-octenal[c]	1377	green, nutty, burdock-like, fatty	12
7	citronellal	1425	Japanese pepper tree-like	11
8	decanal	1447	green, waxy	11
9	linalool	1484	floral	20
10	2-undecanone 4-terpineol	1543	musty, dusty, green, camphoraceous	14
11	(*E*)-2-decenal[c]	1590	fatty, green	11
12	citronellyl acetate	1607	musty, dusty, rosy	16
13	neral	1630	citrus-like, peely	15
14	borneol	1642	dry-camphoraceous	16
15	unknown	1652	musty, dusty	10
16	geranial	1686	citrus-like	17
17	geranyl acetate	1711	floral, rosy	14
18	nerol	1753	floral	10
19	geraniol	1788	floral, rosy	20
20	(*E*)-2-dodecenal[c]	1807	fatty, green	11
21	zingiberenol	2037	metalic, lemony	11
22	isoeugenol	2250	spicy, floral	13

[a] Odor description assigned during AEDA. [b] Tentatively identified. [c] Newly identified in the aroma extract from ginger.

Table 2. Volatile Odor Compounds in the O-Part of the Extract from Fresh Ginger Rhizomes Identified by a Modified MDGC-MS

peak no.	compound	odor description	RI (OV-101)	fraction
1	2-octanol	mushroom-like	987	A
2	2,6-dimethyl-5-heptenal	green, fruity, melon-like	1039	A
3	2-nonanone	fruity, fatty	1093	A
4	2-(3′-methyl-2′-butenyl)-3-methylfuran	green, minty	1099	A
5	nonanal	floral, waxy, green	1102	A
6	2-pinen-5-ol[a]	musty, dusty	1106	B
7	*cis*-rose oxide	floral	1115	A
8	*trans*-rose oxide	floral	1128	A
9	2-octyl acetate[a]	fruity, floral	1132	A
10	(*Z*)-3,7-dimethyl-3,6-octadienal[a]	green	1151	C
11	camphene hydrate	camphoraceous	1152	B
12	isoborneol	musty, dusty	1157	C
13	2-(2′,3′-epoxy-3′-methylbutyl)-3-methylfuran[a]	green, earthy, citrus-like	1161	B
14	(*E*)-3,7-dimethyl-3,6-octadienal[a]	green	1166	B, C
15	4-terpineol	earthy, musty	1175	B
16	neral	citrus-like, peely	1222	B, C
17	geranial	citrus-like	1249	B, C
18	2-undecanone	fruity, rosy	1269	B
19	citronellyl acetate	floral, rosy	1335	C

[a] Newly identified in the aroma extract from ginger.

geraniol have floral and rosy odors, and each of them has the highest FD factor of 20. These findings indicate that 1,8-cineol, borneol, linalool, and geraniol are the important odorants in the fresh ginger odor.

(*E*)-2-Alkenals, (*E*)-2-octenal, (*E*)-2-decenal, and (*E*)-2-dodecenal are known as constituents of bitter orange oil (Boelens and Sindreu, 1988) and of the aroma from Citrus Unshiu juice (Yajima et al., 1979), but they have not been found in ginger oil to date. These compounds have a very strong fatty and green odor; therefore, they could be considered to be characteristic odor components giving freshness to the Japanese fresh ginger rhizomes. Citronellal has a fresh, green-citrus-like, and Japanese pepper tree-like odor and could also contribute to the freshness of the ginger odor.

Bornyl methyl ether was tentatively identified in the ginger for the first time and has an earthy and musty odor.

Position nos. 10 and 12 have high FD factors of 14 and 16, respectively. The odor qualities of these positions, however, did not agree with the odor qualities of compounds identified in these positions. Unknown compounds may be present in these positions and they may cause the high FD factors. For the identification of these unknown compounds in position nos. 4, 5, 10, and 12, the modified MDGC-MS system was used. This system was a very effective tool for the separation and identification of components from a complex mixture. Each fraction (A, B and C, Figure 1) was heart-cut, and the volatiles in these fractions were separately adsorbed on Porapak Q. These procedures were repeated three times. Each volatile in fractions A, B, and C was separately injected into a GC-MS column using the modified MDGC-MS system. The total ion chromatograms obtained by GC-MS of each fraction (A, B and C) are shown in Figure 2. The compounds were further identified by modified MDGC-MS and are summarized in Table 2.

2-Nonanone, nonanal, 2-(3′-methyl-2′-butenyl)-3-methylfuran, 2-octanol, 2,6-dimethyl-5-heptenal, and 2-octyl acetate were identified from fraction A (Figure 2). 2-Octyl acetate was newly identified as an odorant of ginger extract. 2,6-Dimethyl-5-heptenal and 2-octanol were recognized as the constituents of position nos. 4

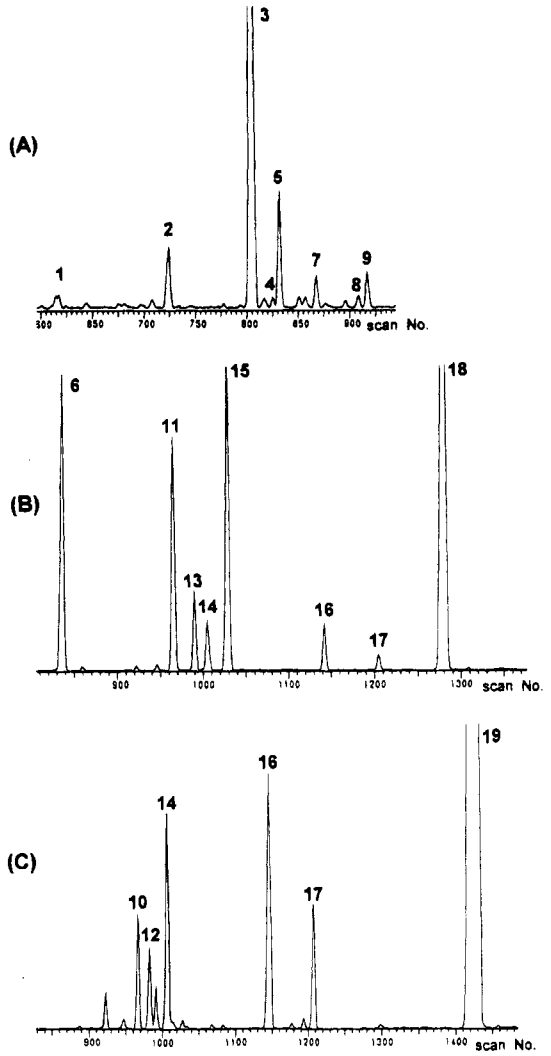

Figure 2. Total ion chromatograms (OV-101 column) of heart-cut fractions **A**, **B**, and **C** in Figure 1. The identified compounds are listed in Table 2.

and 5, respectively, in Figure 1, in agreement with their odor qualities.

Compounds **6**, **13**, and **14** in fraction B, containing 2-undecanone, 4-terpineol, camphene hydrate, neral, and geranial, were still unknown (Figure 2, corresponding part no. 10 in Figure 1). Neral and geranial were contaminated in fraction B because the effect of fronting of the peak, which was caused by overloading of these compounds on the column. Compound **6** (Table 2) was isolated and tentatively identified as 2-pinen-5-ol. Examination of the mass spectrum of **6** indicated that its molecular weight is 152. The IR spectrum of **6** shows a sharp absorption at 3642 cm^{-1} due to the OH stretching vibration of alcohols. The ^1H and ^{13}C NMR spectra of **6** are summarized as follows: ^1H NMR (δ, $CDCl_3$, Me_4Si; J, Hz): 0.83 (3H, s), 1.20 (3H, s), 1.57 (1H, d, J = 8.4), 1.66 (3H, m), 1.86 (1H, dd, J = 6.8, 2.2), 2.2 (2H, m), 2.26 (1H, m), 5.31 (1H, m); ^{13}C NMR (δ, $CDCl_3$, Me_4Si): 18.32, 20.86, 22.22, 38.15, 41.12, 42.51, 44.98, 75.32, 118.99, and 143.38. These spectral data strongly suggest that **6** (Table 2) is 2-pinen-5-ol. To positively identify **6** (Table 2), synthesis work is necessary. Compound **6** is newly identified as an odorant of ginger with a strong musty odor.

Compound **13** was determined to be 2-(2′,3′-epoxy-3′-methylbutyl)-3-methylfuran on the basis of its authentic mass spectra and Kovats index. Compound **13**, with a green, earthy, and citrus-like odor, was previously found in geranium oil (Kaiser, 1984), but is identified here for the first time in ginger. Compounds **10** and **14** (Table 2) were identified as the same compounds, (*Z*)- and (*E*)-3,7-dimethyl-3,6-octadienal, which were isolated from a cubeba oil. The (*Z*)- and (*E*)-forms were determined by measurement of ^1H NMR spectra. The coupling constant values for $J_{4,10}$ were 1.75 and 1.6 Hz for **10** and **14** (Table 2), respectively. The NOE effect between the C_2-H and C_4-H groups of **14** (Table 2) was ~8%, but there was no NOE effect between the C_{10}-H and C_4-H groups of **14** (Table 2). These compounds were previously found in palmarosa oil as iso-citral (Surburg, 1988) and were regarded as artifacts formed during the distillation conditions. In this study, distillation was not performed. Therefore, (*E*)-3,7-dimethyl-3,6-octadienal was considered to be originally present in the fresh ginger rhizomes. Camphene hydrate, 4-terpineol, and 2-pinen-5-ol were considered as contributors of the odor of position no. 10, in agreement with these odor qualities.

Citronellyl acetate as a main component, neral, geranial, (*E*)- and (*Z*)-3,7-dimethyl-3,6-octadienal, and isoborneol were identified in fraction C, as shown in Figure 2 (corresponding to part 12 in Figure 1). The odor description of fraction C was musty, dusty, and rosy (Table 1). The rosy note is due citronellyl acetate, but the musty and dusty note is due isoborneol. The high FD factor of 16 is thought to be due to isoborneol because of its odor quality.

In conclusion, careful examination of the oxygen-containing compounds of the extract from Japanese fresh ginger was accomplished by a modified MDGC-MS technique and AEDA. In addition, the sensory role the identified compounds play in the odor of fresh ginger was estimated. Compounds that showed a high FD factor were linalool, geraniol, geranial, neral, borneol, isoborneol, 1,8-cineol, 4-terpineol, geranyl acetate, 2-pinen-5-ol, (*E*)-2-octenal, (*E*)-2-decenal, and (*E*)-2-dodecenal. No particular compound represented the characteristic odor of ginger. However, the monoterpenoids and (*E*)-2-alkenals that have high FD factors are of great importance for the odor of fresh ginger.

The combined application of a MDGC-MS and AEDA can be a very promising method for the analysis of compounds in complex mixtures such as flavor.

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