

## Identification of Potent Odorants in Chinese Jasmine Green Tea Scented with Flowers of *Jasminum sambac*

YURIKO ITO,<sup>†</sup> AKIO SUGIMOTO,<sup>‡</sup> TAKAMI KAKUDA,<sup>‡</sup> AND KIKUE KUBOTA<sup>\*,†</sup>

Laboratory of Food Chemistry, Ochanomizu University, 2-1-1 Ohtsuka, Bunkyo-ku, Tokyo 112-8610, Japan, and Central Research Institute, Ito En Ltd., 21 Mekami, Sagara-cho, Haibara-gun, Shizuoka 421-0516, Japan

The odorants in Chinese jasmine green tea scented with jasmine flowers (*Jasminum sambac*) were separated from the infusion by adsorption to Porapak Q resin. Among the 66 compounds identified by GC and GC/MS, linalool (floral), methyl anthranilate (grape-like), 4-hexanolide (sweet), 4-nonanolide (sweet), (*E*)-2-hexenyl hexanoate (green), and 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (sweet) were extracted as potent odorants by an aroma extract dilution analysis and sensory analysis. The enantiomeric ratios of linalool in jasmine tea and *Jasminum sambac* were determined by a chiral analysis for the first time in this study: 81.6% ee and 100% ee for the (*R*)-(–)-configuration, respectively. The jasmine tea flavor could be closely duplicated by a model mixture containing these six compounds on the basis of a sensory analysis. The omission of methyl anthranilate and the replacement of (*R*)-(–)-linalool by (*S*)-(+)-linalool led to great changes in the odor of the model. These two compounds were determined to be the key odorants of the jasmine tea flavor.

**KEYWORDS:** Jasmine tea; potent odorant; *Jasminum sambac*; (*R*)-(–)-linalool; methyl anthranilate; enantiomers

### INTRODUCTION

Many types of tea are produced and consumed in China, including green tea, oolong tea, black tea, and scented tea, the latter being consumed in the greatest quantities. Scented tea is reprocessed from processed green, oolong, black, and other teas by absorbing the fragrance of fresh flowers. Various kinds of flowers are used to characterize the flavor of tea such as two kinds of jasmine, *Jasminum sambac* and *J. officinale* var. *grandiflorum* L., magnolia (*Michelia*), rose, pomelo (*Citrus grandis*), daidai (*Citrus aurantium* var. *amara*), *Chloranthus*, and *Osthmanthus* (*I*). Among these scented teas, Chinese jasmine green tea, which is reprocessed green tea scented with flowers of *J. sambac*, is the most popular and forms the basis of this study.

Chinese jasmine green tea is also familiar in Japan, because it has been served in Chinese restaurants for a long time, and PET-bottled and canned jasmine tea has recently become popular with the Japanese.

Jasmine tea is generally produced by the following process (*I*). The flower buds are picked and immediately spread out. When the buds have bloomed to about 90%, these flowers are mixed with green tea leaves at 38–44 °C to absorb the fragrance of the flowers, and the flowers are then separated from the tea leaves. This process is repeated with new flower buds until

sufficient fragrance has been absorbed by the tea leaves. The scented leaves are then dried at about 100 °C for 8–10 min. During this process, the characteristic scented tea aroma gradually forms and this elegant aroma plays an important part in the appeal of jasmine tea. The pan-fired and basket-dried green tea is used to make scented tea. Longjing-tea, which is the most famous green tea in China, is pan-fired and pan-dried green tea. The “week and sweet green” aroma of green tea material used to make Chinese jasmine green tea is different from the “roasted green” aroma of Longjing-tea. The volatile compounds in Chinese jasmine tea have been investigated by Yamanishi et al. with modified Likens–Nickerson simultaneous distillation equipment (SDE), and 25 volatile compounds were identified in the essential oil of jasmine tea (*2*). They have described the correlation between the quality grade of jasmine tea and the ratio of the total concentration of benzyl alcohol, benzyl acetate, (*Z*)-3-hexenyl benzoate, methyl benzoate, and methyl anthranilate to that of linalool. On the other hand, recent flavor research has tended to extract the potent odorants after the volatile components have been investigated in detail, so that the aroma character can be expressed with a mixture of the minimum compounds (*3–5*). The enantiomeric purity of extracted chiral odorants needs to be investigated, because it is known that sensory properties such as the odor quality and strength are often quite different between optical isomers (*6*). A detailed examination of the potent odorants in jasmine tea also remains to be investigated.

To clarify the character of the jasmine tea aroma in this present study, at first a method for isolating the volatile

\* To whom correspondence should be addressed (telephone +81-3-5978-5758; fax +81-3-5978-5759; e-mail Kubota@cc.ocha.ac.jp).

<sup>†</sup> Ochanomizu University.

<sup>‡</sup> Ito En Ltd.

compounds was determined, and then the aroma components were investigated in detail, including their absolute configurations. The potent odorants were also determined by an aroma extract dilution analysis (AEDA) (7), these compounds were then quantified to obtain a model mixture, and the key aroma compounds were finally examined by a sensory analysis.

## MATERIALS AND METHODS

**Materials.** Jasmine tea produced at Fujian province of China in September 2000 was used. It was prepared from green tea (*Camellia sinensis* L.) scented with flowers of *Jasminum sambac*. After the scenting process, the flowers were removed from the tea, and dried flowers of *J. sambac* were newly added to about 1% of the weight of tea.

**Standard Compounds for Identification and the Model Mixture.** (*R*)-(-)-Linalool, (*S*)-(+)-linalool, 3,7-dimethyl-1,5,7-octatrien-3-ol, methyl epijasmone, 2,6-dimethyl-3,7-octadiene-2,6-diol, and linalool oxides were provided by T. Hasegawa Co. Ltd. (Tokyo, Japan). (*R*)-(-)-Linalool and (*S*)-(+)-linalool, respectively, contained as an impurity a small amount of the optical isomer at 4% and 2% by a GC analysis. 4-Methyl-5-hexen-4-olide and dihydroactinidiolide were gifts from Takasago Co. Ltd. (Tokyo, Japan). The other standard compounds were purchased from Tokyo Kasei Kogyo Co. Ltd. (Tokyo, Japan) including the methyl anthranilate, 4-hexanolide, 4-nonanolide, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (Furanol), and (*E*)-2-hexenyl hexanoate.

**Isolation of the Volatile Compounds.** One liter of hot water (95 °C) purified by the Milli-Q system was added to 50 g of jasmine tea comprising 49.5 g of tea leaves and 0.5 g of jasmine flowers. The infusion was filtered through a nylon cloth after 3 min. The filtrate (800 mL) was immediately cooled to 30 °C under tap water and centrifuged for 15 min at 3000 rpm to remove the solids. The supernatant fraction was subjected to chromatography in a column packed with 18 mL of Porapak Q resin. The adsorbed compounds were eluted with 200 mL of a mixture of pentane and diethyl ether (2:3), after the water-soluble compounds such as amino acids and sugars had been removed with 200 mL of purified water. The eluate was dried over anhydrous sodium sulfate, and the solvent was evaporated at 39.5 °C at atmospheric pressure. Then the volatile compounds were concentrated with a nitrogen stream to 30  $\mu$ L just before injecting for the GC or GC-MS analyses.

The volatile compounds of dried jasmine flowers (*J. sambac*) were also isolated. The dried flowers in the jasmine tea were collected, and 1 L of hot purified water was added to 0.5 g of the dried jasmine flowers. After infusing for 3 min, the flowers were filtered off. The resulting filtrate was immediately cooled to 30 °C under tap water and subjected to chromatography in a column of Porapak Q resin. The remaining procedures were performed in the same way as those used for the jasmine tea.

**Gas Chromatography (GC).** An HP 5890 Series II gas chromatograph equipped with a flame-ionization detector was employed. Helium was used as the carrier gas at a flow rate of 1 mL/min with a split ratio of 30:1. The column was 60 m  $\times$  0.25 mm i.d. coated with DB-WAX (J&W Scientific). The oven temperature was held at 60 °C for 4 min and then increased to 180 °C at a rate of 3 °C/min. The injector and detector temperatures were set at 170 °C and 180 °C, respectively, because it has been reported that methyl epijasmone and its isomers, which are important aroma compounds in tea, were significantly isomerized or decomposed at higher than 180 °C (8).

**Gas Chromatography-Mass Spectrometry (GC-MS).** An HP 5890 Series II gas chromatograph equipped with an HP 5972 mass selective detector and Willay library was used for the GC-MS analysis. Injection was performed in the split-less mode, and the GC conditions were the same as those used for the GC analysis. Electron impact (EI) spectra were recorded, the temperature of the ion source being 180 °C, and mass spectra were scanned at 70 eV in an *m/z* range from 30 to 400 mass units.

**Identification of the Components.** The components were identified by agreement of their Kovats' GC retention indices and mass spectra with those of authentic standards.

**Gas Chromatography-Olfactometry (GC-O).** GC-O was conducted in a column of 60 m  $\times$  0.53 mm i.d. (1  $\mu$ m film) coated with DB-WAX (J&W Scientific) in the split-less mode. The GC instrument and temperature conditions were the same as those used for the GC analysis. Helium was employed as the carrier gas at a flow rate of 8.3 mL/min. After adding 18 mL/min of makeup gas at the outlet of the column, the carrier gas was split 1:1 and passed through a glass sniffing port and FID detector. Wet air was pumped into the sniffing port at 45 mL/min to quickly remove the odorant that had previously passed through the port and to keep the nose moist.

**Determination of the Potent Odorants.** An aroma extract dilution analysis (AEDA) was used to determine the potent odorants in jasmine tea (7). The original odor concentrate (65  $\mu$ L) isolated from 50 g of jasmine tea was diluted stepwise 4-fold with diethyl ether by volume and then subjected (1  $\mu$ L) to a GC-O analysis. The flavor dilution (FD) factors of the odorants were determined, and an FD chromatogram (plot of the FD factor of each odorant versus its retention time) was prepared. To identify the compounds, the odor quality during the GC-O analysis was confirmed, as well as the retention indices and mass spectra.

**Investigation of the Enantiomeric Purity.** A column of 50 m  $\times$  0.25 mm i.d. coated with CP-cyclodextrin-B-236-M-19 (Chrompack, Middelburg, The Netherlands) was used for chiral GC and GC-MS analyses. The carrier gas and split ratio were same as those described for the GC analysis. Linalool was investigated with the oven temperature set at 95 °C, and the injector and detector temperatures both set at 200 °C. The other compounds were investigated with the oven temperature increased from 60 °C to 200 °C at the rate of 1 °C/min, and the injector and detector temperatures both set at 200 °C.

**Quantitative Analysis.** The aroma components in the jasmine tea infusion were quantitatively analyzed. Hot water (750 mL) was added to 10 g of jasmine tea (9.9 g of tea leaves and 0.1 g of dried jasmine flowers) and, after infusing for 3 min, the tea leaves and flowers were filtered off, and the filtrate was used as the jasmine tea infusion. Ethyl decanoate (0.1 mg) and 0.02 mg of methyl palmitate in methanol were added to 660 mL of the jasmine tea infusion as internal standards, with these two different concentrated internal standards being added to accurately determine the amounts of the low-concentration peaks and to confirm the reproducibility of the aroma concentrate. The odorants were then isolated by using Porapak Q resin in the same way as that for isolating the volatile compounds and analyzed by GC apparatus equipped with FID. Calibration curves were prepared using the mixture of authentic samples of odor-active compounds and internal standards (ethyl decanoate and methyl palmitate). The equation between the absolute concentration and peak area % by FID response of each authentic standard is shown later in Table 5. The absolute concentrations of the odor-active components in jasmine tea infusion were determined by the ratio of peak area % of each compound to the internal standard and the calibration curves of authentic samples. Injection was performed in the split-less mode.

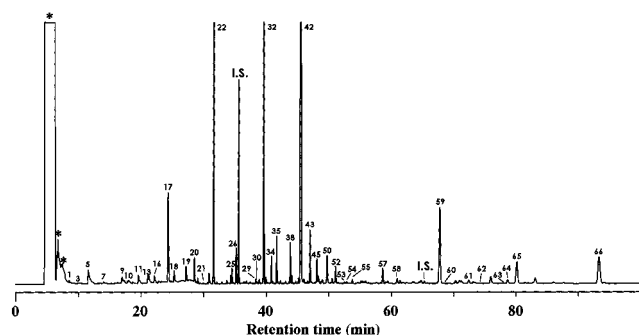
**Sensory Evaluation. Sample Preparation.** A jasmine tea infusion was prepared in the same way as that used for the quantitative analysis to provide a sample for the sensory evaluation. A model mixture was first prepared by using authentic samples to quantify the extracted odor-active compounds. Authentic samples were each diluted with a small amount of propylene glycol and then mixed in the proportions indicated by the data from the quantitative analysis of the jasmine tea infusion (Table 5). The mixture was diluted with purified water until the strength of odor impression of the model mixture reproduced that of the original jasmine tea infusion on the basis of the sensory evaluation.

**Evaluation Terms.** The terms for evaluating the jasmine tea infusion and model mixtures were first determined for the sensory evaluation. Each panelist selected the most appropriate terms for evaluating the jasmine tea infusion from among the 44 words reported by Shimoda et al. as being appropriate expressions for food (9, 10). Additionally, they were requested to write down other words with which they could evaluate the sample, if they thought the above 44 words were not sufficient to describe the odor of the jasmine tea infusion. Seven attributes were finally agreed upon after several consultations among the panelists: floral, woody, sweet, bitter, refreshing, mild, and

**Table 1.** Odor Attributes Selected for the Sensory Evaluation and the Results of Sensory Evaluation of the Aroma of Jasmine Tea Infusion and the Model Mixtures

odor attribute	meaning	jasmine tea infusion <sup>a</sup>	model mixtures <sup>a</sup>	
			I	II
floral	reminded of flower	10.3a	10.7a	6.7b
woody	wet wood-like	7.1ab	4.7a	8.9b
sweet	honey-like sweet aroma	11.6a	11.9a	4.7b
bitter	reminded of bitter taste	5.2ab	4.1a	8.2b
refreshing	generates pleasant feeling	8.9ab	5.8a	9.7b
mild	not irritating	6.7ab	5.1a	10.3b
extending	aroma sensation is wide spreading	8.7a	7.4a	10.1a

<sup>a</sup> Average score of the sensory evaluation (0, absent; 15, strong) by 15 panelists. Different letters (a and b) mean that the values were significantly different by Tukey's multiple-comparison test ( $P \leq 0.05$ ).

**Figure 1.** Gas chromatogram (DB-WAX, 0.25 mm i.d.  $\times$  60 m) of jasmine tea extracted by the column adsorption method. The injection was performed without splitting. \* Solvent peaks. Peak numbers correspond to those in Table 2.

extending. These seven selected attributes and the meanings of each are summarized in Table 1.

**Evaluation Conditions.** A well-trained panel consisting of 15 females in the age range of 21–31 years was used to evaluate the samples by a 15-cm line scale (0, absent; 15, strong) (11). A sample solution (20 mL) was served to each panelist in a 50-mL glass bottle (with a plastic screw cap) covered with a yellow cellophane sheet to unify the color of each sample. The samples were kept at 10 °C throughout the evaluation to reproduce the condition of an appropriately cooled drink. The samples were served in a random order to each panelist.

**Statistical Analysis.** The data were analyzed by Tukey's multiple-comparison test.

## RESULTS AND DISCUSSION

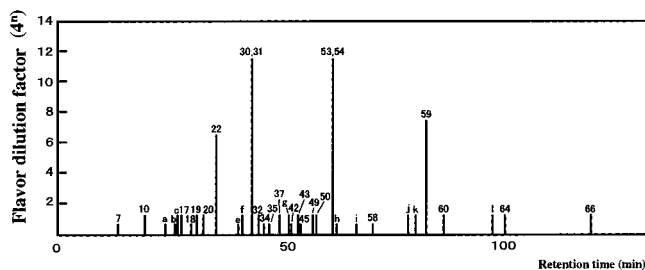
**Yield and Profile of the Aroma Concentrate.** The aroma concentrate well reproduced the characteristic odor of jasmine tea, the yield of the concentrate being  $52.3 \pm 5.1$  mg/100 g of original tea leaves. A gas chromatogram of the aroma concentrate is shown in Figure 1. Sixty-six compounds were identified by agreement of their mass spectra and KI retention indices with those of authentic compounds, and the results are summarized in Table 2. The concentration of each compound is expressed by its peak area % on the GC trace. Forty-six compounds were newly identified as the Chinese jasmine green tea aroma, although almost all compounds were already identified as the aroma compounds of tea (12). (*Z*)-3-Hexenol (peak 17), linalool (peak 22), benzyl acetate (peak 32), benzyl alcohol (peak 42), methyl anthranilate (peak 59), and indole (peak 66) were quantitatively detected as the main peaks. All these compounds have also been reported as the main volatile compounds of fresh flowers of *J. sambac* (13–16) and jasmine tea (2). Yamanishi et al. described that all of these compounds,

**Table 2.** Volatile Compounds Identified in the Aroma Concentrate of Chinese Jasmine Tea

peak	compound	KI <sup>a</sup>	peak area % <sup>b</sup>
Alcohols (Aliphatics and Aromatics)			
3	ethanol <sup>cd</sup>	931	0.10
6	butanol <sup>cd</sup>	1140	trace
7	1-penten-3-ol <sup>cd</sup>	1146	0.13
10	pentanol <sup>d</sup>	1251	0.23
12	( <i>E</i> )-2-penten-1-ol <sup>cd</sup>	1316	0.24
14	( <i>Z</i> )-2-penten-1-ol <sup>d</sup>	1324	0.33
17	( <i>Z</i> )-3-hexenol <sup>d</sup>	1390	2.58
18	cyclohexanol <sup>cd</sup>	1393	0.45
23	octanol <sup>cd</sup>	1564	trace
41	2-methoxyphenol (guaiacol) <sup>cd</sup>	1872	0.05
42	benzyl alcohol <sup>d</sup>	1891	24.68
43	2-phenyl ethanol <sup>d</sup>	1986	0.93
52	phenol <sup>d</sup>	2060	0.40
58	eugenol <sup>cd</sup>	2169	0.30
61	cinnamyl alcohol <sup>cd</sup>	2299	0.14
Alcohols (Terpenoids)			
19	linalool oxide ( <i>trans</i> , furanoid) <sup>d</sup>	1454	0.57
20	linalool oxide ( <i>cis</i> , furanoid) <sup>d</sup>	1483	0.59
22	linalool <sup>d</sup>	1552	4.46
25	3,7-dimethyl-1,5,7-octatrien-3-ol <sup>cd</sup>	1621	0.44
29	$\alpha$ -terpineol <sup>cd</sup>	1706	0.11
33	linalool oxide ( <i>trans</i> , pyranoid) <sup>cd</sup>	1751	0.10
34	linalool oxide ( <i>cis</i> , pyranoid) <sup>cd</sup>	1772	0.40
39	Geraniol <sup>d</sup>	1854	0.13
45	2,6-dimethyl-3,7-octadiene-2,6-diol <sup>cd</sup>	2004	0.52
62	1-hydroxylinalool <sup>ce</sup>	2321	trace
Esters			
1	ethyl formate <sup>cd</sup>	811	0.15
2	ethyl acetate <sup>cd</sup>	882	0.10
13	( <i>Z</i> )-3-hexenyl acetate <sup>d</sup>	1320	0.32
26	methyl benzoate <sup>d</sup>	1636	0.59
31	( <i>E</i> )-2-hexenyl hexanoate <sup>cd</sup>	1733	0.06
32	benzyl acetate <sup>d</sup>	1743	12.70
36	phenethyl acetate <sup>cd</sup>	1821	trace
57	( <i>Z</i> )-3-hexenyl benzoate <sup>d</sup>	2139	0.81
Carbonyl Compounds			
16	2-methyl-2-hepten-6-one <sup>cd</sup>	1344	0.31
21	( <i>E</i> )-2, ( <i>E</i> )-4-heptadienal <sup>cd</sup>	1503	0.03
24	3,5-octadien-2-one <sup>ce</sup>	1583	0.05
40	$\alpha$ -ionone <sup>cd</sup>	1866	0.10
46	<i>cis</i> -jasmone <sup>d</sup>	2014	0.19
48	$\beta$ -ionone <sup>d</sup>	2028	trace
51	5,6-epoxy- $\beta$ -ionone <sup>cd</sup>	2053	trace
Lactones			
28	4-methyl-5-hexen-4-olide <sup>cd</sup>	1683	trace
30	4-hexanolide <sup>cd</sup>	1709	0.12
53	4-nonanolide <sup>cd</sup>	2087	0.15
60	jasmine lactone <sup>cd</sup>	2256	trace
65	dihydroactinidiolide <sup>cd</sup>	2354	1.01
Acids			
27	2-methylbutyric acid <sup>cd</sup>	1674	0.18
38	hexanoic acid <sup>cd</sup>	1849	0.82
47	heptanoic acid <sup>cd</sup>	2022	0.14
55	octanoic acid <sup>cd</sup>	2096	0.18
N-Containing Compounds			
15	2,5-dimethylpyrazine <sup>cd</sup>	1331	0.19
44	benzyl cyanide <sup>d</sup>	2000	trace
50	2-acetylpyrrole <sup>cd</sup>	2044	0.82
56	methyl <i>N</i> -methylanthranilate <sup>ce</sup>	2118	trace
59	methyl anthranilate <sup>d</sup>	2248	3.90
66	indole <sup>d</sup>	2436	1.83
Hydrocarbons			
4	decane <sup>cd</sup>	1003	0.05
5	undecane <sup>cd</sup>	1094	0.88
9	2-ethyltoluene <sup>ce</sup>	1231	0.44
11	trimethylbenzene <sup>ce</sup>	1297	0.45
Others			
8	1,8-cineol <sup>cd</sup>	1214	trace
35	methyl salicylate <sup>d</sup>	1794	0.85
37	2-hydroxy-3-methyl-2-cyclopenten-1-one (cycloten) <sup>cd</sup>	1831	trace
49	2-hydroxy-3-methyl-4 <i>H</i> -pyran-4-one (maltol) <sup>cd</sup>	2030	trace
54	4-hydroxy-2,5-dimethyl-3(2 <i>H</i> )-furanone (Furaneol) <sup>cd</sup>	2090	0.30
63	methyl jasmonate <sup>cd</sup>	2345	0.05
64	methyl epijasmone <sup>cd</sup>	2349	0.15

<sup>a</sup> KI, Kovats index. <sup>b</sup> Peak area % on GC. <sup>c</sup> Newly identified compound as Chinese jasmine green tea aroma composition. <sup>d</sup> Identified by agreement of the mass spectrum and the KI index with those of the authentic compound. <sup>e</sup> Tentatively identified only by the mass spectrum.





**Figure 2.** FD chromatogram of the volatile fraction of jasmine tea. The peak numbers correspond to those in **Table 3**.

including (*Z*)-3-hexenyl benzoate and methyl benzoate instead of (*Z*)-3-hexenol and indole, were contributors to the quality grade of jasmine tea as already mentioned in the Introduction section. It was considered that the small difference of aroma composition between the report of Yamanishi et al. and our study was due to the isolation method used. Methyl salicylate (peak 35), 2-phenylethanol (peak 43), 2,6-dimethyl-3,7-octadiene-2,6-diol (peak 45), 2-acetylpyrrole (peak 50), (*Z*)-3-hexenyl benzoate (peak 57), and dihydroactinidiolide (peak 65), which are also known as important components of oolong, black, and green teas (12, 17–19), were also detected as fairly large peaks. In addition, jasmine lactone (peak 60), methyl jasmonate (peak 63), and methyl epijasmolate (peak 64), which are known as potent odorants of oolong tea (20), were identified for the first time in this study as flavor compounds of jasmine tea. Although their concentrations were very low, 2-hydroxy-3-methyl-2-cyclopenten-1-one (cycloten, peak 37), 2-hydroxy-3-methyl-4*H*-pyran-4-one (maltol, peak 49), and 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (Furaneol, peak 54), which were recently newly identified in Longjing-tea (18), also seemed to contribute to the flavor of jasmine tea to some extent, because each compound has a characteristic odor and its odor threshold value is relatively low (21).

**Evaluation of the Potent Odor Compounds.** To clarify the contribution of each identified compound to the odor of jasmine tea, the AEDA method was carried out, and the resulting FD chromatogram is shown in **Figure 2**. In total, 34 peaks were recognized as odor-active compounds, and these are shown with their odor profiles in **Table 3**. Among them, 24 compounds could be identified. Almost all of the twelve unknown compounds in **Table 3** did not appear as GC peaks, but were recognized by sniffing in the GC–O analysis. The FD chromatogram consisted of 4 main peaks and 30 minor peaks: the most characteristic odor-active constituents being linalool (peak 22) and methyl anthranilate (peak 59). Linalool is one of the most popular aroma compounds found in plants. The floral note of linalool was detected by sniffing during this study. Methyl anthranilate exhibits a sweet grape-like odor and is a unique volatile compound in *J. sambac*, not being found in the volatiles of another variety of the jasmine flower (*J. grandiflorum* L.) (15). Apart from these two compounds, mixtures of 4-hexanolide (peak 30) and (*E*)-2-hexenyl hexanoate (peak 31) and of 4-nonanolide (peak 53) and Furaneol (peak 54) showed the largest FD factors. To confirm the assignment, each authentic sample mixture of 4-hexanolide and (*E*)-2-hexenyl hexanoate, and of 4-nonanolide and Furaneol was subjected to the GC–O analysis in the same condition of jasmine tea analysis. The desired odor appeared at the retention time between the former peak and the latter peak and this phenomenon agreed well with that of jasmine tea aroma analysis. It was considered that the tailing of chromatographic peak for the former compound resulted in overlapping with the latter peak in this GC condition.

**Table 3.** Potent Odor Compounds in the Jasmine Tea Aroma

peak	compound	flavor dilution factor (4 <sup>n</sup> )	odor description
7	1-penten-3-ol	1	green
10	1-pentanol	2	green
a	unknown	1	green
b	unknown	1	sweet
c	unknown	2	roasted
17	( <i>Z</i> )-3-hexenol	2	green
d	unknown	1	green
19	linalool oxide ( <i>trans</i> , furanoid)	2	leafy, citrus
20	linalool oxide ( <i>cis</i> , furanoid)	2	leafy, citrus
22	linalool	7	floral
e	unknown	1	sweet
f	unknown	2	very sweet, fruity
30	4-hexanolide	12 <sup>a</sup>	green, sweet
31	( <i>E</i> )-2-hexenyl hexanoate	12 <sup>a</sup>	green, sweet
32	benzyl acetate	2	floral
34	linalool oxide ( <i>cis</i> , pyranoid)	1	fruity
35	methyl salicylate	1	floral, green
37	2-hydroxy-3-methyl-2-cyclopenten-1-one (cycloten)	2	very sweet
g	unknown	2	herbal, slightly bitter
42	benzyl alcohol	1	fruity
43	2-phenyl ethanol	2	floral
45	2,6-dimethyl-3,7-octadiene-2,6-diol	1	sweet
49	2-hydroxy-3-methyl-4 <i>H</i> -pyran-4-one (maltol)	2	very sweet
50	2-acetylpyrrole	2	roasted, smoke
53	4-nonanolide	12 <sup>a</sup>	very sweet
54	4-hydroxy-2,5-dimethyl-3(2 <i>H</i> )-furanone (Furaneol)	12 <sup>a</sup>	very sweet
h	unknown	1	green, sweet
i	unknown	1	sweet, fruity
58	eugenol	1	spicy, green
j	unknown	2	sweet
k	unknown	2	sweet
59	methyl anthranilate	8	citrus, fruity
60	jasmine lactone	2	milky
l	unknown	2	sweet
64	methyl epijasmolate	2	sweet
66	indole	2	sweet

<sup>a</sup> FD-factor of the mixture of 4-hexanolide and (*E*)-2-hexenyl hexanoate and of 4-nonanolide and 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone.

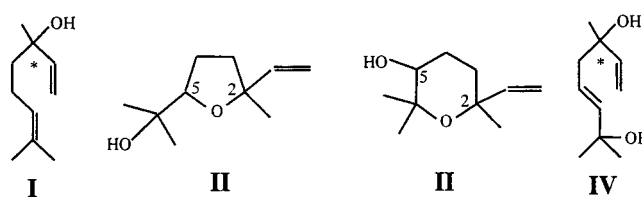
The more detailed investigation about this phenomenon is now in progress in our laboratory. The aroma character of each peak was green-sweet and sweet, respectively. In green tea, oolong tea, and black tea, (*Z*)-3-hexenyl hexanoate has been reported as one of the most important aroma compounds exhibiting a green note, although its concentration was not fairly high (17, 22–24). On the other hand, (*E*)-2-hexenyl hexanoate was not noted as strongly as (*Z*)-3-hexenyl hexanoate because of its small amount or nondetectability in tea. It is noteworthy that (*E*)-2-hexenyl hexanoate was one of the main odor-active compounds in the jasmine tea aroma. Six green-note compounds and 13 sweet-note compounds were detected as minor peaks. In addition to these compounds, citrus and floral notes from compounds such as linalool oxides (peaks 19, 20, and 34), benzyl acetate (peak 32), methyl salicylate (peak 35), benzyl alcohol (peak 42), and 2-phenyl ethanol (peak 43) seem to have contributed to the jasmine tea aroma.

**Absolute Configuration of Linalool and Its Related Compounds.** It is important to investigate the enantiomeric purity of chiral aroma components, because the flavor character of some compounds is often different between enantiomers. Linalool, linalool oxides (LOs), and 2,6-dimethyl-3,7-octadiene-2,6-diol were examined in this study with direct enantiomeric separation, using modified  $\beta$ -cyclodextrin as the chiral stationary phase. The elution order of these compounds were determined from the results of our previous study (25) and by co-chromatography with optically pure standards, and is summarized in **Table 4**. The enantiomeric ratio of linalool in jasmine

**Table 4.** Concentration of the Enantiomers of Linalool and Its Related Compounds in the Volatile Fraction of Chinese Jasmine Tea and Their Odor Characteristics

compound	configuration	GC <sup>a</sup> t <sub>R</sub> (min)	peak area <sup>b</sup> (%)	% ee	odor description
<b>linalool [I]</b> jasmine tea <sup>c</sup>	<i>R</i>	39.16	90.8	81.6	strong, green floral note <sup>e</sup> oily, heavy green note <sup>e</sup>
	<i>S</i>	39.82	9.2		
<i>Jasminum sambac</i> <sup>d</sup>	<i>R</i>	39.30	100	100	strong, green floral note <sup>e</sup> oily, heavy green note <sup>e</sup>
	<i>S</i>		0		
<b>linalool oxides</b> <i>trans</i> , furanoid [II]	2 <i>R</i> ,5 <i>R</i>	44.93	100	100	leafy, earthy <sup>f</sup> sweet, floral, creamy <sup>f</sup>
	2 <i>S</i> ,5 <i>S</i>		0		
<i>cis</i> , furanoid [III]	2 <i>R</i> ,5 <i>S</i>	47.91	100	100	leafy, earthy <sup>f</sup> sweet, floral, creamy <sup>f</sup>
	2 <i>S</i> ,5 <i>R</i>		0		
<i>cis</i> , pyranoid [III]	2 <i>S</i> ,5 <i>S</i>	66.29	66.2	-32.4	sweet, floral, creamy <sup>f</sup> earthy <sup>f</sup>
	2 <i>R</i> ,5 <i>R</i>	67.27	33.8		
<b>2,6-dimethyl-3,7-octadiene-2,6-diol [IV]</b>	<i>R</i>	77.35	23.0	-54.0	citrus, sweet, powdery <sup>g</sup> herbal green <sup>g</sup>
	<i>S</i>	77.75	77.0		

<sup>a</sup> Retention times by GC when using the CP-cyclodextrin-B-236-M19 column. The conditions are defined in the Materials and Methods section. <sup>b</sup> Peak area was calculated according to the response of the FID detector. <sup>c,d</sup> Enantiomeric ratios of linalool in the volatile compounds of jasmine tea and of dried *Jasminum sambac* flowers, respectively. <sup>e,f</sup> Cited from references 29 and 25, respectively. <sup>g</sup> Evaluated by GC-sniffing.



tea was almost optically active for the (*R*)-(-)-isomer (81.6% ee). To examine the origin of (*R*)-(-)-linalool, the enantiomeric ratio of linalool in the volatile concentrate from flowers of *J. sambac* collected from jasmine tea was also investigated. The enantiomeric ratio of linalool in *J. sambac* was optically active for the (*R*)-(-)-isomer. It has been reported that linalool in green tea volatiles existed almost as a racemate (11.4% ee for the (*S*)-(+)-isomer) (26). It thus seems that almost all (*R*)-(-)-linalool in jasmine tea was derived from the absorption of fragrance from the jasmine flowers and that (*S*)-(+)-linalool in jasmine tea originated from the green tea material. The enantiomeric ratio of linalool in *J. sambac* and jasmine tea was determined for the first time in this study. It is notable that linalool in *J. sambac* and jasmine tea was optically active or almost optically active for the (*R*)-(-)-isomer. Linalool is well-known as one of the potent odorants, as well as linalyl acetate, in lavender (*L. angustifolia*) (27) which is widely used as an aromatic herb throughout the world, and the enantiomeric ratio of linalool showed optically active for the (*R*)-configuration (91–100% ee) (28) like *J. sambac*. In contrast, the optical structure of linalool in another variety of jasmine flower, *J. grandiflorum* L., has been described to show optically active for the (*S*)-isomer (28, 29). The aroma of *J. grandiflorum* L. is quite different from that of *J. sambac*, the former being an important aromatic plant for the perfume industry. The large difference has been reported between the aroma of the (*R*)-(-)-isomer with its strong, green floral note and that of the (*S*)-(+)-isomer with its oily, heavy green note (29). It is considered that the absolute configuration of linalool plays a very important role in the overall aroma of jasmine tea.

The optical ratios of LOs and 2,6-dimethyl-3,7-octadiene-2,6-diol were also examined, although their concentrations were very low. LOs (*cis* and *trans*-furanoids) in the jasmine tea all showed optically active for the (2*R*)-isomer. It has been reported that the odor character of LOs (furanoids) was regulated by the absolute configuration of C2 (25); the (2*R*)-isomer had a leafy, earthy note, and the (2*S*)-isomer had a sweet floral creamy note.

The (2*S*)-configuration was dominant in LOs (*cis*-pyranoids). The aroma character of LOs (*cis*-pyranoids) has also been reported to be regulated by the C2 configuration (25); the (2*S*)-isomer had a sweet, floral and creamy note, and the (2*R*)-isomer had an earthy note. The concentration of LOs (*trans*-pyranoids) was too low to examine the optical ratio in jasmine tea. The absolute configuration of 2,6-dimethyl-3,7-octadiene-2,6-diol in jasmine tea also showed that the (*S*)-isomer was in excess (54.0% ee). Kawakami et al. reported that the (*S*)-isomer was in excess in the black tea (85.6% ee) and the (*R*)-isomer was in excess in the oolong tea (83.6% ee) (30). The difference in aroma character of the two isomers of 2,6-dimethyl-3,7-octadiene-2,6-diol was investigated in this study by sniffing at the exit port of the  $\beta$ -cyclodextrin column; the (*R*)-isomer had a citrus, sweet and slightly powdery note, and the (*S*)-isomer had a herbal and green note. Among linalool and its related compounds, the concentration and FD factor of linalool were much higher than those of the other compounds. In addition, the threshold values for both (*R*)- and (*S*)-linalool are very low: 0.8 ppb for the (*R*)-isomer and 7.4 ppb for the (*S*)-isomer (31). These results indicate the substantial contribution of (*R*)-(-)-linalool to the overall odor of jasmine tea.

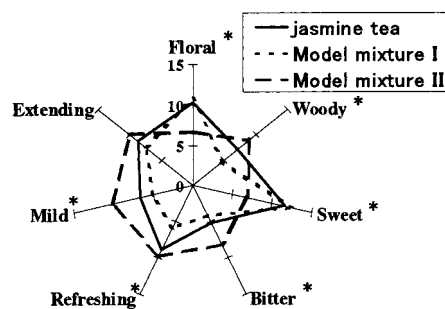
**Contribution of (*R*)-(-)-Linalool and Methyl Anthranilate to the Odor of Jasmine Tea.** To clarify whether the odorants with high FD factors were actually the key compounds of jasmine tea, model mixtures were prepared with authentic samples of the six main odor-active compounds: (*R*)-(-)-linalool, 4-hexanolide, (*E*)-2-hexenyl hexanoate, 4-nonanolide, fureneol, and methyl anthranilate. (*R*)-(-)-Linalool used here contained a small amount (4%) of the (*S*)-isomer as an impurity. The absolute concentrations of these six odor-active compounds in the jasmine tea infusion were first determined against the calibration curves of authentic samples (Table 5). A model was then prepared by mixing the authentic chemicals in a small amount of propylene glycol in the same proportions as those of the jasmine tea infusion. The odor quality of the model mixture was then compared with that of the original jasmine

**Table 5.** Equations for the Calibration Curves of Authentic Samples for Quantitative Analyses and Contents of the Main Odor-Active Compounds in the Jasmine Tea Infusion and Model Mixtures for Sensory Evaluation

compound	calibration curve <sup>a</sup>	content in tea infusion (10 <sup>-9</sup> g/mL)	content in model mixture (10 <sup>-6</sup> g/mL)	
			I	II
linalool	$y = 0.99x + 0.0136^b$ $R^2 = 0.9986$	260.7 ± 19.9		
( <i>R</i> )-(-)-linalool		236.7 <sup>d</sup>	12.5	0.3
( <i>S</i> )-(+)-linalool		24.0 <sup>d</sup>	0.5	6.2
( <i>E</i> )-2-hexenyl hexanoate	$y = 0.70x + 0.1026^c$ $R^2 = 0.9815$	9.5 ± 4.0	0.5	0.5
4-hexanolide	$y = 0.99x + 0.0455^c$ $R^2 = 0.9934$	7.5 ± 3.4	0.4	0.4
4-nonanolide	$y = 0.41x + 0.1072^c$ $R^2 = 0.9186$	16.6 ± 2.7	0.9	0.9
Furaneol	$y = 0.52x + 0.0821^c$ $R^2 = 0.9388$	13.3 ± 3.0	0.7	0.7
methyl anthranilate	$y = 0.58x + 0.0058^b$ $R^2 = 0.9997$	533.3 ± 40.7	26.6	26.6

<sup>a</sup> *y*: Peak area ratio of authentic chemical to internal standard detected by FID detector. *x*: Ratio of absolute concentration of authentic chemical to internal standard. <sup>b</sup> <sup>c</sup> The equations were prepared using ethyl decanoate or methyl palmitate for internal standard, respectively. <sup>d</sup> These values were calculated from the ratio in GC peak area of (*R*)-(-)-linalool and the (*S*)-isomer in the jasmine tea infusion in Table 4.

tea infusion by diluting the mixture with water until the odor intensity of each matched by a sensory evaluation. A preliminary experiment had revealed that the lack of any compound among these six potent odorants reduced the jasmine tea flavor in the model solution. In particular, the absence of methyl anthranilate or (*R*)-(-)-linalool markedly reduced the characteristic jasmine tea aroma. These results showed that both (*R*)-(-)-linalool and methyl anthranilate were very important constituents of jasmine tea. Therefore, in this study, the odor quality of the model mixture composed of the six potent odorants was examined and compared with that of the jasmine tea infusion by a sensory analysis. Additionally, to investigate whether the absolute configuration of linalool contributed to the odor of the jasmine tea infusion, two more model mixtures were prepared. Model mixture I contained (*R*)-(-)-linalool dominantly in excess of the (*S*)-isomer, and the other five compounds were also included in this model solution in the same ratio as that of the jasmine tea infusion. The concentration was then adjusted to be about fifty times greater than that of the jasmine tea infusion to provide an equal odor intensity between the infusion and the model mixture on the basis of the sensory evaluation already described (Table 5). The composition of model mixture II was the same as that of model I, except that (*S*)-(+)-linalool was dominantly in excess of (*R*)-(-)-linalool and the concentration was half that of model I (Table 5). This concentration adjustment was made because the intensity of the (*S*)-isomer was stronger than that of the (*R*)-isomer at this concentration, and all the panelists requested this reduction in concentration to evaluate the quality of the model. Models I and II and the original jasmine tea infusion were evaluated at the same time by the 15 panelists on a scale of 15 cm for the seven attributes (Table 1). Each sample was served at 10 °C to reproduce the temperature of the PET bottled or canned tea drink. The results are presented in Figure 3 and Table 1, indicating that the odor of the jasmine tea infusion was closely duplicated by model I. The panelists detected the strong floral and sweet notes in both solutions, and there were no significant differences in other attributes between model I and the original jasmine tea infusion. The floral and sweet notes in model II, however, were significantly less intense than those in the original jasmine tea infusion and model I. The woody, bitter, refreshing and mild notes were significantly more

**Figure 3.** Odor profiles of the original jasmine tea infusion and aroma model mixtures composed of six odor-active compounds. The composition of each model mixture is shown in Table 5; \*  $p \leq 0.05$ .

intense in model II than in model I. Consequently, the aroma profile of the jasmine tea infusion could not be reproduced by model II.

In conclusion, (*R*)-(-)-linalool and methyl anthranilate were found to be the most important odor-active compounds in jasmine tea, with the absolute configuration of linalool definitively influencing the odor profile. Minor components such as (*E*)-2-hexenyl hexanoate, lactones, and furaneol also supported the basic flavor character of jasmine tea with their green or sweet note, because when any of these compounds was absent from the model mixture, the odor profile was different from that of jasmine tea.

#### LITERATURE CITED

- Hara, Y.; Luo, S.; Wickremasinghe, L. R.; Yamanishi, T. Processing of tea IV. *Food Rev. Int.* **1995**, *11*, 409–434.
- Yamanishi, T. Aroma of Chinese scented green tea. In *Frontiers of Flavor*. Proceedings of 5th International Flavor Conference, Porto Karras, Chalkidiki, Greece, 1–3 July 1987; Charalambous, G., Ed.; Elsevier Science Publishers B. V.: Amsterdam, 1988; pp 181–190.
- Guth, H.; Grosch, W. Identification of potent odourants in static headspace samples of green and black tea powders on the basis of aroma extract dilution analysis (AEDA). *Flavour Fragrance J.* **1993**, *8*, 173–178.
- Kumazawa, K.; Masuda, H. Identification of potent odorants in Japanese green tea (sen-cha). *J. Agric. Food Chem.* **1999**, *47*, 5169–5172.

- (5) Guth, H.; Grosch, W. Identification of the character impact odorants of stewed beef juice by instrumental analyses and sensory studies. *J. Agric. Food Chem.* **1994**, *42*, 2862–2866.
- (6) Mosandl, A. Chirality in flavor chemistry – recent developments in synthesis and analysis. *Food Rev. Int.* **1988**, *4*, 1–43.
- (7) Ullrich, F.; Grosch, W. Identification of the most intense volatile flavour compounds formed during autoxidation of linoleic acid. *Z. Lebensm. Unters. Forsch.* **1987**, *4*, 277–282.
- (8) Kobayashi, A.; Kawamura, M.; Yamamoto, Y.; Shimizu, K.; Yamanishi, T. Methyl epijasmone in the essential oil of tea. *Agric. Biol. Chem.* **1988**, *52* (9), 2299–2303.
- (9) Shimoda, M.; Sasaki, H.; Doi, Y.; Kameda, W.; Osajima, Y. Characterization of abstract terms for odor-description of food products. *Nippon Shokuhin Kogyo Gakkaishi* **1989**, *36*, 7–16.
- (10) Shimoda, M.; Tsukamoto, Y.; Ito, H.; Doi, Y.; Kameda, W.; Osajima, Y. Preparation and evaluation of odor reference solution. *Nippon Shokuhin Kogyo Gakkaishi*, **1991**, *38*, 371–376.
- (11) Morita, K.; Kubota, K.; Aishima, T. Sensory characteristics and volatile components in aromas of boiled prawns prepared according to experimental designs. *Food Res. Int.* **2001**, *34*, 473–481.
- (12) Hara, Y.; Luo, S.; Wickremasinghe, L. R.; Yamanishi, T. Flavor of tea VIII. *Food Rev. Int.* **1995**, *11*, 477–525.
- (13) Musalam, Y.; Kobayashi, A.; Yamanishi, T. Aroma of Indonesian jasmine tea. In *Flavors and Fragrances: A World Perspective*. Proceedings of 10th International Congress of Essential Oil; Lawrence, M. B., Mookherjee, D. B., Willis, J. B., Eds.; Fragrances and Flavors: Washington, DC, 1986; pp 659–668.
- (14) Joulain, D. Cryogenic vacuum trapping of scents from temperate and tropical flowers. In *Bioactive Volatile Compounds from Plants*. ACS Symposium Series 525; Teranishi, R., Buttery, G. R., Sugisawa, H., Eds.; American Chemical Society: Washington, DC, 1993; pp 187–204.
- (15) Mookherjee, D. B.; Trenkle, W. R.; Wilson, A. R. The chemistry of flowers, fruits and spices: live vs. dead a new dimension in fragrance research. *Pure Appl. Chem.* **1990**, *62* (7), 1357–1364.
- (16) He, C.; Liang, Z.; Liu, H. Study on chemical constituents of jasmine absolute extracted by supercritical carbon dioxide. *Nat. Prod. Res. Dev.* **1998**, *11* (3), 53–57.
- (17) Nose, M.; Nakatani, Y.; Yamanishi, T. Studies on the flavor of green tea part IX. Identification and composition of intermediate and high boiling constituents in green tea flavor. *Agric. Biol. Chem.* **1971**, *35*, 261–271.
- (18) Kawakami, M.; Yamanishi, T. Formation of aroma components in roasted or pan-fired green tea. *Nippon Nôgeikagaku Kaishi*. **1999**, *73*, 893–906.
- (19) Kawakami, M.; Luo, S. Aroma composition of various Chinese teas using a brewed extraction method. *J. Ibaraki Christian University* **2000**, *34*, 89–99.
- (20) Yamanishi, T.; Kosuge, M.; Tokotomo, Y.; Maeda, R. Flavor constituents of pouchong tea and a comparison of the aroma pattern with jasmine tea. *Agric. Biol. Chem.* **1980**, *44*, 2139–2142.
- (21) Pittet, O. A.; Rittersbacher, P.; Muralidhara, R. Flavor properties of compounds related to maltol and isomaltol. *J. Agric. Food Chem.* **1970**, *18*, 929–933.
- (22) Renold, W.; Näf-Müller, R.; Keller, U.; Willhalm, B.; Ohloff, G. An investigation of the tea aroma part I. New volatile black tea constituents. *Helv. Chim. Acta* **1974**, *57*, 143–144.
- (23) Yamanishi, T. Flavor of green tea. *JARQ* **1978**, *12*, 206–210.
- (24) Tokitomo, Y.; Ikegami, M.; Yamanishi, T. Effects of withering and mass-rolling processes on the formation of aroma components in pouching type semi-fermented tea. *Agric. Biol. Chem.* **1984**, *48*, 87–91.
- (25) Wang, D.; Ando, K.; Morita, K.; Kubota, K.; Kobayashi, A. Optical isomer of linalool and linalool oxides in tea aroma. *Biosci. Biotechnol. Biochem.* **1994**, *58* (11), 2050–2053.
- (26) Kobayashi, A.; Kubota, K.; Yano, M. Formation of some volatile components of tea. In *Bioactive Volatile Compounds from Plants*; ACS Symposium Series 525; Teranishi, R., Buttery, G. R., Sugisawa, H., Eds.; American Chemical Society: Washington, DC, 1993; pp 49–56.
- (27) Pallado, P.; Tassinato, G.; D’Alpaos, M.; Traldi, P. Gas chromatography/mass spectrometry in aroma chemistry: a comparison of essential oils and flavors extracted by classical and supercritical techniques. *Rapid Commun. Mass Spectrom.* **1997**, *11*, 1335–1341.
- (28) Casabianca, H.; Grajji, B. J.; Faugier, V.; Fleig, F.; Grenier, C. Enantiomeric distribution studies of linalool and linalyl acetate. *J. High Resolut. Chromatogr.* **1998**, *21*, 107–112.
- (29) Tamogami, S.; Awano, K.; Kitahara, T. Analysis of the enantiomeric ratios of chiral components in absolute jasmine. *Flavour Fragrance J.* **2001**, *16*, 161–163.
- (30) Kawakami, M.; Ganguly, N. S.; Banerjee, J.; Kobayashi, A. Aroma composition of oolong tea and black tea by brewed extraction method and characterizing compounds of Darjeeling tea aroma. *J. Agric. Food Chem.* **1995**, *43*, 200–207.
- (31) Padrayuttawat, A.; Yoshizawa, T.; Tamura, H.; Tokunaga, T. Optical isomers and odor thresholds of volatile constituents in *Citrus sudachi*. *Food Sci. Technol. Int. Tokyo*, **1997**, *3*, 402–408.

---

Received for review March 4, 2002. Accepted June 1, 2002.

JF020282H