Volatile Flavor Compounds in Spray-Dried Skim Milk Powder

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The volatile flavor compounds of skim milk powder have been investigated. Commercially processed spray-dried skim milk was homogenized with water, and the volatiles were isolated by simultaneous steam distillation-extraction under reduced pressure (SDE) using diethyl ether as solvent. The odor concentrate was analyzed by gas chromatography and gas chromatography-mass spectrometry. Among 196 individual peaks detected, 187 peaks were definitely or tentatively identified by mass spectrum and modified Kovats indices. Major compounds were 48 hydrocarbons, 18 aldehydes, 20 ketones, 21 alcohols, 29 fatty acids, 8 esters, 2 furans, 7 phenolic compounds, 10 lactones, and 14 nitrogenous compounds, which constituted over 99% of total volatiles recovered. Most of them originated by breakdown of the major constituent of milk, especially fat to smaller, volatile chemicals or their secondary reaction, or by transfer from the forage.

INTRODUCTION

Skim milk powder is one of the most widely used raw materials for various food products, due to its low water content (at most 5%), convenience in transport for its light weight, and good preservation. Dairy products not only play an important role in a well-balanced diet but provide a great sense of eating pleasure for their characteristic flavor and smooth taste. However, certain basic flavors in milk and off-flavors that may occur could be carried over to the final products. Since milk possesses a bland and soft flavor, such occurrence of off-flavor is readily noticeable and objectionable.

To date, no detailed information is yet available on the flavor of skim milk powder, although extensive work has been carried out on the effects of heat treatment and storage on the aroma of fluid milk and milk fat (Scanlan et al., 1968; Forss, 1971, 1979; Jeon et al., 1978; Jaddou, 1978; Badings and Neeter, 1980; Rerkrai et al., 1987; Badings, 1991; Gaafar, 1991). We have carried out, therefore, a study on the isolation and identification of volatiles in skim milk powder to establish a suitable quality evaluation method for skim milk.

MATERIALS AND METHODS

Materials. The commercially processed spray-dried skim milk was obtained from Meiji Milk Products Co., Ltd. (Tokyo), with the declared ingredients as shown in Table 1. Diethyl ether and anhydrous sodium sulfate were from Nakarai Tesque, Inc. (Kyoto, Japan), and 2-ethylhexyl acetate was from Tokyo Kasei Kogyo Co. Ltd. (Tokyo).

Isolation of Volatile Flavor Compounds. The skim milk powder (300 g) was homogenized in 900 mL of deionized water using a high-speed blender and the homogenate was placed in a 2000-mL round-bottom flask. Volatiles were separated with 80 mL of diethyl ether from the homogenate by simultaneous distillation-extraction under reduced pressure (approximately 80 mmHg) for 1 h, using a modified Likens-Nickerson apparatus (SDE method). After addition of 30 μL of 0.1% 2-ethylhexyl acetate, the extract was dried over anhydrous sodium sulfate for 3 h and concentrated to about 200 μL.

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Table 1. Formulation of the Skim Milk Powder

ingredient	composition, %	ingredient	composition, %
carbohydrate	52.2	water	4.2
protein	34.8	ash	5.1
fat	1.0	other	2.7

Capillary Gas Chromatography (GC). Capillary GC analysis was carried out on a Hewlett-Packard Model 5890A gas chromatograph equipped with a flame ionization detector (FID) and connected to a Shimadzu Chromatopak C-R5A integrator. Separation was achieved on a 60 m × 0.25 mm i.d. fused silica capillary column, coated with cross-linked polyethylene glycol 20M, film thickness 0.25 μm (DB-Wax; J&W Scientific, Folsom, CA). The oven temperature was programmed from 50 to 230 °C at 2 °C/min (60-min hold). The injector and detector temperatures were 200 and 250 °C, respectively. The helium carrier gas flow rate was 22 cm/s with an injection splitter at a split ratio of 30:1. Retention indices were estimated in accordance with a modified Kovats method (Van den Dool and Kratz, 1963).

Capillary Gas Chromatography-Mass Spectrometry (GC-MS). Electron impact mass spectrometric data were collected on a JEOL Automass 50 mass spectrometer interfaced to a Hewlett-Packard 5890 Series II gas chromatograph. The column and chromatographic conditions were the same as described for GC analysis. The mass spectrometer was operated at an ionization voltage of 70 eV and an ion source temperature of 200 °C. The mass spectra of the unknown compounds were compared with those in the *Wiley/NBS Registry of Mass Spectral Data* by using a computer system and other published spectra (*Eight Peak Index of Mass Spectra*, 1983; *Wiley/NBS Registry of Mass Spectral Data*, 1989).

RESULTS AND DISCUSSION

The yield of total volatiles was 0.0012% (w/w, relative to the skim milk powder used). Figure 1 shows a typical gas chromatogram of the volatile components from the skim milk. Of a total of 196 individual peaks detected, 187 peaks were definitely or tentatively identified (Table 2). The area of these 187 peaks represented about 99% of the chromatogram surface (excluding solvent and 2-ethylhexyl acetate). The compounds identified included 48 hydrocarbons, 18 aldehydes, 20 ketones, 21 alcohols, 29 fatty acids, 8 esters, 2 furans, 7 phenolic compounds, 10 lactones, 14 nitrogenous compounds, and 10 miscellaneous compounds.

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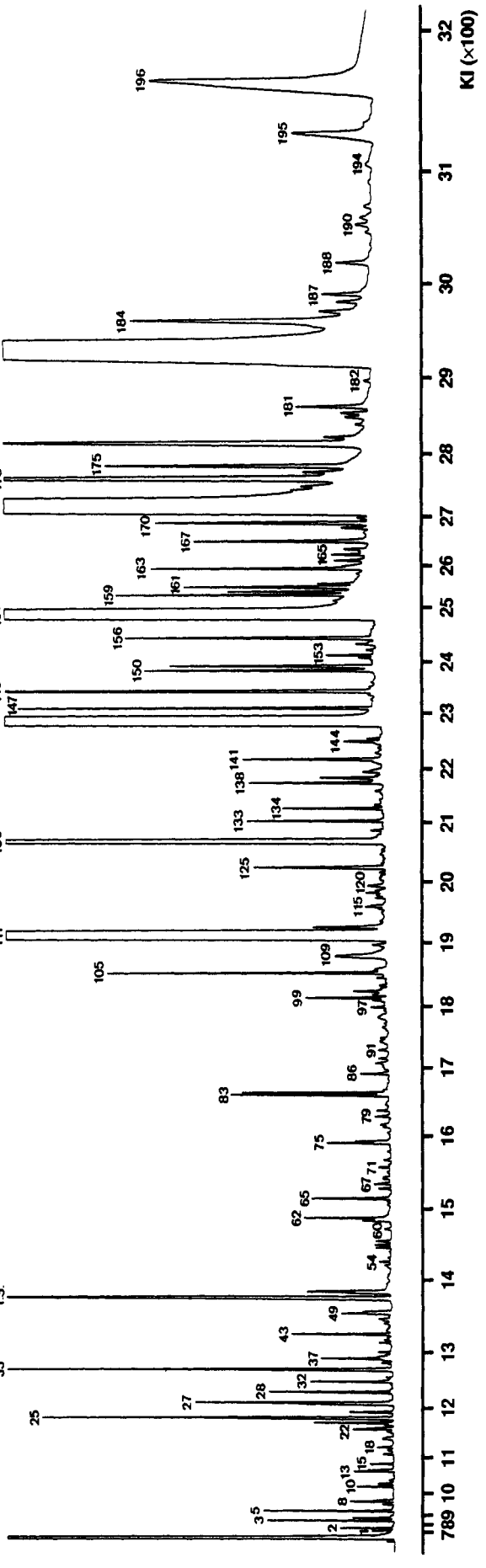

Figure 1. Gas chromatogram of flavor concentrate from skim milk powder. I.S. (internal standard) corresponds to 2-ethylhexyl acetate. For more information, see Table 2 and Materials and Methods.

Among the hydrocarbons, alkanes and alkenes (C_9–C_{19}) were identified. They may come from the autoxidation of free fatty acids naturally present in animal tissues and milk or perhaps from a nonsaponifiable fraction of plants used in cow feed. Most of the mono- and sesquiterpene hydrocarbons were identified for the first time in milk. Since terpene biosynthesis is carried out only by plants and some microorganisms, they must be transferred from feed to the milk via the rumen. The aliphatic hydrocarbons identified have a weak odor, and since they were present in concentrations much below their odor thresholds, they may not contribute to milk flavor. Finally, it is interesting to note that many aromatic compounds were detected. While their odor thresholds are not so high, aromatic hydrocarbons have high volatility and characteristic odor, and so they could be of concern with the skim milk flavor.

The alkanals and alkenals with more than six carbon atoms are typical products of lipid oxidation. Indeed, the autoxidative mechanisms of the unsaturated fatty acids lead to the complete inferior homologous series of aldehydes (Loury, 1971; Paquette et al., 1985). They have very low flavor thresholds (Kinsella et al., 1967) and are characterized by an oily, fatty, or tallowy odor, so that it is said that these compounds, when their concentrations pass a certain level, are the most significant contributors to the oxidation flavor defects of milk (Azzara et al., 1992). However, in the present study they were in extremely small amounts, near or below the threshold level, and the aliphatic aldehydes (C_6–), when present in the concentrations found in skim milk, could have been responsible for desirable flavors of skim milk. On the other hand, no aliphatic aldehydes with fewer than five carbon atoms, mainly derived from Strecker degradation of amino acids, were found, whereas these aldehydes have been reported to exist in heat-treated milk (Baddings, 1991).

Among the ketones identified, methyl ketones were the most abundant. They were proposed to form from free fatty acids which were first oxidized to β-keto acids and then decarboxylated into the corresponding methyl ketones (Hawke, 1966; Kinsella et al., 1967; Forss, 1979). Methyl ketones, particularly 2-heptanone and 2-nonanone, play an important role in the flavor of blue or Camembert cheeses, as typical metabolites of *Penicillium* molds (Dartey and Kinsella, 1971; Dwivedi and Kinsella, 1974; Okumura, 1985; Matsuura, 1988). Langler and Day (1965) found that flavor potency varied with carbon-chain length and 2-heptanone had the lowest flavor threshold value (700 ppb). The level of methyl ketones in the skim milk powder was extremely low, far below the flavor thresholds in milk; therefore, they play only a secondary role in skim milk flavor.

Alcohols were represented by primary alcohols, probably produced by reduction of the corresponding aldehydes. Because of their high flavor thresholds in milk (0.4–2.0 ppm; Jeon et al., 1978), it is unlikely that primary alcohols contributed to the flavor of skim milk. Among other alcohols, 1-octen-3-ol, the character impact flavor compound of fresh mushrooms, was identified. It was proposed to be the autoxidation product of linoleic acid (Stark and Forss, 1964). Even though it has a very low odor threshold in skim milk (10 ppb), 1-octen-3-ol probably did not influence the flavor of the milk because of its very low amount (Table 1). Finally, three terpene alcohol, linalool, nerolidol, and δ-cadinol, were identified for the first time in milk. They may have been derived from feed, as well as other terpenes.

The chemical family with the highest proportion was free fatty acids, comprising about 79% of the total volatile

Table 2. Volatile Flavor Compounds Identified in Skim Milk Powder

peak[a]	compound	Kovats index[b]	content, ppb	peak[a]	compound	Kovats index[b]	content, ppb
1	carbon disulfide	791	1.05	76	hexadecane	1599	4.34
2	ethyl formate	825	3.40	77	2-formyl-1-methylpyrrole	1620	1.43
3	ethyl acetate	888	9.37	78	*N*-methylacetamide	1623	2.34
4	nonane	906	3.19	79	ethylene glycol	1635	2.70
5	ethanol	947	10.90	80	(*Z*)-2-decenal	1644	2.06
6	unknown	977	0.95	81	β-caryophyllene	1653	1.22
7	3-methylnonane	983	0.73	82	acetophenone	1655	1.29
8	octamethylcyclotetrasiloxane[c]	988	3.39	83	2-furanmethanol	1667	22.47
9	2-pentanone and diacetyl	993	1.87	84	cyclohexyl isothiocyanate	1670	16.70
10	2-methyl-3-buten-2-ol	1040	2.92	85	salicylaldehyde	1674	1.55
11	toluene	1049	2.25	86	heptadecane	1697	5.01
12	2,5-dimethylnonane	1059	0.76	87	2-dodecanone	1704	1.38
13	unknown	1082	3.15	88	dodecanal	1708	2.14
14	hexanal	1085	2.32	89	unknown	1714	4.44
15	undecane	1099	1.93	90	*N*-butyl-*N*-nitrosobutanamine[c]	1722	2.13
16	1-methyl-4-(1-methylethyl)cyclohexane	1122	0.98	91	naphthalene	1740	2.90
17	(*Z*)-3-penten-2-one	1133	1.15	92	α-cubebene and 1-decanol	1748	4.51
18	ethylbenzene	1138	1.85	93	3-methylheptadecane	1754	1.80
19	*p*-xylene	1156	2.25	94	*N*,*N*-dibutylformamide	1767	1.23
20	*m*-xylene	1158	0.88	95	unknown	1771	2.62
21	*o*-xylene	1169	1.28	96	diethylene glycol butyl ester and 4′-hydroxyacetophenone	1786	7.62
22	unknown	1173	7.34				
23	(1-methylethyl)benzene	1181	1.39	97	octadecane	1801	1.41
24	2-heptanone	1185	8.77	98	*N*,*N*-dibutylacetamide	1813	3.74
25	*d*-limonene	1193	52.37	99	2-tridecanone	1817	11.56
26	propylbenzene	1203	5.98	100	tridecanal	1821	4.73
27	*m*-ethyltoluene	1221	37.11	101	naphthodioxane	1827	6.56
28	1,3,5-trimethylbenzene	1241	15.02	102	silanoid compound[c]	1836	2.08
29	γ-terpinene	1244	0.64	103	calamenene	1841	1.71
30	1-pentanol	1252	0.46	104	1-octadecene	1848	2.70
31	2,10-dimethylundecane	1254	0.49	105	hexanoic acid	1855	35.63
32	*o*-ethyltoluene	1258	10.63	106	geranylacetone	1858	1.90
33	2,9-dimethylundecane	1262	0.80	107	2′,3′,4′-trimethylacetophenone	1861	3.74
34	*p*-cymene	1265	0.97	108	?-tridecenal[c]	1881	0.85
35	1,2,4-trimethylbenzene	1278	56.96	109	?-tridecenal[c]	1886	27.47
36	octanal	1287	1.47	110	unknown	1897	4.88
37	tridecane	1293	13.38	111	2,6-di-*tert*-butyl-*p*-cresol (BHT)	1921	1273.85
38	*p*-propyltoluene	1297	1.33	112	tetradecanal	1927	18.55
39	1,3-diethylbenzene	1302	1.98	113	β-ionone and 1-nonadecene	1938	3.03
40	7-methyl-3-octen-2-one	1317	1.94	114	2-ethylhexanoic acid and heptanoic acid	1950	3.79
41	2-methyl-3-octanone	1322	1.52	115	benzothiazole	1962	8.79
42	2,2,4-trimethylpentanol	1326	1.26	116	1-dodecanol	1977	2.69
43	1,2,3-trimethylbenzene	1331	12.40	117	unknown	1982	2.76
44	unknown	1336	1.20	118	2-tridecanol	1986	3.52
45	2,3-dimethyl-2,3-butanediol	1338	1.19	119	3,7-dimethyl-1,7-octanediol	1988	2.69
46	5-methyltridecane	1342	0.77	120	?-tetradecenal[c]	1997	6.65
47	1,1,3,3-tetraethoxypropane[c]	1347	2.22	121	phenol	2004	1.63
48	4-ethyl-1,2-dimethylbenzene	1354	3.95	122	methyl myristylate	2008	1.63
49	1-ethyl-2,3-dimethylbenzene	1362	6.93	123	methyl *N*,*N*-dimethyldithiocarbamate[c]	2012	1.63
50	2,3-dihydro-1*H*-indene	1365	5.38	124	unknown	2020	1.67
51	2-nonanone, nonanal, and tetradecane	1391	27.85	125	2-pentadecanone	2028	24.26
52	2-butoxyethanol and (*E*)-3-octen-2-one	1411	1.15	126	pentadecanal	2042	1.48
53	1,2,3,4-tetramethylbenzene[c] and 3-methyltridecane	1430	1.01	127	unknown	2049	1.30
				128	nerolidol	2050	2.74
54	(*E*)-2-octenal	1435	1.85	129	9-acetoanthrasene	2059	1.42
55	4-ethyl-*m*-xylene	1441	1.03	130	octanoic acid	2075	405.53
56	1,4-dichlorobenzene	1450	0.78	131	*p*-cresol	2091	4.18
57	unknown	1455	1.86	132	*m*-cresol	2099	2.53
58	acetic acid and 1-octen-3-ol	1459	2.50	133	unknown	2108	18.19
59	furfural	1465	2.62	134	2-*tert*-butylindole	2131	13.75
60	6-methylheptyl 2-propionate	1480	1.73	135	hexadecanal	2137	3.02
61	2-ethylhexanol and α-copaene	1491	5.38	136	2-oxo-1-methyl-3-isopropylpyrazine[c]	2160	2.68
62	pentadecane	1494	14.22	137	2-undecanone *O*-methyloxime[c]	2163	4.42
63	1*H*-pyrrole	1512	0.65	138	nonanoic acid	2177	19.80
64	(*E*,*E*)-3,5-octadien-2-one	1517	0.56	139	carboxylic acid[c]	2187	14.36
65	benzaldehyde	1522	10.68	140	δ-cadinol	2212	8.50
66	(*E*)-2-nonenal	1536	1.71	141	δ-decalactone	2221	27.67
67	unknown	1542	3.02	142	methyl palmitate	2233	3.91
68	1-pentadecene	1550	1.28	143	cadalene	2242	2.00
69	linalool	1552	1.10	144	2-heptadecanone	2255	10.48
70	6-propyltridecane	1559	1.13	145	6-*tert*-butyl-*m*-cresol	2260	5.10
71	1-octanol	1565	2.01	146	decanoic acid	2300	1586.57
72	(*E*,*Z*)-3,5-octadien-2-one	1578	1.38	147	2,4-di-*tert*-butylphenol	2321	56.95
73	limonene oxide	1586	1.00	148	δ-undecalactone	2329	0.67
74	(*E*,*Z*)-2,6-nonadienal	1590	0.52	149	10-undecenoic acid	2351	118.34
75	2-undecanone	1596	8.14	150	undecanoic acid and 1-hexadecanol	2400	43.37

Table 2 (Continued)

151	γ-dodecalactone	2409	38.00	175	?-methyltetradecanoic acid[c]	2789	61.17
152	unknown	2422	4.52	176	pentadecanoic acid	2821	183.63
153	γ-2-dodecenolactone	2428	16.07	177	2-heptadecanone *O*-methyloxime[c]	2827	10.48
154	indole	2435	2.78	178	dioctyl adipate	2846	3.80
155	methyl stearate	2445	9.27	179	unknown	2855	7.76
156	δ-dodecalactone	2458	60.50	180	14-methylpentadecanoic acid	2861	5.61
157	dodecanoic acid	2517	1928.22	181	*N,N*-dibutylhexanamide	2869	19.46
158	dihydrophytol	2537	2.78	182	δ-heptadecalactone	2903	3.80
159	1,13-tridecanolide	2548	26.66	183	palmitic acid	2946	1766.80
160	11-methyldodecanoic acid	2555	15.02	184	palmitoleic acid	2960	179.75
161	11-dodecenoic acid[c]	2565	24.10	185	15-methylhexadecanoic acid	2980	10.48
162	carboxylic acid (C_{13})[c]	2571	6.80	186	16-hexadecanolactone	2990	14.51
163	1-octadecanol and tridecanoic acid	2603	34.37	187	unknown	2998	20.97
164	2-pentadecanone *O*-methyloxime[c]	2619	4.54	188	heptadecanoic acid	3027	16.63
165	*p*-phenylaniline and γ-tetradecalactone	2636	6.16	189	unknown	3055	6.37
166	(*E*)-phytol	2646	4.11	190	?-heptadecenoic acid[c]	3062	11.95
167	12-methyltridecanoic acid	2657	29.88	191	unknown	3068	8.48
168	unknown	2685	3.71	192	unknown	3079	7.05
169	(*Z*)-9-octadecenal	2693	5.10	193	unknown	3099	2.45
170	δ-pentadecalactone	2693	38.93	194	unknown	3113	7.05
171	tetradecanoic acid	2733	2293.26	195	stearic acid	3136	77.63
172	unknown	2757	6.16	196	oleic acid	3173	281.45
173	myristoleic acid	2768	191.78		total		11790.88
174	13-methyltetradecanoic acid	2776	6.35				

[a] Peak number in Figure 1. [b] Modified Kovats indices calculated for DB-Wax capillary column on the GC system. [c] Tentative identification by mass spectrum alone.

fraction of the skim milk. These compounds are released from triglycerides and phospholipids by the action of milk lipase or bacterial lipases, and their presence in excess results in a rancid flavor defect (Badings, 1991). Among the free fatty acids, even-carbon-numbered saturated fatty acids (C_8–C_{16}) were most abundant. In spite of the relatively high odor thresholds (0.2–0.8 ppm; Osawa, 1987), these compounds have buttery, milky, creamy, or waxy odor. Therefore, they were considered to form an important part on the skim milk flavor. Although the threshold value for other free fatty acids has not been reported, they could somewhat contribute to the flavor of the skim milk since most of them are present in large quantity compared with other volatiles (Table 1).

Among esters, the main compounds were short-chain fatty acid ethyl esters and long-chain fatty acid methyl esters. It seems probable that the short-chain alcohols and free fatty acids were esterified by action of bacterial esterase. Considering their odor characteristics, they may have slight concern in milk flavor.

The only two furan derivatives were furfural and furfuryl alcohol, which are known as products of sugar degradation and dehydration by Maillard reaction. These furans might be derived from milk carbohydrates, mainly lactose. They are associated with a sweet, nutty, and caramel-like aroma and could be important for the odor of a condensed milk. However, they may not contribute to the aroma of the skim milk, since the level was much below the perception threshold for these two compounds (100–300 ppb in water; Osawa, 1987).

2,6-Di-*tert*-butyl-*p*-cresol (BHT) was the most predominant phenolic compound in the extract (95% of phenolic fraction). A large portion of it may be chemical added to diethyl ether as an antioxidant, and we had to use the ether prepurified by distillation. Other interesting compounds were phenol, *p*-cresol, and *m*-cresol, which are well-known to have a medicinal or smoky odor. Lignin and other phenolic compounds are degraded by microorganisms into phenols. Also, these compounds have been found previously as conjugates in cows' skim milk (Brewington et al., 1974). Although these phenols may contribute to the off-flavor of milk (Waker and Manning, 1976), they were present in low concentrations.

Lactones with more than 10 carbon atoms were identified in the present study and were most represented by γ- and δ-lactones, the products of dehydration and cyclization of 4- and 5-hydroxy acids, respectively, by heating (Forss, 1979). Among lactones which are characterized by milky, buttery, or creamy odor, δ-decalactone and δ-dodecalactone are associated with the smell of milk with sweet and body attributes (Osawa, 1987). They could be very important contributors to the flavor of the skim milk powder along with free fatty acids. The content of δ-lactones was higher than that of γ-lactones, and this may be due to the predominance of 5-hydroxy acids in bovine milk fat.

The most important odorants among the N-containing compounds identified were indoles, which may be metabolic products of tryptophan. It has also been demonstrated that indoles can be transferred from the forage by the rumen, respiratory tract, or cow's metabolism (Honkanen et al., 1964; Forss, 1979). Badings (1991) suggested that it causes a flavor defect termed weed taints, as it produces a nauseating smell when present in very high amount. However, it has also been reported that indole at a certain level (60 ppb) has a creamy odor and is an important component in butter (Urbach et al., 1972). Thus, when present at low level, this compound may probably enhance the creamy odor and make the flavor of milk more attractive. Although they were identified tentatively by mass spectrum alone, methyl ketone *O*-methyloximes were reported for the first time in milk. They were considered to be formed from carbonyls and amines widely identified in milk, but the detailed pathway of their formation was unknown. Moreover, the threshold value for these oximes has not been reported, so it is impossible to discuss their contribution to the aroma of milk.

Compounds containing both N and S atoms are generally very flavor-active constituents due to their characteristic odor. Thus, cyclohexyl isothiocyanate and benzothiazole can contribute to the skim milk flavor. The silanized compounds may be contaminants of silicone grease used to seal the ground-glass joint of the SDE apparatus.

In conclusion, results obtained showed that levels of flavor compounds in the skim milk powder were very low and their compositions extremely complicated. Among them, free fatty acids and lactones present in relatively high level were considered to be fundamental contributors

to the skim milk flavor. Moreover, aldehydes, aromatic hydrocarbons, and a part of the heterocyclic compounds, such as indoles or thiazole, seem to participate indirectly in the skim milk flavor.

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