

Analysis of Green and Yellow Yuzu Peel Oils (*Citrus junos Tanaka*). Novel Aldehyde Components with Remarkably Low Odor Thresholds

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Yuzu (*Citrus junos Tanaka*) is a kind of sour orange, which originated in central China. The fruit has a pleasant and fresh odor and has been used widely as a raw material for vinegar and seasoning in Japan. In general, it is well-known that aldehyde components play an important role of odor in citrus essential oil because of their low odor threshold. Therefore, the aldehydes of yuzu peel oil were isolated and analyzed by GC, GC-MS, and NMR. Twenty-two aldehydes in yuzu peel oil of yellow and green maturity were identified. Three of them, 6-methyloctanal, 8-methylnonanal, and 8-methyldecanal, have not been previously reported to occur in nature. The three novel aldehydes were synthesized and discussed in more detail. Further odor distinction between green maturity and yellow maturity is also discussed.

INTRODUCTION

There are some citrus species that are cultivated and utilized as the raw materials for juice and essential oils in Japan. In particular, we were much interested in yuzu because of its very pleasant aroma. Many investigations have been done on the constituents of yuzu peel oil. Shinoda et al. (1970) characterized some constituents in cold pressed yuzu oil by means of fractional distillation, extraction, silica gel column chromatography, IR, and GC-MS. In addition, they identified a number of components found in amounts of less than 0.01%. Matsuura et al. (1977) isolated a novel macrolide, *cis*-9-dodecen-12-olide, from cold pressed yuzu peel oil. Ohta and Osajima (1983) examined the yuzu juice by GC-MS. Watanabe et al. (1983) identified 124 compounds by GC and GC-MS. Also, a novel monoterpene diol was isolated by distillation, silica gel column chromatography, and preparative GC and presumed to be 2,6-dimethyl-2,7-octadien-1,6-diol by means of IR and MS and further identified as the same by synthesis. Sawamura et al. (1986) identified β -sinensal in both yuzu peel oil of green maturity and that of yellow maturity, which is regarded as an important component in citrus essential oil.

The above papers have reported that the characteristic odor of yuzu peel oil seems to be dependent on the minor components of various alcohols and aldehydes. In addition, many other papers have reported that aldehyde components contribute to the odor of citrus peel oil. Therefore, this paper deals with the isolation and characterization of aldehyde components in yuzu peel oil by means of our own unique technique, which is a selective separation using physical and chemical properties. Unknown aldehyde components which had not been previously reported were analyzed by GC and GC-MS. Their aldehydes were synthesized to confirm their chemical structure and to study their odor characteristics. Moreover, odor differences between green mature and yellow mature yuzu peel oils were studied from the point of aldehyde components.

EXPERIMENTAL PROCEDURES

Procedure. The yuzu used were green fruit collected at Fukuoka prefecture in Japan in October. Thirty kilograms of peel were prepared from 80 kg of the fruits. These yuzu peels

were extracted twice with 20 kg of methylene chloride. The resulting extracts were combined to filter. After removal of the solvent, subsequent distillation under reduced pressure of 2 mmHg gave 195 g (yield 0.25%) of orange colored oil, having an odor similar to that of the fresh fruits, and 26 g of residue, having a woody, earthy, and sweet odor. Fifty grams of the distillate was subjected to fractionation by means of silica gel column chromatography, with hexane and methylene chloride-diethyl ether (1:1 v/v) as eluted. The procedure was repeated four times, giving hydrocarbons (178.2 g, ca. 95.7%) and non-hydrocarbons (8.0 g, ca. 4.3%).

At first, 5 g of non-hydrocarbon fraction was added to 3.0 g of saturated aqueous sodium bisulfite solution and stirred for 3 h at room temperature. After separation of the organic layer, the aqueous layer, containing the sodium salts of aldehydes, was washed three times with methylene chloride to remove unreactive organic compounds, neutralized with 1 N sodium hydroxide solution, and extracted with methylene chloride. The organic layer was dried over anhydrous sodium sulfate, and the solvent was removed to give 0.077 g of aldehyde fraction A.

On the other hand, 3 g of non-hydrocarbon fraction was added to 50 mL of 50% 1.2 N sodium 6-aminohexanoate in ethanol. After separation of the organic layer, the aqueous layer was washed with 10% hydrochloric acid and extracted with methylene chloride. The organic layer was dried over anhydrous sodium sulfate, and removal of the solvent gave 0.057 g of aldehyde fraction B. The whole procedure for the isolation of aldehyde fraction of yuzu peel oil is given schematically in Figure 1. The same procedure was carried out for yellow ripened yuzu.

Apparatus. IR spectra were measured as a smear on sodium chloride plate with a Perkin-Elmer 1710 spectrometer. ^1H NMR spectra were obtained on a JEOL FX-90 (90 MHz) FT NMR spectrometer. All samples were dissolved in CDCl_3 with Me_4Si as internal standard. Quantitative analyses of aldehyde components in yuzu oil were determined by peak area percent on two Hewlett-Packard 5890 gas chromatographs equipped with FID. Separations were performed on a 50-m glass capillary column (0.2 mm i.d.) coated with HP-20M (Carbowax-20M) or Ultra 2 (cross-linked 5% phenylmethylsilicone). The temperature program was from 60 to 230 °C with a rate of 2 °C/min on HP-20M and from 70 to 240 °C with a rate of 2 °C/min on Ultra 2; carrier gas flow was 1 mL of He/min. A Hewlett-Packard 5985A gas chromatography-mass spectrometer coupling system was used. The glass capillary column and other GC conditions were the same as above. Sniffing or preparative gas chromatography was performed on Hewlett-Packard 5890 with TCD. Separations were performed on a

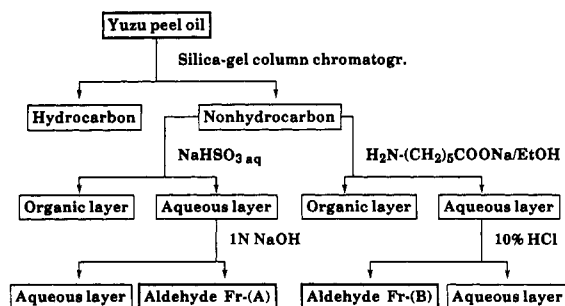


Figure 1. Treatment of yuzu peel oil.

30-m glass megabore column with DB-Wax and temperature programmed from 60 to 210 °C with a rate of 2 °C/min.

General Synthetic Methods. *Materials.* Commercial reagents were utilized without further purification. Diethyl ether and acetonitrile were distilled with calcium hydride.

Ethyl 8-Methyl-6-decenoate (3c). Ethyl 6-bromohexanoate (1b) (22.3 g, 100 mmol) and triphenylphosphine (27.5 g, 105 mmol) in acetonitrile (200 mL) were refluxed for 30 h. After evaporation of the solvent (100 mL), 2-methylbutyraldehyde (8.6 g, 100 mmol) was added to the reaction mixture and 60% oil-dispersed sodium hydride (4.0 g, 100 mmol) was added slowly with stirring under nitrogen atmosphere. The reaction temperature was kept below 40 °C by occasional cooling. The reaction mixture was stirred at room temperature overnight. Then, this mixture was hydrolyzed by addition of water (50 mL) and was extracted with *n*-hexane (3 × 50 mL). The combined extracts were washed with water and dried over anhydrous sodium sulfate. After removal of the solvent, the residue was distilled under reduced pressure to give ethyl 8-methyl-6-decenoate (3c), 13.8 g (65%).

8-Methyl-6-decen-1-ol (4c). A solution of the ethyl 8-methyl-6-decenoate (3c) (12.7 g, 60 mmol) in dry ether (60 mL) was added to lithium aluminum hydride (1.1 g, 30 mmol) in dry ether (200 mL) under dry nitrogen with cooling in an ice bath. After stirring for 2 h at room temperature, the reaction mixture was poured onto crushed ice and extracted with diethyl ether (3 × 100 mL). The combined extracts were dried over anhydrous sodium sulfate. After removal of the solvent, the desired alcohol (4c) was obtained, 10.2 g (96%).

8-Methyldecanol (5c). A solution of the alkenol (4c) (8.8 g, 50 mmol) in dry ethanol (50 mL) was hydrogenated over 5% palladium/carbon (1.3 g) at ordinary pressure and temperature. After 1 h, the theoretical amount of hydrogen was consumed. The solvent was evaporated, and purification by distillation gave the desired alkenol (5c), 8.3 g (98%).

8-Methyldecanal (6c). A suspension of pyridinium chlorochromate (PCC) (12.9 g, 60 mmol) in dry methylene chloride (120 mL) was stirred under nitrogen atmosphere at room temperature. 8-Methyldecanol (5c) (6.9 g, 40 mmol) was rapidly added, and the suspension was stirred at room temperature. After 2 h, the black mixture was diluted with diethyl ether (600 mL), and the solution was decanted. The residue was extracted three times with diethyl ether (3 × 30 mL). The combined extracts were passed through florisil and concentrated. Final purification was accomplished by distillation under reduced pressure to give 6c, 6.1 g (89%).

6-Methyloctanal (6a) and 8-methylnonanal (6b) were synthesized following the procedure shown in Figure 6. Spectral data of synthesized aldehydes and their intermediates are shown in Table I.

Odor Threshold Determinations. The odor thresholds were determined by triangle comparison, and panels comprised 10 perfumers. Three methyl-branched aldehydes were dissolved in purified limonene (99%). An equivalent amount of limonene was added to the control sample in each case. The results were interpolated from the regression curve of the log of concentration vs correct answer probability at each different concentration. Odor thresholds at the concentration of 50% probability were taken as the odor threshold in each case. For comparison, *n*-nonanal, *n*-decanal, and *n*-undecanal, which were their unbranched isomers, were measured in the same manner.

RESULTS AND DISCUSSION

Separation of Aldehyde Components. In this paper, aldehyde components were separated from nonhydrocarbon fraction by use of sodium bisulfite and sodium 6-aminohexanoate. Sodium bisulfite is a well-known reagent for separation of aldehyde. It reacts predominantly with straight-chain aldehydes, rarely with unsaturated ones, e.g., citral. On the other hand, sodium 6-aminohexanoate is inclined to form an aqueous Schiff base with unsaturated or aromatic aldehydes rather than saturated ones. Therefore, unsaturated or aromatic aldehydes were separated by use of sodium 6-aminohexanoate. This separation was a modification of method reported by Ohta and Okamoto (1980).

Analyses of Aldehyde Fractions. Figures 2 and 3 show gas chromatograms of aldehyde fractions A and B in green yuzu peel oil, respectively. These aldehyde components were identified by GC and GC-MS. Among unknown aldehyde components the peaks A-5, A-7, B-5, and A-9 had interesting odor, which were regarded as important constituents by sniffing gas chromatography. The odors of three peaks were as follows: peak A-5 (unknown I), powerful, fatty, citrus green; peak A-7, B-5 (unknown II), powerful, soft sweet, mild green, diffusive; peak A-9 (unknown III), mild, fatty, sweet, citrus, diffusive.

Structure Elucidations of Unknowns I-III. The clue to the structure of unknown II was given by its GC retention time, NMR spectrum, and mass spectrum. The GC retention time of unknown II was between those of *n*-nonanal and *n*-decanal in both polar and nonpolar columns. This evidence suggests that the unknown was a *n*-decanal isomer having the molecular weight of 156. The NMR spectrum of unknown II, which was collected by preparative GC, gave a doublet signal at 0.87 ppm as shown in Figure 4. In addition, a relative abundance of the peak at *m/e* 123 (*M* - 33) was observed to be much larger than that of *n*-decanal as shown in Figure 5. This peak of *M* - 33 can account for a loss of H₂O and subsequent loss of a methyl group. These observations show that the molecular terminal consists of an iso form. Therefore, unknown II was presumed to be isodecanal (8-methyl-nonanal).

As for unknowns I and III, their emissions were too slight to be collected by preparative GC, and NMR spectra could not be taken. Information from GC retention time in both polar and nonpolar columns suggested that unknowns I and III were *n*-nonanal isomer (*M_r* = 142) and *n*-undecanal isomer (*M_r* = 170), respectively. Also from mass spectra of unknowns I and III, relative abundances of the peaks *M* - 33 and *M* - 47 were observed in common which are much larger than those of two corresponding aldehydes *n*-nonanal and *n*-undecanal. The peaks of *M* - 33 and *M* - 47 can account for a loss of water and a subsequent loss of a methyl or ethyl group. From these observations, unknowns I and III were presumed to be 6-methyloctanal and 8-methyldecanal, respectively. As it was impossible to get more determinative information from these data and these compounds were not available, we decided to synthesize these three aldehydes.

Syntheses of Three Terminal Branched Aldehydes and Identifications. A C-C bond formation of terminal branched carbon skeleton is derived from several reactions, which are the Grignard, coupling, and Wittig reactions and acetylene addition. Taking into account the availability of starting material, formation of byproduct, and reactivity, the Wittig reaction was considered to be well suited for laboratory-scale preparation in this case. As shown in Figure 6, these three aldehydes were

Table I. Boiling Point and Spectral Data of Synthesized Aldehydes and Their Intermediates

no.	component	boiling point and spectral data [IR, cm^{-1} ; MS, m/e (rel intense); ^1H NMR δ]
3a	ethyl 6-methyl-4-octenoate	bp 67 °C/4 mmHg ^1H NMR: δ 0.83 (t, 3 H, $J = 6.8$ Hz, CHCH_2CH_3), 0.93 (d, 3 H, $J = 6.6$ Hz, CHCH_3), 1.26 (t, 3 H, $J = 7.0$ Hz, OCH_2CH_3), 1.17–1.40 (m, 2 H, CHCH_2CH_3), 2.25–2.50 (m, 5 H), 4.12 (q, 2 H, $J = 7.0$ Hz, OCH_2), 5.15–5.35 (m, 2 H, $\text{CH}=\text{CH}$)
3b	ethyl 8-methyl-6-nonenoate	bp 64 °C/3 mmHg IR: 3344 (s), 2934 (s), 1656 (m), 1464 (s), 1378 (m), 1362 (m) ^1H NMR: 0.93 (d, 6 H, $J = 6.6$ Hz, $\text{CH}_3 \times 2$), 1.24 (t, 3 H, $J = 7.1$ Hz, OCH_2CH_3), 1.35–1.80 (m, 4 H), 1.94–2.15 (m, 2 H), 2.30 (t, 2 H, $J = 7.0$ Hz, CH_2), 2.35–2.75 (m, 1 H, CH), 4.12 (q, 2 H, $J = 7.1$ Hz, OCH_2CH_3), 5.14–5.25 (m, 2 H, $\text{CH}=\text{CH}$)
3c	ethyl 8-methyl-6-decenoate	bp 75 °C/3 mmHg ^1H NMR: 0.83 (t, 3 H, $J = 7.0$ Hz, CH_2CH_3), 0.92 (d, 3 H, $J = 6.6$ Hz, CHCH_3), 1.25 (t, 3 H, $J = 7.2$ Hz, OCH_2CH_3), 1.20–1.75 (m, 6 H), 2.01 (d, 2 H, CH_2), 2.30 (t, 2 H, $J = 7.2$ Hz, CH_2), 2.10–2.45 (m, 1 H, CHCH_3), 4.12 (q, 2 H, $J = 7.2$ Hz, OCH_2CH_3), 4.98–5.42 (m, 2 H, $\text{CH}=\text{CH}$)
4a	6-methyl-4-octen-1-ol	bp 70 °C/3 mmHg IR: 3330 (s), 2959 (s), 1655 (w), 1456 (m), 1061 (s) ^1H NMR: 0.89 (t, 3 H, $J = 5.3$ Hz, CH_2CH_3), 0.94 (d, 3 H, $J = 5.3$ Hz, CHCH_3), 1.12–1.36 (m, 2 H, CH_2CH_3), 1.54–1.72 (m, 2 H), 2.02–2.14 (m, 2 H, $\text{CH}=\text{CHCH}_2$), 2.30–2.50 (m, 1 H, CH), 3.44–3.65 (m, 2 H, CH_2OH), 5.00–5.46 (m, 2 H, $\text{CH}=\text{CH}$)
4b	8-methyl-6-nonen-1-ol	bp 82 °C/3 mmHg IR: 3344 (s), 2932 (s), 1656 (m), 1462 (s), 1378 (m), 1362 (m) ^1H NMR: 0.94 [d, 6 H, $J = 6.6$ Hz, $\text{CH}(\text{CH}_3)_2$], 1.25–1.75 (m, 7 H), 1.92–2.15 (m, 2 H), 2.45–2.70 [m, 1 H, $\text{CH}(\text{CH}_3)_2$], 3.63 (t, 2 H, $J = 6.2$ Hz, CH_2OH), 5.04–5.34 (m, 2 H)
4c	8-methyl-6-decen-1-ol	bp 92 °C/3 mmHg IR: 3329 (s), 3030 (w), 2958 (s), 1655 (w), 1337 (m) ^1H NMR: 0.82 (t, 3 H, $J = 7.2$ Hz, CH_2CH_3), 0.92 (d, 3 H, $J = 6.4$ Hz, CHCH_3), 1.14–1.70 (m, 9 H), 1.92–2.16 (m, 2 H, CH_2), 2.10–2.25 (m, 1 H, CH), 3.30 (t, 2 H, $J = 7.2$ Hz, CH_2OH), 4.95–5.22 (m, 2 H, $\text{CH}=\text{CH}$)
5a	6-methyloctanol	bp 73 °C/2.5 mmHg IR: 3359 (s), 2959 (s), 1463 (s), 1378 (m), 1059 (s) ^1H NMR: 0.81–0.92 (m, 6 H, $\text{CH}_3 \times 2$), 1.12–1.68 (m, 12 H), 3.58 (t, 2 H, CH_2OH)
5b	8-methylnonanol	bp 87 °C/3 mmHg IR: 3340 (s), 2927 (s), 1467 (m), 1384 (m), 1367 (m) ^1H NMR: 0.85 [d, 6 H, $J = 6.2$ Hz, $\text{CH}(\text{CH}_3)_2$], 1.15–1.70 (m, 14 H), 3.62 (t, 2 H, CH_2OH)
5c	8-methyldecanol	bp 93 °C/3 mmHg IR: 3340 (s), 2926 (s), 1463 (m), 1378 (m), 1058 (s) ^1H NMR: 0.80–0.95 (m, 6 H), 1.18–1.70 (m, 16 H), 3.67 (t, 2 H, CH_2OH)
6a	6-methyloctanal	bp 52 °C/3 mmHg IR: 2932 (s), 1729 (s), 1463 (s), 1378 (m) MS: 57 (100), 95 (77), 69 (50), 96 (45), 70 (39), 56 (35), 41 (34), 55 (28), 67 (24), 43 (18) ^1H NMR: 0.72–0.92 (m, 6 H, $\text{CH}_3 \times 2$), 1.08–1.44 (m, 7 H), 1.52–1.72 (m, 2 H), 2.43 (t, 2 H, CH_2CHO), 9.76 (t, 1 H, $J = 1.8$ Hz, CHO)
6b	8-methylnonanal	bp 66 °C/3 mmHg IR: 2950 (s), 1730 (s), 1470 (m), 1385 (m), 1375 (m) MS: 57 (100), 82 (65), 43 (57), 69 (55), 55 (52), 56 (49), 41 (48), 72 (42), 81 (38), 95 (37) ^1H NMR: 0.87 [d, 6 H, $J = 6.2$ Hz, $\text{CH}(\text{CH}_3)_2$], 1.20–1.72 (m, 11 H), 2.43 (t, 2 H, $J = 7.5$ Hz, CH_2CHO), 9.76 (t, 1 H, $J = 1.8$ Hz, CHO)
6c	8-methyldecanal	bp 75 °C/3 mmHg IR: 2930 (s), 1730 (s), 1460 (s) MS: 57 (100), 81 (97), 70 (93), 55 (77), 71 (74), 82 (61), 67 (55), 123 (53), 95 (51), 97 (45) ^1H NMR: 0.80–0.93 (m, 6 H, $\text{CH}_3 \times 2$), 1.14–1.72 (m, 13 H), 2.43 (t, 2 H, $J = 6.6$ Hz, CH_2CHO), 9.76 (t, 1 H, $J = 1.8$ Hz, CHO)

synthesized from C–C bond formation by using the Wittig reaction, followed by conversion of the functional group. GC retention times of the three synthesized aldehydes were identical with those of unknowns I–III in both polar and nonpolar columns using linear aldehydes (C_8 – C_{11}) as internal standard. Mass spectral identifications of the three unknown aldehydes were verified by comparison with the synthesized authentic samples.

Odor Characterization of 8-Methylnonanal, 8-Methyldecanal, and 6-Methyloctanal. Characteristics of the above three synthesized aldehydes were compared with those of corresponding normal chain aldehydes which were contained in yuzu peel oil as perfume raw materials. The sensory contributions of these synthesized aldehydes to the odor pattern of a complex mixture of yuzu substances were investigated. The relative importance of each

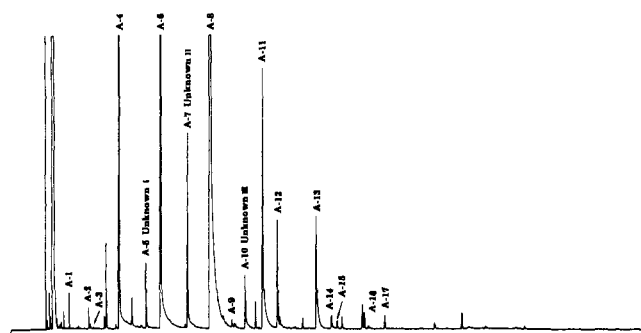


Figure 2. Gas chromatogram [50 m capillary column (0.2 mm i.d.); Carbowax 20M] of aldehyde fraction A in green yuzu peel oil.

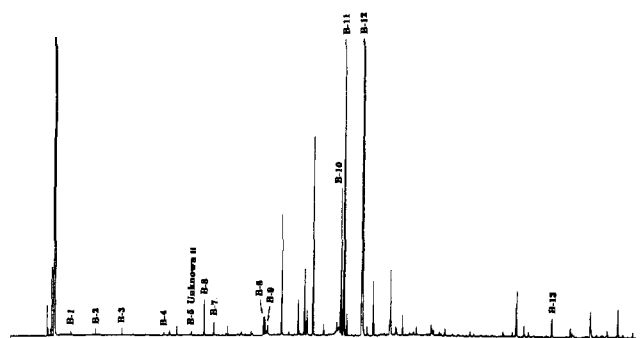


Figure 3. Gas chromatogram [50 m capillary column (0.2 mm i.d.); Carbowax 20M] of aldehyde fraction B in green yuzu peel oil.

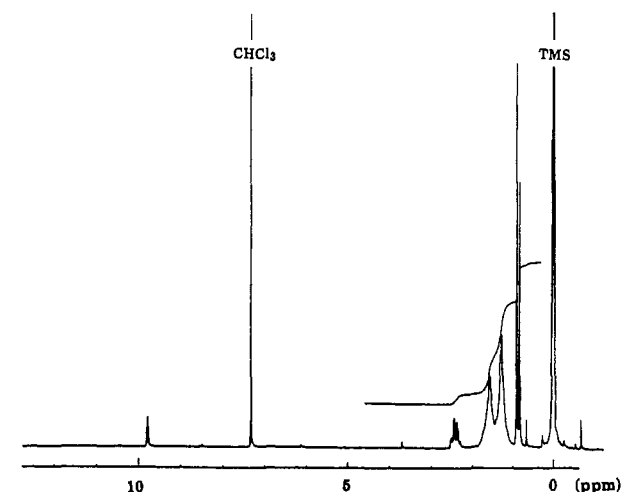


Figure 4. ¹H NMR spectrum of unknown II.

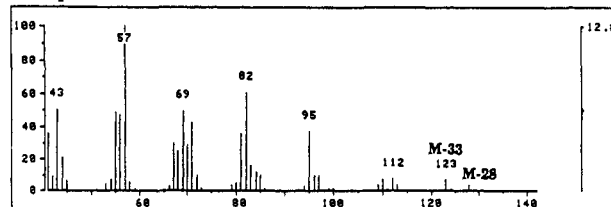
constituent will be expressed in its threshold and odor value, which is defined as the ratio of the concentration of a constituent to its threshold concentration. The following equation was described by Ohloff (1977).

$$\text{odor value} = \text{concn in oil} / \text{threshold concn}$$

The odor value shows how much the actual concentration of a substance exceeds its threshold. Differences in odor values between constituents may therefore be taken as an index of effectiveness.

Figure 7 shows an example of the determination of odor threshold, and Table II shows all of the results. The odor thresholds of three new aldehydes in limonene were 20–2000 times lower than those of corresponding aldehydes. Odor thresholds of these aldehydes are nearly equal to that of 2,4-nonadienal, which is known as the aldehyde having the lowest threshold. Further, it was found that the odor values of the new synthesized aldehydes were

Mass Spectrum of Unknown II



Mass Spectrum of n-Decanal (Mw = 156)

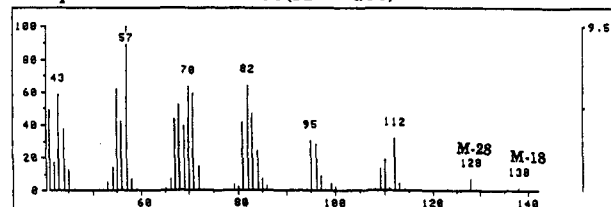


Figure 5. Comparison of mass spectrum of unknown II with that of *n*-decanal.

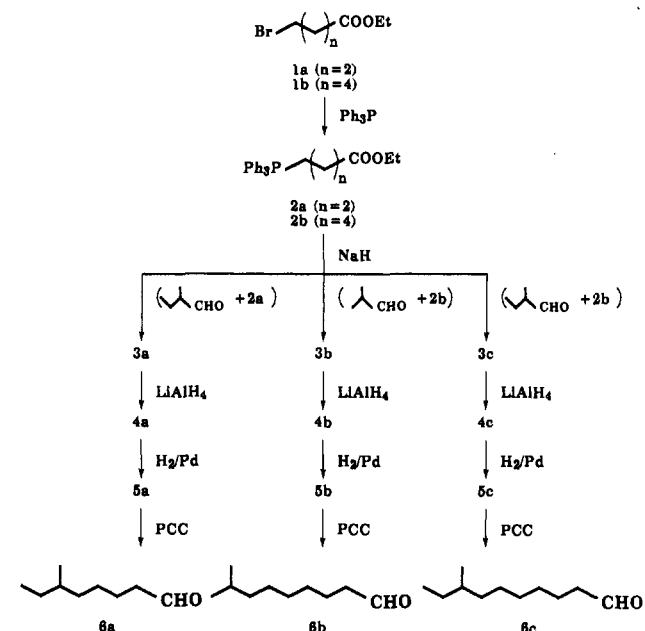


Figure 6. Syntheses of methyl-branched aldehydes.

approximately 1–100 times higher than those of *n*-nonanal, *n*-decanal, and *n*-undecanal, respectively. This can account for the fact that the sensory contribution of synthesized aldehydes plays the most important role among aldehyde components in yuzu peel oil. In other words, 6-methyloctanal, 8-methylnonanal, and 8-methyldecanal could be regarded as one of the most important components of yuzu peel oil. Therefore, we attempted to compare the odor of synthesized yuzu essential oil containing the three new aldehydes with that not containing them. The comparison confirmed the three new aldehydes contribute to fresh green, bitter citrus, and sweet notes which were part of the essential odor of yuzu. Twenty-two aldehyde compounds from green and yellow yuzu were identified as shown in Table III. Among these aldehydes, eight compounds were identified in yuzu peel oil for the first time. 6-Methyloctanal, 8-methylnonanal, and 8-methyldecanal have not been reported in any literature of natural essential oil. In addition, they were recognized to have extremely low odor thresholds. *cis*-4-Decenal, *trans*-2-decenal, and *p*-menthen-9-al, which were known to have reasonably low odor thresholds, were also identified in yuzu peel oil. The concentration of alde-

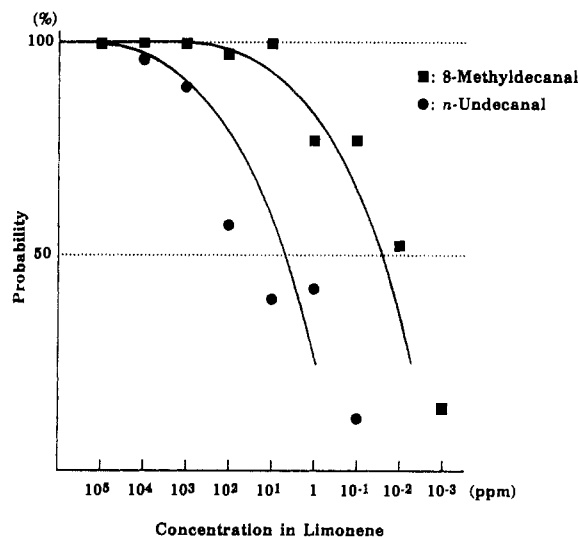


Figure 7. Determination of odor threshold of C₁₁ aldehydes.

Table II. Comparison of Odor Thresholds (OT) and Odor Values of New Aldehydes with Known Aldehydes in Yuzu Peel Oil

component	concn, %	OT, ppm	odor value
<i>n</i> -nonanal	0.0100	5.000	20
<i>n</i> -decanal	0.0510	10.000	51
<i>n</i> -undecanal	0.0100	5.000	20
6-methyloctanal	0.0005	0.250	20
8-methylnonanal	0.0025	0.005	5000
8-methyldecenal	0.0015	0.010	1500

hyde components in total weight was about 0.15%. This value seems to be very low in comparison with other citrus essential oils. However, odor thresholds of most of the aldehyde components included in yuzu peel oil are lower than that of any other aldehydes.

As for the odor distinction between green and yellow mature yuzu, the odor of green mature yuzu peel oil was more fresh, bitter, and metallic than that of yellow mature yuzu peel oil. This distinction was presumed to be derived from the difference in the ratio of individual aldehyde components, which were *trans*-2-decenal, *trans*-2-dodecenal, citronellal, and 2,4-decadienal. These unsaturated aldehydes were known to have lower odor thresholds than those of saturated ones. It was found that the riper the yuzu fruit becomes, from green to yellow, the more the contents of unsaturated aldehydes increase. For these reasons, complex and delicate combinations of many

Table III. Components of Green and Yellow Yuzu Peel Oils

peak no.	component	green, %	yellow, %
A-1, B-1	<i>n</i> -hexanal	tr	tr
A-2	<i>trans</i> -2-hexenal	tr	tr
A-3, B-2	<i>n</i> -heptanal	tr	tr
A-4, B-3	<i>n</i> -octanal	0.0068	0.0115
A-5	6-methyloctanal ^a	0.0005	0.0006
A-6, B-4	<i>n</i> -nonanal	0.0100	0.0114
A-7, B-5	8-methylnonanal ^a	0.0025	0.0021
B-6	citronellal	tr	0.0009
A-8, B-7	<i>n</i> -decanal	0.0510	0.0480
A-9	<i>cis</i> -4-decenal ^a	0.0050	0.0020
A-10	8-methyldecenal ^a	0.0015	0.0007
B-8	<i>p</i> -menthen-9-al ^a	tr	tr
A-11, B-9	<i>n</i> -undecanal	0.0100	0.0072
A-12	<i>trans</i> -2-decenal ^a	tr	0.0064
A-13	<i>n</i> -dodecanal	0.0040	0.0091
A-14	<i>trans</i> -2-undecenal ^a	tr	tr
A-15	cumin aldehyde	tr	0.0004
B-10,12	2,4-decadienal	0.0070	0.0152
B-11	perillaldehyde	0.0190	0.0034
A-16	<i>n</i> -tridecanal ^a	tr	0.0002
A-17	<i>trans</i> -2-dodecenal	tr	0.0008
B-13	β -sinensal	tr	tr

^a Not reported earlier as a constituent of yuzu peel oil.

characteristic aldehydes are very important for the odor of yuzu peel oils.

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