Volatile Compounds Formed from Cooked Whole Egg, Egg Yolk, and Egg White

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Headspace samples obtained from heated whole egg, egg white, and egg yolk with use of a simultaneous purging-extraction apparatus were analyzed by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS). The volatile chemicals identified included aldehydes, ketones, alcohols, indoles, sulfides, nitriles, furans, thiazoles, thiophenes, pyridines, pyrroles, phenols, pyrazines, benzenes, and miscellaneous compounds. A total of 141 compounds were isolated and identified: 87 from whole egg, 75 from egg yolk, and 57 from egg white. Cooked whole egg contained alkylbenzenes, nitriles, and ketones as major components. Cooked egg yolk had large numbers of aldehydes and pyrazines, while the major components of cooked egg white were ketones and pyrazines.

There have been virtually no reports on the analysis of headspace volatiles collected from heated egg samples. Only a few papers on the composition of volatiles of cooked egg have been published (MacLeod and Cave, 1975, 1976). Macleod and Cave (1975) identified 65 volatile compounds out of 116 components isolated from cooked eggs. They used a simultaneous steam distillation-solvent extraction apparatus that collected steam volatile chemicals rather than headspace volatiles. They found a series of saturated straight-chain hydrocarbons from C_7 to C_{17} in the extracts, with *n*-pentadecane the major constituent of the series at about 4% of the extract. Later, the same authors studied the variations of flavor components in samples from different types of eggs and egg products (Macleod and Cave, 1976). They reported no significant differences between the results for battery eggs and free range eggs and between those for white-shelled and brown-shelled eggs. Differences were observed between eggs of different ages and between egg yolk and whole egg. Kato et al. (1978), who reported pyridines, pyrazines, and thiazoles in the heated egg white, suggested that heterocyclic nitrogen-containing compounds play a major role in cooked egg flavor.

In the present study, volatile flavor chemicals formed in cooked whole egg, egg yolk, and egg white were isolated and identified.

EXPERIMENTAL SECTION

Materials. Brown-shelled eggs were obtained from free-range chickens (Tamba-machi, Funai-gun, Kyoto, Japan). The feed was a mixture of wheat, oyster shell, barley, soya, rice bran, maize, fish meal, and pasturage. The eggs used were 3-4 days old. Gas chromatographic authentic chemicals were purchased from commercial sources and used without further treatment.

Sample Preparation. Volatile flavor chemicals formed from heated egg samples (whole egg, egg yolk, egg white) were collected with use of a simultaneous purging-extraction apparatus developed by Umano and Shibamoto (1987; Figure 1). An egg sample was placed in a three-neck round-bottom flask and heated at 200 °C with a sand-bath for 1 h. The amounts of whole egg, egg yolk, and egg white used were 538, 377, and 500 g, respectively. The sample was stirred during heating. The sand bath temperature was controlled by a thermistor probe. The volatile chemicals formed were purged with purified air (1 mL/min) into a water trap (250 mL), and dissolved chemicals were simultaneously extracted with 70 mL of dichloromethane. The water trap temperature (15 °C) was controlled with a Brinkman RM6 constant-temperature water circulator. After the extract was dried over anhydrous sodium sulfate, dichloromethane was removed by fractional distillation on a Vigreux column. The solvent was further reduced with a purified nitrogen stream to a final sample weight 150 mg.

Analysis of Volatiles. The GC Kovats retention index (*I*) and MS fragmentation pattern of each component were compared to those of the authentic compounds. Quantitative analysis of GC components was done by the method described by Ettre (1967), using diallyl succinate as an internal standard. A Shimazu Model 9A GC equipped with a flame ionization detector (FID) and a 30 m × 0.25 mm (i.d.) bonded-phase DB-WAX fused silica capillary column (J&W Scientific, Folsom, CA) was used. The oven temperature was held at 40 °C for 10 min and programmed to 200 °C at 2 °C/min. The helium carrier gas flow rate was 30 cm/s. Detector and injector temperatures were 250 °C. A Finnigan MAT Model 800 ion trap MS interfaced to a Varian Model 3500 GC equipped with a 30 m × 0.25 mm (i.d.) bonded-phase DB-WAX fused silica capillary column was used for GC/MS analysis of volatiles. The GC oven conditions were as described for the Shimazu instrument.

RESULTS AND DISCUSSION

It is generally recognized that cooked eggs do not possess strong characteristic flavors. However, eggs do contain proteins and lipid in large quantities, suggesting the possibility of formation of flavor compounds via the Maillard reaction. A total of 141 volatile compounds were identified in cooked whole egg, egg yolk, and egg white in the present study. Table I lists the volatile compounds identified in the present study, grouped by chemical class. Table I was prepared in a format used previously. Some compounds were not identified positively because of the lack of authentic compounds, so the Kovats index could not be compared.

The percent yields of total volatiles from cooked whole egg, egg yolk, and egg white were 0.0083, 0.0030, and 0.0014%, respectively. Whole egg produced considerably more volatiles than did either egg yolk or egg white alone. Cooked whole egg samples contained nitriles (total peak area (%) = 20.73), alkylbenzenes (20.07), ketones (12.43), aldehydes (10.73), pyrazines (6.65), pyrroles (6.64), and pyridines (2.81) as major components. Cooked egg yolk contained aldehydes (64.12) and pyrazines (8.69) as major constituents, while cooked egg white contained

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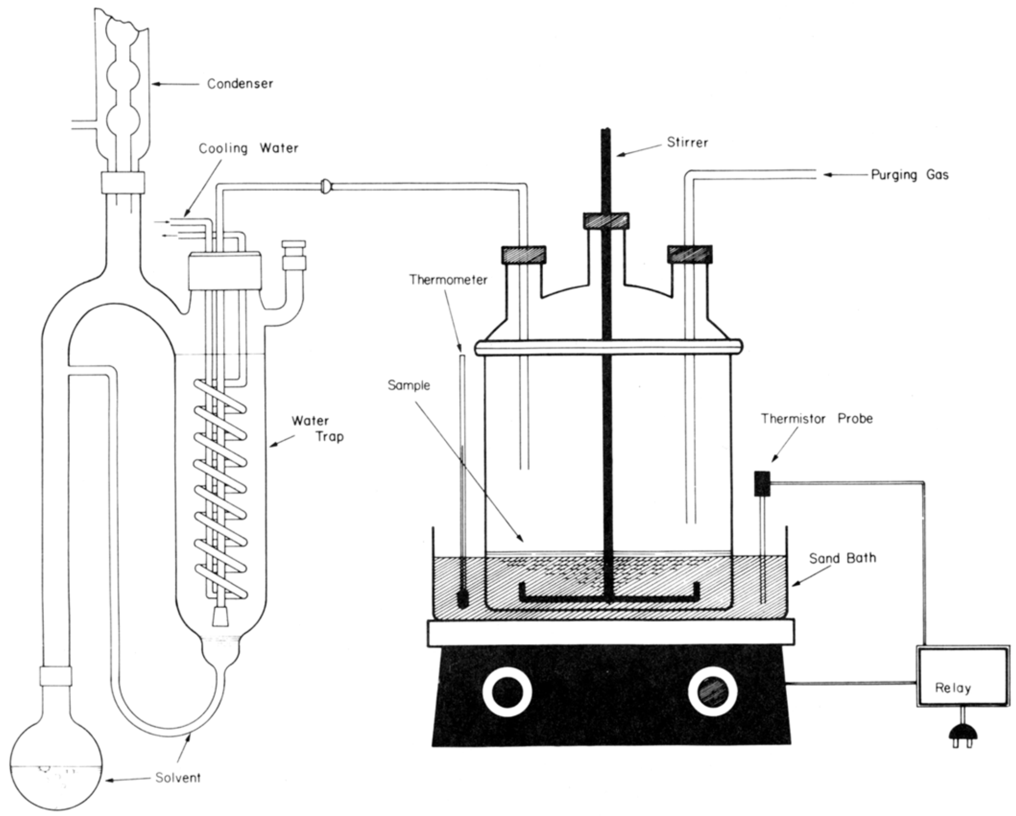

Figure 1. Apparatus used for collection of headspace volatiles formed from heated egg samples.

ketones (31.08), pyrazines (18.24), and nitriles (17.61) as major components.

Aldehydes, along with pyrazines, were the major components of volatiles of cooked egg yolk. Whole egg and egg white, on the other hand, yielded only a few volatile aldehydes. MacLeod and Cave (1976) reported that 2-methylbutanal was the most abundant aldehyde in egg yolk and suggested that certain amino acids play a role in the formation of 2-methylbutanal. In the present study, 2-methylpropanal was the major aldehyde in egg yolk and whole egg volatiles. 2-Methylpropanal may form from valine upon Strecker degradation or from oxidative decomposition of lipids (Lundberg, 1962). Egg yolk is rich in both valine and lipids (Belitz and Grosch, 1987). Low molecular weight ketones such as acetone were found in large amounts in whole egg and egg white, but not in egg yolk. On the other hand, higher molecular weight 2-methylketones (C_7–C_{10}) were found only in egg yolk. The formation of 2-methyl ketones from glycerides has been proposed (Shibamoto et al., 1980). Several alcohols were identified in egg yolk, but only two were found in whole egg and none was in egg white, suggesting that alcohol formation is also associated with lipid oxidation.

All samples from cooked whole egg, egg yolk, and egg white had a characteristic cooked egg flavor. Sulfides contribute a sulfurous flavor to cooked eggs. It has been proposed that sulfides form from sulfur-containing amino acids such as methionine (Wainwright et al., 1972). Methionine is evenly distributed between egg yolk and egg white (Belitz and Grosch, 1987). Sulfides were distributed almost evenly among the three different samples. The presence of a large number of nitriles is somewhat unusual in volatiles from cooked foods. MacLeod and Cave (1975, 1976) also reported several nitriles in cooked egg volatiles. Nitriles were found only in whole egg and egg white samples except propanenitrile, which was found in all three samples. 4-Methylpentanenitrile was the most abundant nitrile in whole egg and egg white, consistent with the report of MacLeod and Cave (1975).

Nitrogen-containing heterocyclic compounds including pyridines, pyrazines, pyrroles, and thiazoles were the major flavor compounds identified in the egg samples. No pyridines have been reported in cooked egg samples except in heated ovalbumin (Kato et al., 1978). Ovalbumin is the major egg white protein comprising 60% of total egg white protein. Many alkylpyrazines were identified in the present study. Pyrazines are the major volatile flavor chemicals produced in Maillard reactions, and they contribute roasted or toasted flavors to cooked foods. In the Maillard reaction, carbonyl compounds produced from sugars or lipids undergo secondary reactions with amino acids or protein to give heterocyclic compounds including pyrazines, pyrroles, thiophenes, and thiazoles. A whole egg contains ideal reactants for the Maillard reaction, including proteins, amino acids, carbohydrates, and lipids. Pyrroles have not received as much attention as pyrazines from flavor chemists even though the number of derivatives identified in cooked foods is almost the same as that of pyrazines. The numbers of pyrazines and pyrroles identified in the egg samples are also almost equal in the present study. Some pyrroles have been found to contribute to off-flavors of food products (Peterson et al., 1975). On the other hand, 2-acetylpyrrole, which was found in whole egg and egg yolk samples, has a pleasant, caramel-like flavor. Thiazoles were found only in egg white samples, with the exception of methylpropylthiazole. Thiazoles were proposed to form from a reaction between a sugar and the sulfur-containing amino acid cysteine. Egg white, which is rich in proteins, gave the most thiazoles among the egg samles in the present study.

As MacLeod and Cave (1975) reported, a series of straight-chain saturated alkylbenzenes was identified in the present study. Toluene was one of the major constituents of egg volatiles. Alkylbenzenes were proposed

Table I. Volatile Compounds Identified in Cooked Egg Samples

compound	GC peak area,[a] % whole egg	egg yolk	egg white	ident I	MS	compound	GC peak area,[a] % whole egg	egg yolk	egg white	ident I	MS
						Aldehydes					
propanal	b	5.55	b	+	+	(E)-2-octenal	b	0.71	b	+	+
2-methylpropanal	8.31	40.18	2.74	+	+	3-(methylthio)propanal	b	0.41	b	+	+
butanal	c	1.00	b	+	+	decanal	b	0.29	b	+	+
pentanal	b	0.53	b	+	+	(E,E)-2,4-heptadienal	b	0.31	b	+	+
hexanal	0.34	4.92	b	+	+	(E)-2-nonenal	b	0.14	b	+	+
(E)-2-methyl-2-butenal	0.43	0.65	b	+	+	(E,E)-2,4-octadienal	b	0.06	b	+	+
(E)-2-pentenal	b	0.49	b	+	+	(E)-2-undecenal	b	c	b	+	+
(E)-2-methyl-2-pentenal	b	0.14	b	+	+	(E,E)-2,4-decadienal	b	0.65	b	+	+
heptanal	0.11	0.76	b	+	+	hexadecanal	b	c	b	+	+
(E)-2-hexenal	b	0.12	b	+	+	benzaldehyde	1.40	2.14	1.65	+	+
(Z)-4-heptenal	b	0.18	b	+	+	phenylacetaldehyde	b	2.39	b	+	+
octanal	b	0.92	b	+	+	5-methylfurfural	0.07	0.27	b	+	+
nonanal	0.07	1.31	0.14	+	+	3-thiophenecarboxaldehyde	b	c	b	+	+
						Ketones					
acetone	10.02	b	22.39	+	+	4-phenyl-2-butanone	0.13	b	c	+	+
2-butanone	0.21	b	b	+	+	acetophenone	0.05	b	0.23	+	+
2-pentanone	0.50	b	b	+	+	2,3-pentanedione	b	0.65	0.23	+	+
2,3-butanedione	b	1.84	3.20	+	+	2-heptanone	b	0.65	b	+	+
4-methyl-2-pentanone	0.66	b	b	+	+	2-octanone	b	0.20	c	+	+
3-methyl-2-pentanone	0.28	b	b	+	+	2-nonanone	b	0.55	b	+	+
5-methyl-2-hexanone	0.40	b	0.23	+	+	2-decanone	b	0.20	b	+	+
1-phenyl-2-propanone	0.18	b	0.18	+	+	2-hydroxy-2-methyl-4-pentanone	b	0.16	0.37	+	+
						Alcohols					
1-octen-3-ol	0.05	1.39	b	+	+	pentanol	b	0.57	b	+	+
2-methyl-2-butanol	b	0.12	b	+	+	heptanol	b	0.14	b	+	+
1-penten-3-ol	b	0.65	b	+	+	octanol	b	0.06	b	+	+
3-methyl-3-buten-2-ol	b	0.29	b	+	+	menthol	0.17	b	b	+	+
						Indoles					
indole	0.42	c	c	+	+	3-methylindole	0.03	b	c	+	+
						Sulfides					
methyl mercaptan	b	b	0.3.06	+	+	dimethyl disulfide	1.40	0.29	0.14	+	+
dimethyl sulfide	b	0.55	0.59	+	+	dimethyl trisulfide	0.11	0.29	b	+	+
						Nitriles					
2-methylpropanenitrile	2.01	b	b	+	+	4-methylpentanenitrile	6.51	b	5.76	+	+
propanenitrile	2.45	0.04	2.88	+	+	hexanenitrile	0.49	b	b	+	+
butanenitrile	1.00	b	1.10	+	+	5-methylhexanenitrile	0.26	b	b	+	+
3-methylbutanenitrile	4.54	b	4.30	+	+	4-(methylthio)butanenitrile	0.16	b	b	+	+
pentanenitrile	0.16	b	0.14	+	+	phenylacetonitrile	a	b	0.50	+	+
3-methylpentanenitrile	2.81	b	2.15	+	+	phenylpropanenitrile	0.34	b	0.78	+	+
						Furans					
2-methylfuran	b	b	0.69	+	+	2-butylfuran	b	b	b	+	+
2-ethylfuran	b	0.67	b	+	+	2-pentylfuran	0.52	1.80	b	+	+
						Thiazoles					
4-methylthiazole	b	b	0.37	+	+	2(or4)-methyl-4(or2)-propylthiazole	b	b	0.32		+
2-methylthiazole	b	b	0.96	+	+	isothiazole	b	0.18	b		+
2,4,5-trimethylthiazole	b	b	0.18	+	+						
						Thiophenes					
thiophene	b	0.12	b	+	+	3-methylthiophene	b	b	0.14	+	+
2-methylthiophene	b	b	0.27	+	+						
						Pyridines					
pyridine	1.28	b	0.46	+	+	2,4-dimethylpyridine	0.18	b	b	+	+
2-methylpyridine	0.52	b	0.41	+	+	3-ethylpyridine	0.13	b	b	+	+
3-methylpyridine	0.34	b	0.14	+	+	5-ethyl-2-methylpyridine	0.06	b	b		+
4-methylpyridine	0.23	b	c	+	+	3,4-dimethylpyridine	0.07	b	b	+	+
						Pyrroles					
pyrrole	3.54	0.92	2.24	+	+	2-ethylpyrrole	0.33	b	b	+	+
1-acetylpyrrole	0.21	1.56	b	+	+	2-ethyl-4-methylpyrrole	0.59	b	b		+
2-methylpyrrole	1.08	0.12	0.69	+	+	2-ethyl-3,5-dimethylpyrrole	0.08	b	b		+
3-methylpyrrole	0.41	0.12	b		+	2-ethyl-3,4,5-trimethylpyrrole	b	b	0.09		+
2,5-dimethylpyrrole	0.40	b	0.27	+	+						
						Phenols					
phenol	0.23	b	0.23	+	+	4-ethylphenol	0.05	b	b	+	+
4-methylphenol	0.31	b	b	+	+						
						Pyrazines					
pyrazine	0.19	b	4.43	+	+	2,3,5-trimethylpyrazine	0.90	1.02	0.59	+	+
2-methylpyrazine	0.76	1.14	9.60	+	+	2-ethyl-3,6-dimethylpyrazine	0.59	0.31	b	+	+
2,5-dimethylpyrazine	1.94	4.98	1.60	+	+	2-ethyl-3,5-dimethylpyrazine	0.20	b	b	+	+
2,6-dimethylpyrazine	0.50	0.35	1.19	+	+	2-methyl-5(or6)-vinylpyrazine	0.08	b	b		+
2-ethylpyrazine	0.23	0.10	0.23	+	+	2-methyl-5(or6)-vinylpyrazine	0.25	b	b		+
2,3-dimethylpyrazine	0.16	0.16	0.23	+	+	dimethyl-2-vinylpyrazine	0.24	b	b		+
2-ethyl-6-methylpyrazine	0.10	0.14	0.37	+	+	2-methyl-5(or6)-(1-propenyl)pyrazine	a	0.14	b		+
2-ethyl-5-methylpyrazine	0.43	0.35	b	+	+	2,5-dimethyl-3-(3-methylbutyl)pyrazine	0.08	b	b		+

[a] Peak area of solvent was excluded. [b] Not detected. [c] Value less than 0.01.

to form from thermal sugar degradation (Heyns et al., 1966). However, a browning model system consisting of a sugar and an amino acid did not produce significant amounts of alkylbenzenes (Umano and Shibamoto, 1987). On the other hand, several alkylfurans, which are well-known sugar degradation products, were found in all the egg samples in the present study. Phenoles and indoles, which with the exception of phenol were found only in the whole egg samples, may be derived from their corresponding amino acids tyrosine and tryptophan, respectively. Thialdine (2,4,6-trimethyldihydro-1,3,5-dithiazine), which has a cooked shrimplike odor, has never been reported in egg samples prior to the present study. Thialdine has been reported in many other cooked foods such as beef broth (Brinkman et al., 1972), lamb fat (Buttery et al., 1977), and shrimp (Choi et al., 1983).

In order to obtain sufficient headspace volatiles, it was necessary to heat samples for 1 h, which may exceed usual cooking conditions for eggs. Therefore, some of the volatiles identified in the present study may not be obtained from normally cooked eggs.

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Registry No. Propanal, 123-38-6; 2-methylpropanal, 78-84-2; butanal, 123-72-8; pentanal, 110-62-3; hexanal, 66-25-1; (*E*)-2-methyl-2-butenal, 497-03-0; (*E*)-2-pentenal, 1576-87-0; (*E*)-2-methyl-2-pentenal, 14250-96-5; heptanal, 111-71-7; (*E*)-2-hexenal, 6728-26-3; (*Z*)-4-heptenal, 6728-31-0; octanal, 124-13-0; nonanal, 124-19-6; (*E*)-2-octenal, 2548-87-0; 3-(methylthio)propanal, 3268-49-3; decanal, 112-31-2; (*E,E*)-2,4-heptadienal, 4313-03-5; (*E*)-2-nonenal, 18829-56-6; (*E,E*)-2,4-octadienal, 30361-28-5; (*E*)-2-undecenal, 53448-07-0; (*E,E*)-2,4-decadienal, 25152-84-5; hexadecanal, 629-80-1; benzaldehyde, 100-52-7; phenylactaldehyde, 122-78-1; 5-methylfurfural, 620-02-0; 3-thiophenecarboxaldehyde, 498-62-4; acetone, 67-64-1; 2-butanone, 78-93-3; 2-pentanone, 107-87-9; 2,3-butanedione, 431-03-8; 4-methyl-2-pentanone, 108-10-1; 3-methyl-2-pentanone, 565-61-7; 5-methyl-2-hexanone, 110-12-3; 1-phenyl-2-propanone, 103-79-7; 4-phenyl-2-butanone, 2550-26-7; acetophenone, 98-86-2; 2,3-pentanedione, 600-14-6; 2-heptanone, 110-43-0; 2-octanone, 111-13-7; 2-nonanone, 821-55-6; 2-decanone, 693-54-9; 2-hydroxy-2-methyl-4-pentanone, 123-42-2; 1-octen-3-ol, 3391-86-4; 2-methyl-2-butanol, 75-85-4; 1-penten-3-ol, 616-25-1; 3-methyl-3-buten-2-ol, 10473-14-0; pentanol, 30899-19-5; heptanol, 53535-33-4; octanol, 29063-28-3; menthol, 89-78-1; indole, 120-72-9; 3-methylindole, 83-34-1; methyl mercaptan, 74-93-1; dimethyl sulfide, 75-18-3; dimethyl disulfide, 624-92-0; dimethyl trisulfide, 3658-80-8; 2-methylpropanenitrile, 78-82-0; propanenitrile, 107-12-0; butanenitrile, 109-74-0; 3-methylbutanenitrile, 625-28-5; pentanenitrile, 110-59-8; 3-methylpentanenitrile, 21101-88-2; 4-methylpentanenitrile, 542-54-1; hexanenitrile, 628-73-9; 5-methylhexanenitrile, 19424-34-1; 4-(methylthio)butanenitrile, 124154-43-4; phenylacetonitrile, 140-29-4; phenylpropanenitrile, 124154-44-5; 2-methylfuran, 534-22-5; 2-ethylfuran, 3208-16-0; 2-pentylfuran, 3777-69-3; 4-methylthiazole, 693-95-8; 2-methylthiazole, 3581-87-1; 2,4,5-trimethylthiazole, 13623-11-5; 2-methyl-4-propylthiazole, 41981-63-9; isothiazole, 288-16-4; 4-methyl-2-propylthiazole, 52414-87-6; thiophene, 110-02-1; 2-methylthiophene, 554-14-3; 3-methylthiophene, 616-44-4; pyridine, 110-86-1; 2-methylpyridine, 109-06-8; 3-methylpyridine, 108-99-6; 4-methylpyridine, 108-89-4; 2,4-dimethylpyridine, 108-47-4; 3-ethylpyridine, 536-78-7; 5-ethyl-2-methylpyridine, 104-90-5; 3,4-dimethylpyridine, 583-58-4; pyrrole, 109-97-7; 1-acetylpyrrole, 609-41-6; 2-methylpyrrole, 636-41-9; 3-methylpyrrole, 616-43-3; 2,5-dimethylpyrrole, 625-84-3; 2-ethylpyrrole, 1551-06-0; 2-ethyl-4-methylpyrrole, 69687-77-0; 2-ethyl-3,5-dimethylpyrrole, 32990-59-3; 2-ethyl-3,4,5-trimethylpyrrole, 69687-79-2; phenol, 108-95-2; 4-methylphenol, 106-44-5; 4-ethylphenol, 123-07-9; pyrazine, 290-37-9; 2-methylpyrazine, 109-08-0; 2,5-dimethylpyrazine, 123-32-0; 2,6-dimethylpyrazine, 108-50-9; 2-ethylpyrazine, 13925-00-3; 2,3-dimethylpyrazine, 5910-89-4; 2-ethyl-6-methylpyrazine, 13925-03-6; 2-ethyl-5-methylpyrazine, 13360-64-0; 2,3,5-trimethylpyrazine, 14667-55-1; 2-ethyl-3,6-dimethylpyrazine, 13360-65-1; 2-ethyl-3,5-dimethylpyrazine, 13925-07-0; 2-methyl-5-vinylpyrazine, 13925-08-1; 2-methyl-6-vinylpyrazine, 13925-09-2; dimethyl-2-vinylpyrazine, 124154-45-6; 2-methyl-5-(1-propenyl)pyrazine, 108653-51-6; 2-methyl-6-(1-propenyl)pyrazine, 104638-11-1; 2,5-dimethyl-3-(3-methylbutyl)pyrazine, 18433-98-2; benzene, 71-43-2; toluene, 108-88-3; ethylbenzene, 100-41-4; 1,3-dimethylbenzene, 108-38-3; 1,2,4-trimethylbenzene, 95-63-6; propylbenzene, 103-65-1; butylbenzene, 104-51-8; 1-phenyl-1-propene, 637-50-3; pentylbenzene, 538-68-1; styrene, 100-42-5; *p*-cymene, 99-87-6; propoxybenzene, 622-85-5; 3-ethyl-2-pentene, 816-79-5; 3-ethyl-2-methyl-1,3-hexadiene, 61142-36-7; undecane, 1120-21-4; dodecane, 112-40-3; limonene, 138-86-3; γ-terpinene, 99-85-4; 1-methyl-4-propyl-2-pyrazoline, 33063-77-3; methylindan, 27133-93-3; butenylcyclohexene, 74664-14-5; methyl palmitate, 112-39-0; 2,4,5-trimethyloxazole, 20662-84-4; dimethylformamide, 68-12-2; 2,4,6-trimethyldihydro-1,3,5-dithiazine, 638-17-5.

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