

Quality Properties of Seasoned-Dried Pacific Saury Treated with Liquid Smoke

1. Volatile Flavor Compounds in Commercial Liquid Smokes

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(Received September 2001, Accepted December 2001)

In order to identify of volatile flavor compounds and polycyclic aromatic hydrocarbons (PAHs) in commercial liquid smokes, this study was conducted to analyze volatile flavor compounds by solvent extraction and/or Purge & Trap method/GC/MSD. A total of 156 volatile flavor compounds were detected in 6 commercial liquid smokes, and these compounds were composed mainly of 12 aldehydes, 60 ketones, 7 alcohols, 14 acids, 20 esters, 24 aromatic compounds, 7 furans and 12 miscellaneous compounds. Ketones (806.6~7,573.9 $\mu\text{g/mL}$) and aromatic compounds (282.6~7,896.3 $\mu\text{g/mL}$) were more abundant than others. The PAHs known as carcinogen have not been detected in this study. The acids (422.9~4,903.1 $\mu\text{g/mL}$) was identified in relatively high concentration compared to other groups. Phenol and its derivatives among aromatic compounds were in relatively high concentration. Especially, the phenol and its derivatives including *o*-cresol, guaiacol, 4-ethylguaiacol and syringol were in higher concentration.

Key words: Liquid smoke, Polycyclic aromatic hydrocarbons (PAHs),
Volatile flavor compounds

Introduction

A long with the beginning of civilization in the west, smoking technique has been used in most of their lives as a means of preservation and flavoring since other various preservation methods have been developed (Sink, 1979). In addition, as a food additive, when liquid smoke is used for an antioxidative or antimicrobial agent, it is labeled as "Smoked" or "Naturally Smoked" (USDA-FSIS, 1996).

However, the traditional techniques of smoking foods were known to be needed huge facilities and many times and works. Moreover, the techniques were difficult not only to make equal quality of products, but also to minimize pollution problems due to excretion of smoke in the air (Lee et al., 1985;

Park et al., 1994). Recently, developments in food packaging and processing technique and extensive cold chain system have led to the reduced importance of smoking as a preservation method (Sofos et al., 1988).

Generally, liquid smoke is made by concentration of smoke generated from charcoal of broad-leaved trees, or by refinement of smoke originated from incomplete combustion of wood condensate (Park et al., 1994).

On the other hand, lots of researches about smoke and liquid smoke have been studied, especially volatile flavor compounds in liquid smoke were revealed to be composed mainly of aromatic hydrocarbons, ketones and acids (Park et al., 1994; Guillen and Ibargoitia, 1996; Guillen and Ibargoitia, 1998). Furthermore, it is well known that phenol and its derivatives have antioxidative effect, while acids and

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formaldehyde have antimicrobial effect (Park et al., 1994).

Meanwhile, some studies (Alonge, 1988; Dungal, 1961) reported that the high incidence of stomach cancer to some people who live in Nigeria or Baltic countries has been associated with the consumption of smoked fishes, and these were investigated to direct intake of fishes treated with smoke, containing polycyclic aromatic hydrocarbons (PAHs) (Moret et al., 1999). These compounds are known as carcinogen and a major class of environmentally hazardous organic compounds, generated from incomplete combustion of some sorts of chemical fuels such as oil and coal (Wang et al., 1999). Therefore, the effects of human being health by PAHs have highly been concerned in many researches.

The objectives of this study are to identify volatile flavor compounds and simultaneously estimate whether it might be safe or not for application to the fishery processing industry through presence of PAHs in commercial liquid smokes.

Materials and Methods

Materials

Liquid smokes used were Scansmoke PB 2110 (P. Broste A/S, Denmark), Zestismoke (Hickory Specialties Inc., USA), Imported product (Backgeung Food Inc., Korea), SF-3, SF-10 and SF-26 (Donghae Hwasung Co. LTD., Japan), and these were donated from each product company and stored in a refrigerator (3°C) until used.

Analysis of Brix, total acidity and specific gravity

Five mL of sample were diluted to 100 mL with distilled water, and 20 mL of the solution diluted were transferred to 50 mL beaker and titrated with 0.1 N NaOH to pH 8.3. The total acidity was calculated as acetic acid content (g/100 g). Brix was measured by using refractometer (Atago, Japan), and specific gravity was measured by glass gravimeter.

Analysis of volatile flavor compounds

1. Solvent extraction method

One mL of liquid smoke, 50 mL of dichlorome-

thane as an extraction solvent and tridecene (75.83 µg/mL) (Aldrich, USA) as an internal standard compound were put into Erlenmeyer flask (50 mL), mixed for 15 min, and then dehydrated by using Na₂SO₄. After filtration, the solution obtained were condensed to 5 mL under N₂ gas and used for analysis. Duplicate extraction were performed on each sample.

2. Purge and trap (P&T) method

The instrument used for P&T method was HP 7695 P&T concentrator (Hewlett-Packard Co., USA). Ten µL of diluted solution and 10 µL of tridecene were taken into P&T sparger. Analysis conditions were as follows. Sample temperature, 50 °C; flow rate, 22 psi; trap, Tenax/Silica gel/Charcoal trap (HP 14-0124-303, Hewlett-Packard Co., USA); purge time, 10 min; desorption temperature, 225°C; desorption time, 5 min; cryo-focusing temperature, -150°C; cryo-injection temperature, 200°C.

3. Gas chromatography/mass spectrometry (GC/MS)

1) Analysis conditions of solvent extraction method

A 0.3 µL of extract was injected into HP 6890 GC/5973 mass selective detector (MSD) (Hewlett-Packard Co., USA) (splitless mode; helium carrier gas at 1.2 mL/min) equipped with a Ultra-2™ capillary column (50 m length × 0.2 mm i.d × 0.33 µm film thickness, Hewlett-Packard Co., USA). Oven temperature was programmed at 40°C initially (5 min hold), increased to 280°C at 3°C/min (10 min hold) and increased to 290°C (25 min) for post-run. MSD conditions were as follows: capillary direct MS interface temperature, 280°C; ion source temperature, 204°C; ionization energy, 70 eV; mass range, 33~350 amu; and electron multiplier voltage, 1400 V. Duplicate analysis were performed on each extract.

2) GC/MS conditions for Purge & Trap Method

The sample was programmed to automatically inject into GC/MSD after cryofocusing, oven temperature was programmed from 50 to 240°C at 5°C/min with initial and final hold times of 5 and 0 min, respectively, and hold time of 30 min for 280°C. MSD analysis was performed under the same con-

ditions with solvent extraction method, and triplicate analysis were performed on each sample.

4. Identification and relative abundance of volatile flavor compounds

Volatile compounds were identified by matching retention indices (RI) and mass spectra of unknown with those authentic standard compounds. Tentative identifications of each flavor compound were based on the mass library database (Hewlett-Packard Co., 1998). Relative concentrations ($\mu\text{g}/\text{mL}$) of positively identified compounds were determined using MS response factors for each compound relative to the internal standard (tridecene), and factor of all compounds appearing on total ion chromatogram was calculated as one. The peak area of coeluted compounds was minimized by Hites and Biemann method (1970).

Results and Discussion

pH, Brix, total acidity and specific gravity in commercial liquid smokes

As a results of analysis for pH, Brix, total acidity and specific gravity of 6 commercial liquid smokes (Table 1), pH was quite low range of 2.41~3.13. Brix of SS (Scansmoke PB 2110), ZS (Zestismoke) and SF (Imported product) showed 29.0~49.4 range, but SF3, SF10 and SF26 (Japan products) were relatively lower (3.1~9.0) than the others. The contents of total acidity were 9.5~11.4 g/100 g range for SS, ZS and SF and 3.0~5.2 g/100 g range for SF 3, SF10 and SF26, respectively. The total acidity content of each product was related with that of pH. Specific gravities of all samples were in 1.006~1.149 range.

Table 1. Results of pH, Brix, total acidity and specific gravity of commercial liquid smokes

Sample codes	pH	Brix	TA ¹⁾	S.G. ²⁾	Remark
SS	2.54 (2.1~2.9) ³⁾	49.4	9.5 ± 2.8	1.149	Scansmoke PB 2110, P. Broste A/S, Denmark
ZS	2.41 (2.0~2.5)	29.0	10.8 ± 0.4	1.070~1.088	Zestismoke, Hickory Specialties Inc., USA
SF	1.96	31.8	11.4 ± 0.1	1.082	Imported product, Backgeung Food Inc., South Korea
SF3	2.75 (2.5 ± 0.3)	4.0	3.8 ± 0.5	1.008 ± 0.005	SF-3, Donghae Hwasung Co. LTD., Japan
SF10	2.60 (2.7 ± 0.3)	9.0	5.2 ± 0.5	1.020 ± 0.010	SF-10, Donghae Hwasung Co. LTD., Japan
SF26	3.13 (2.9 ± 0.2)	3.1	3.0 ± 0.5	1.006 ± 0.005	SF-26, Donghae Hwasung Co. LTD., Japan

¹⁾TA: total acidity (g/100 g).

²⁾S.G.: specific gravity.

³⁾Data suggested from company.

Volatile flavor compounds in commercial liquid smokes

By comparing of two methods for detection of volatile flavor compounds in commercial liquid smokes, solvent extraction method was detected more amounts than those of Purge & Trap method. However, solvent extraction method was difficult to detect the compounds appearing before retention time (RT) 8~10 min in which solvent fraction and some volatile compounds appear at the same time in total ion chromatogram. Therefore, both of Purge & Trap and solvent extraction method were simultaneously conducted in this study to detect the kinds of compounds before RT 8~10 min.

A total of 156 volatile compounds were detected by GC/MSD with Purge & Trap and/or solvent extraction method in 6 commercial liquid smokes (Table 2). Among these, 121 compounds were detected by solvent extraction method and 40 compounds by P&T method.

In the volatile flavor compounds of liquid smokes detected, polycyclic aromatic hydrocarbons (PAHs), which were known as a carcinogen in human and detected in several commercial liquid smokes (Yabiku et al., 1993; Gomaa et al., 1993), were not detected in this study. It was suggested that commercial liquid smokes used in this study might safely be applied to any food processing without healthy problems with carcinogenic PAHs.

Among the groups of the identified compounds, 60 ketones had the most abundances compared to the others such as aldehydes (12), alcohols (7), acids (14), esters (20), aromatic compounds (24), furans (7) and miscellaneous compounds (12) in the whole liquid smokes. In the aspect of contents of compounds, ketones (806.6~7,573.9 $\mu\text{g}/\text{mL}$) and

aromatic compounds (282.6~7,896.3 $\mu\text{g/mL}$) were more amount than the others. As shown in the Table 2, the number of compounds detected in each commercial liquid smoke were different in this study. It was suggested that each liquid smoke might be produced from different sorts of wood ingredients and made with different concentration (Brix) as shown Table 1. In the group of aldehydes, furfural (147.9~1,168.5 $\mu\text{g/mL}$) and 4-hydroxy-3,5-dimethoxybenzaldehyde (116.4~421.4 $\mu\text{g/mL}$) have been relatively identified in high concentrations. In general, it was known that furfural is generated from the thermal degradation of cellulose and hemicellulose pyrolysis and has a similar odor with benzaldehyde (Martha et al., 1976). And 4-hydroxy-3-methoxybenzaldehyde (vanillin) and 4-hydroxy-3,5-dimethoxybenzaldehyde having pleasant aromatic vanilla taste and aroma are derived from thermal pyrolysis of lignin. However, these compounds were not detected in some samples including SF3 and SF26. It has been used in the field of beverage, food and perfume as a flavoring agent (Martha et al., 1976). Lignin as one of the major constituents of wood is a high molecular mass and randomly cross-linked polymer, being consisted of an irregular array with differently bonded hydroxy- and methoxy- substituted phenylpropanoid side chains (Guillen and Ibargoitia, 1998; Alen et al., 1996). During pyrolysis, thermal degradation reactions take place to generate different bond cleavages according to their bond energies (Guillen and Ibargoitia, 1998). In general, phenolic compounds as most important flavor compounds arise from oxidative lignin decomposition (Wittkowski et al., 1992). Tressl et al. (1976) found that heating ferulic acid, which arises from oxidation of coniferyl alcohol, at 240~260°C was sufficient to cause a stepwise propanoid side chain degradation leading to the final guaiacol and its derivatives, vanillin, acetovanillone, eugenol and isoeugenol. Therefore, these substances must be regarded as final products formed by lignin pyrolysis (Wittkowski et al., 1992).

Sixty compounds of ketones identified presented approximately 16.5~37.4% of the total concentrations of all volatile compounds. Several compounds such as acetone (96.5~721.2 $\mu\text{g/mL}$), 2,3-butanedione (62.3~593.4 $\mu\text{g/mL}$), hydroxyacetone (24.0~680.7 $\mu\text{g/mL}$), 2-hydroxy-3-methyl-2-cyclopenten-1-one

(43.6~1,103.8 $\mu\text{g/mL}$), γ -crotonlactone (38.6~492.7 $\mu\text{g/mL}$) and 3-hydroxy-2-methyl-4H-pyran-4-one (maltol) (116.2~681.6 $\mu\text{g/mL}$) in the ketones were in higher concentrations. γ -Crotonlactone is originated from pyrolysis of cellulose and hemi-cellulose (Guillen and Manzanos, 1996a; Guillen and Manzanos, 1996b), and the presence of 2-hydroxy-3-methyl-2-cyclopenten-1-one in these liquid smoke might be interested. This compound was present in subfractions of the weakly acidic fraction of cigarette smoke condensate which showed cocarcinogenic and tumor-promoting activity on mouse skin (Hecht et al., 1981). However, subsequent research about this compound demonstrated that it was not active as a cocarcinogen or tumor promoter (Stephen et al., 1981). Therefore, taking account of the healthy aspects, it was considered that further studies will be necessary in this kind of compound. Maltol, found in bark of young larch trees, pine needles, and wood tars, has caramel-like odor and used as a flavor agent applying to bread and cake processing (Martha et al., 1976).

Pyrocatechol (145.1~1,266.7 $\mu\text{g/mL}$) was appeared in most high concentration in the group of alcohols (57.3~2,113.3 $\mu\text{g/mL}$) and known to be formed by lignin pyrolysis (Wittkowski et al., 1992).

In relation to the group of acids, which is well-known one of important substances of liquid smoke and gives a role as antibacterial substances (Sofos et al., 1988), this group (422.9~4,903.2 $\mu\text{g/mL}$) was identified in relatively high concentration compared to the total content of all volatile compounds identified. It was expected that shelf-life of any products should be extended when the liquid smoke applied to them. On the other hand, among 6 products, total acids of SF (4,903.1 $\mu\text{g/mL}$) and SS (2,240.4 $\mu\text{g/mL}$) were in relatively higher amounts than those of the others. Acetic acid (367.2~2,647.5 $\mu\text{g/mL}$) presented 54~87% of the total acids in samples. Ferulic acid, which existed in very low concentration, is known a precursor of phenolic compounds (Wittkowski et al., 1992).

Amounts of ester compounds were 462.2~3,231.4 $\mu\text{g/mL}$. However, aromatic compounds was in one of most high concentration, and especially most of compounds were phenol and its derivatives in this group.

Phenol and its derivatives were basically origi-

Table 2. Volatile flavor compounds in commercial liquid smokes by Purge & Trap and solvent extraction methods¹⁾

Compound named by class	RI ²⁾	Sample codes ³⁾					
		ZS	SS	SF	SF3	SF10	SF26
Aldehydes (12)		2,046.3	1,673.8	1,597.7	1,062.2	1,100.9	794.9
*Acetaldehyde	<600	2.5	5.4	7.7	2.8	7.7	4.0
3-Furancarboxaldehyde	819	23.8	— ⁴⁾	—	—	—	—
Furfural	829	1,168.5	195.9	854.7	907.0	147.9	721.7
5-Methylfurfural	968	37.8	—	—	152.4	756.2	69.2
2,3-Dihydroxybenzaldehyde	1173	56.2	107.0	40.8	—	—	—
5-Hydroxymethylfurfural	1244	—	331.9	—	—	—	—
3-Ethoxy-4-hydroxybenzaldehyde	1375	88.9	266.9	—	—	—	—
4-Hydroxy-3-methoxybenzaldehyde	1412	124.7	181.9	194.9	—	72.7	—
2,4-Dimethoxybenzaldehyde	1445	30.7	87.9	5.4	—	—	—
2-Ethoxy-4-methoxybenzaldehyde	1596	48.8	91.7	36.4	—	—	—
2,5-Dimethoxy-4-ethylbenzaldehyde	1662	93.2	122.8	36.4	—	—	—
4-Hydroxy-3,5-dimethoxybenzaldehyde	1675	371.2	282.4	421.4	—	116.4	—
Ketones (60)		7,491.5	7,573.9	5,851.5	806.6	1,255.6	1,110.8
*Acetone	<600	335.8	—	721.2	103.4	96.5	331.3
*2,3-Butanedione	600	313.2	111.7	593.4	62.3	62.5	178.7
*2-Butanone	604	178.4	19.0	405.8	15.3	18.3	133.0
*3-Methyl-2-butanone	661	26.4	—	82.9	4.9	7.1	24.9
*Hydroxyacetone	684	317.2	680.7	414.0	54.8	24.0	61.0
*2-Pentanone	693	62.5	—	95.4	2.2	—	23.5
*2,3-Pentanedione	701	106.5	—	123.1	64.2	11.9	45.5
*3-Pentanone	703	20.0	—	30.9	—	—	—
*3-Hydroxy-2-butanone	722	17.5	21.2	78.1	9.1	7.8	14.6
*4-Methyl-2-pentanone	740	—	3.1	—	1.0	2.8	2.0
*3-Penten-2-one	742	17.9	6.9	45.3	7.3	6.2	12.0
*2-Methyl-3-pentanone	753	2.0	—	5.3	—	—	—
*2,3-Hexanedione	787	22.3	—	16.4	—	—	—
*2-Hexanone	793	40.6	—	20.0	—	—	—
**1-Hydroxy-2-butanone	778	191.1	52.6	139.4	153.8	102.6	91.1
**Cyclopentanone	797	94.4	93.1	93.1	14.5	3.2	12.1
1-Hydroxy-3-methyl-2-butanone	826	26.1	—	24.4	—	—	—
1-Hydroxy-2-pentanone	860	—	—	—	9.1	3.9	—
3-Methyl-2-hexanone	863	237.1	156.1	188.2	49.0	33.2	23.8
Acetoxyacetone	874	69.6	39.3	52.0	—	—	—
Dihydro-2H-pyran-3(4H)-one	887	19.4	9.1	8.6	7.7	4.7	—
Cyclohexanone	898	13.2	—	—	—	—	—
2-Methyl-2-cyclopenten-1-one	911	96.9	27.9	60.8	57.3	14.5	33.3
Butyrolactone	918	—	—	—	—	48.6	13.3
γ -Crotonlactone	921	492.7	385.2	400.5	—	38.6	—
2-Hydroxy-2-cyclopenten-1-one	931	230.4	154.0	48.0	—	—	—
2,5-Hexanedione	933	169.8	198.5	62.7	—	8.8	—
β -Angelicalactone	944	60.4	93.2	45.3	—	—	—
2,5-Dimethyl-3(2H)-furanone	952	34.1	68.1	26.0	—	2.0	—
3-Methyl-2-cyclopenten-1-one	969	213.7	139.1	134.6	—	—	—
2H-Pyran-2-one	976	—	8.6	—	—	—	—
3-Methyl-2(5H)-furanone	984	62.4	70.1	52.6	—	—	—
3-Methylcyclohexanone	1005	45.4	98.9	32.2	14.6	22.6	6.2
2-Hydroxy-3-methyl-2-cyclopenten-1-one	1035	821.4	1,103.8	623.0	92.2	222.0	43.6
2,3-Dimethyl-2-cyclopenten-1-one	1043	79.8	64.8	74.8	42.4	39.0	26.5
4-Methyl-2(5H)-furanone	1048	—	142.5	—	—	—	—
2-Methyl-1-penten-1-one	1051	118.3	—	—	—	—	—
3,5-Dimethyl-cyclopentan-1,2-dione	1061	82.9	245.8	82.3	13.6	38.1	—
3-Ethyl-2-cyclopenten-1-one	1079	—	316.0	—	—	—	—
2-Acetylcyclopentanone	1080	315.3	—	—	—	—	—
3,4-Dimethylcyclopentanone	1084	48.9	—	—	—	—	—
4-Hydroxy-2,5-dimethyl-3(2H)-furanone	1090	138.5	326.7	104.5	—	—	—
Trimethyl-2-cyclopenten-1-one	1105	—	—	—	11.2	—	25.6
4,4-Dimethyl-2-cyclohexen-1-one	1107	59.2	—	37.9	—	—	—

Table 2. (continued)

Compound named by class	RI ²⁾	Sample codes ³⁾					
		ZS	SS	SF	SF3	SF10	SF26
3-Hydroxy-2-methyl-4H-pyran-4-one	1120	185.4	681.6	184.0	—	116.2	—
3-Ethyl-2-hydroxy-2-cyclopenten-1-one	1125	217.5	—	164.2	8.9	98.2	—
γ -Heptanolactone	1134	22.6	—	—	—	—	—
5-Acetyldihydro-2(3H)-furanone	1135	—	85.7	16.2	—	—	—
5-Hydroxy-2-methyl-4H-pyran-4-one	1144	—	24.1	—	—	—	—
Tetramethylcyclopentan-1-one	1149	32.3	—	—	—	—	—
Camphor	1151	—	—	—	7.8	—	8.8
3-Ethyl-5-hydroxy-2-methyl-2-cyclopenten-1-one	1152	20.7	—	—	—	—	—
4-Hydroxy-3-methoxyacetophenone	1499	237.2	204.6	107.6	—	42.9	—
4-Hydroxy-3-methoxyacetone	1543	255.1	190.5	34.6	—	—	—
Zingerone	1637	—	44.2	—	—	—	—
4-Hydroxy-3,5-dimethoxyacetophenone	1745	475.7	487.5	331.5	—	128.4	—
1-(2,4,6-Trihydroxy-3-methylphenyl)-1-butanone	1773	—	81.3	—	—	—	—
1-(2,4,6-Trihydroxyphenyl)-2-pentanone	1785	825.3	1,137.6	75.8	—	—	—
Aspidinol	1795	38.4	93.9	14.9	—	3.5	—
1,2-Diphenylcyclobutendione	1964	—	—	—	—	47.5	—
Alcohols (7)		349.5	2,113.2	891.9	64.6	268.4	57.3
*Ethanol	<600	0.3	1.9	6.4	—	—	—
*2-Propanol	<600	71.1	—	245.4	18.6	35.6	29.6
*Propanol	<600	—	—	65.4	10.3	13.8	13.0
Tetrahydro-2-furanmethanol	836	102.9	79.6	85.5	35.7	38.1	14.7
Pyrocatechol	1225	145.1	1,266.7	469.7	—	180.9	—
4,5-Dimethyl-1,3-benzenediol	1298	30.1	103.3	19.5	—	—	—
4-Methyl-1,2-benzenediol	1315	—	661.7	—	—	—	—
Acids (14)		807.3	2,240.4	4,903.1	717.0	458.6	422.9
*Acetic acid	652	464.7	1,418.9	2,647.5	628.8	367.2	380.7
*Propionic acid	712	—	49.4	—	32.1	31.5	21.7
**2-Methyl propionic acid	762	209.5	129.4	175.9	—	—	—
Butyric acid	806	68.9	—	119.3	56.1	33.1	20.5
2-Furancarboxylic acid	1091	—	—	—	—	26.8	—
Capric acid	1370	—	—	137.4	—	—	—
Undecylic acid	1566	—	—	152.6	—	—	—
Ferulic acid	1682	44.4	77.0	—	—	—	—
Isoferulic acid	1712	19.8	210.4	3.9	—	—	—
Lauric acid	1762	—	87.9	—	—	—	—
Myristic acid	1962	—	126.0	108.4	—	—	—
Oleic acid	2143	—	141.4	1,427.8	—	—	—
Palmitic acid	2165	—	—	75.5	—	—	—
Linoleic acid	2186	—	—	54.8	—	—	—
Esters (20)		1,127.0	622.7	3,231.4	462.2	2,606.6	631.9
*Methyl acetate	<600	777.6	286.4	2,329.6	401.9	518.8	552.7
*Ethyl acetate	617	11.6	—	—	—	—	—
*Methyl 2-propenoate	618	9.3	—	—	—	—	—
*Methyl propionate	631	124.1	—	387.4	44.5	39.0	60.2
*Propyl acetate	717	9.5	—	33.8	—	—	—
*Methyl 3-butenate	719	21.1	—	39.9	—	—	—
*Methyl butyrate	725	56.5	—	131.6	15.8	16.7	19.0
*Methyl 2-methyl propionate	733	3.5	—	6.8	—	—	—
*Methyl (Z)-2-butenate	737	4.0	—	10.4	—	—	—
*Methyl (E)-2-butenate	766	16.6	—	30.5	—	—	—
Methyl 2-furancarboxylate	980	8.9	—	—	—	—	—
Ethyl crotonate	1050	—	—	98.5	—	—	—
Methyl undecyrate	1401	—	—	40.7	—	—	—
Methyl 3-hydroxy benzoate	1440	—	95.6	—	—	—	—
Methyl 4-hydroxy-3-methoxy benzoate	1528	32.8	185.3	38.0	—	—	—
Dimethyl 3-methyl-4-propyl-2,4-hexadienedioate	1833	51.5	—	20.5	—	—	—
Methyl palmitate	1925	—	—	43.6	—	—	—

Table 2. (continued)

Compound named by class	RI ²⁾	Sample codes ³⁾					
		ZS	SS	SF	SF3	SF10	SF26
Ethyl palmitate	1993	—	24.2	—	—	—	—
Methyl stearate	2127	—	—	20.1	—	—	—
Ethyl linolate	2162	—	31.2	—	—	—	—
Aromatic compounds (24)		4,628.6	7,896.3	2,117.1	634.9	1,093.7	282.6
*Benzene	665	19.9	5.7	24.5	2.0	11.2	10.6
**Toluene	772	13.7	10.4	12.5	59.4	49.2	54.5
Phenol	997	453.9	153.9	77.8	76.7	30.2	32.7
o-Cresol	1067	250.4	85.8	141.4	15.6	10.8	—
p-Cresol	1088	81.8	473.2	98.9	—	—	—
Guaiacol	1094	573.7	349.7	276.0	341.1	78.2	154.6
2,5-Dimethylphenol	1114	36.1	—	—	—	—	—
2,4-Dimethylphenol	1157	76.0	49.2	66.3	—	—	—
4-(Methylthio)phenol	1167	—	—	—	—	24.6	—
Trimethylphenol	1168	—	—	11.0	—	—	—
2-Ethyl-5-methylphenol	1247	83.0	—	36.4	—	—	—
2,6-Bis(1,1-dimethylethyl)naphthalene	1269	68.5	226.0	29.3	7.1	70.9	—
4-Ethylguaiacol	1285	322.9	630.0	123.9	47.2	77.5	—
Syringol	1360	1,626.1	3,605.8	874.2	75.3	585.8	30.2
Eugenol	1365	166.3	176.4	41.5	—	—	—
1,2,3-Trimethoxybenzene	1385	—	100.7	—	—	—	—
1,2,3-Trimethoxy-5-methylbenzene	1408	28.5	234.7	—	—	—	—
Isoeugenol	1416	56.8	119.5	—	—	—	—
4-Propylguaiacol	1471	55.3	81.2	12.1	—	—	—
1,1'-Methylenebisbenzene	1517	—	—	—	—	5.6	—
1,2,5-Trimethoxy-3-methylbenzene	1535	435.6	1,142.0	180.7	10.5	97.3	—
Methoxyeugenol	1612	225.4	342.3	88.2	—	52.4	—
4-(Ethoxymethyl)-2-methoxyphenol	1664	—	109.8	—	—	—	—
1-Methoxy-2-(2-phenylethenyl)benzene	1758	54.7	—	22.4	—	—	—
Furans (7)		3,194.5	69.1	2,418.2	48.0	740.0	7.7
*2,3-Dihydro-5-methylfuran	670	—	1.4	—	—	—	—
**3-Hydroxy tetrahydrofuran	782	73.4	—	107.9	13.3	15.2	—
2,5-Dimethoxytetrahydrofuran	900	27.7	—	7.0	—	—	—
2-Acetylfuran	916	53.4	—	35.7	34.7	4.6	7.7
2-Hydroxy-2-methyl-tetrahydrofuran	1010	3,040.0	—	—	—	—	—
Tetrahydro-2-(methoxymethyl) furan	1083	—	—	—	—	720.2	—
2,3-Dihydro-2-methyl-4(or 6)-phenylbenzofuran	1689	—	67.7	2,267.6	—	—	—
Miscellaneous compounds (12)		398.2	1,812.2	176.8	3.3	98.6	—
*2-Methylbutane	<600	0.7	0.9	—	—	—	—
*Pentane	<600	—	128.2	—	—	—	—
*Dimethoxymethane	<600	87.0	—	104.3	—	—	—
1,2-Dimethoxycyclopropane	861	38.9	23.4	27.2	—	—	—
2,5-Dimethyl-2,4-hexadiene	1025	19.1	33.6	6.3	—	—	—
2-Propyl-1,3-dioxolane	1048	14.4	—	—	—	—	—
2,3-Dihydroxyanisole	1273	125.4	1,109.2	39.0	—	95.4	—
1,1-Diphenylpropane	1618	—	458.4	—	—	—	—
2-Bromooctane	1699	19.5	—	—	—	—	—
4-Ethyl-diphenylmetane	1716	80.5	—	—	—	—	—
Cyclopentadecane	1943	—	58.5	0.0	—	—	—
Docosane	2196	12.7	—	—	3.3	3.2	—

¹⁾ Mean concentration ($\mu\text{g/mL}$) of samples, and concentration of each compound was calculated as a relative content to n-tridecene concentration ($75.83 \mu\text{g/mL}$) put in sample (factor=1).

²⁾ Retention Index on Ultra-2TM column (50 m length \times 0.2 mm i.d. \times 0.33 μm film thickness, Hewlett-Packard Co., USA).

³⁾ Refer to comment in Table 1.

⁴⁾ Not Detected.

*Detected by Purge & Trap method only.

**Detected in both Purge and Trap and solvent extraction methods. In this case, the data were expressed with mean values detected by solvent extraction.

nated from the thermal pyrolysis of lignin. Due to a high molecular polymer and the high structural diversity of lignin, its pyrolysis should provide a high number of compounds. Most of the derivatives of phenol, guaiacol and syringol have been considered as key compounds responsible for the antioxidant properties of liquid smoke (Fretheim et al., 1980) and would be expected to extend the storage time of products. In relation to sensory properties, the flavor of these liquid smokes might be basically affected by phenol derivatives, and phenol flavor such as pungent, cresolic, sweet, smoky and burnt odors (Kim et al., 1974; Toth and Potthast, 1984; Baltés and Söchtig, 1979) are present. Additionally, compounds having odors described as sweet (2-furancarboxaldehyde and 5-methyl-2-furancarboxaldehyde) or grassy, sweet-spicy (3-methyl-2-cyclopenten-1-one) (Kim et al., 1974) are also present and are expected to contribute to the overall aroma of these liquid smokes.

In the group of aromatic compound, guaiacol (78.2~573.7 $\mu\text{g}/\text{mL}$) and syringol (30.2~3,605.8 $\mu\text{g}/\text{mL}$) were in very high concentrations even though phenol, *o*-cresol, guaiacol and 4-ethylguaiacol were in relatively low concentrations, but higher than the others. Also, among 6 products, SS (7,896.3 $\mu\text{g}/\text{mL}$) and ZS (4,628.6 $\mu\text{g}/\text{mL}$) have relative higher amounts of aromatic compounds than the other. The compounds, phenol and *o*-cresol, were used as a disinfectant and an antibacterial agent in several industrial fields (Martha et al., 1976).

Amounts of furans, derived from distillation of pine wood (Martha et al., 1976), were 7.7~3,194.5 $\mu\text{g}/\text{mL}$ in the liquid smokes. Fiddler et al. (1967) reported that furans and pyran derivatives could be generated from cellulose and hemicellulose pyrolysis. In miscellaneous compounds (3.3~1,812.2 $\mu\text{g}/\text{mL}$), 10 hydrocarbons were detected. These types of compounds are originated from the wood pyrolysis and present in smoke (Guillen and Manzanos, 1996a; Guillen and Manzanos, 1996b).

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