

Identification of Major Volatile Odor Compounds in Frankfurters

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More than 100 volatile compounds have been identified in the headspace of frankfurter sausages. Most abundant were the terpene hydrocarbons, monoterpene alcohols, phenyl propanoids, and phenols. Separate analyses of solutions of spices and smoke demonstrated that most of the terpenes were derived from the spices, whereas the phenols originated mostly from the smoke ingredients. Many of the compounds contributing to the overall odor of the frankfurters have been identified. Some odors, characteristic of the smokiness and spiciness of frankfurters, were caused by phenols and terpenes, whereas others were due to compounds derived from the meat fraction; these compounds included aldehydes, ketones, furan thiols, and alicyclic sulfur compounds.

Keywords: Flavor; odor; volatile; odor compound; frankfurter

INTRODUCTION

Frankfurters are nonfermented sausages of the emulsion type. They are usually formulated from beef and pork and are flavored with spices and smoke. These sausages have been the subject of various studies concerning their textural, structural, and sensory properties (Simon et al., 1980; Lee et al., 1987; Beilken et al., 1990), but little is known about their volatile flavor constituents.

Other types of processed meat products, such as fermented (Berger et al., 1990; Johansson et al., 1994; Mateo and Zumalacarregui, 1996) or nonfermented sausages (Shiratsuchi et al., 1993), beefburgers (El-Magoli et al., 1996), processed duck fillet (Lesimple et al., 1995), or ham (Berdague et al., 1991; Garcia et al., 1991; Barbieri et al., 1992; Sabio et al., 1998), have been more widely investigated. Many of the volatile compounds identified in these products have been found to originate from smoke and seasoning, whereas others were the result of the enzymatic or bacterial reactions occurring in the meat (Johansson et al., 1994; Berger et al., 1990; Mateo and Zumalacarregui, 1996). When pepper was included in the formulation of these products, terpenes were the chemical compounds with the highest area proportion in the total chromatogram (Johansson et al., 1994; Berger et al., 1990). Dry fermented sausages, which had been formulated with garlic, contained many sulfur-containing volatile compounds (Johansson et al., 1994; Berger et al., 1990; Mateo and Zumalacarregui, 1996). Aldehydes appeared to be the major volatiles detected in dry cured ham (Baloga et al., 1990; Berdague et al., 1991; Barbieri et al., 1992; Sabio et al., 1998). Great differences in volatile composition have been observed among examples of the same product; for example, the typical feature of the Parma ham was defined by a high content of various kinds of esters (Barbieri et al., 1992) compared with the

French and Iberian products, which were characterized by a greater number of alcohols and aldehydes (Berdague et al., 1991; Garcia et al., 1991; Sabio et al., 1998).

The odor impact compounds of the above meat products have not been widely investigated, apart from those contributing to the aroma of dried sausages; these seem to be acids ("vinegar, cheesy" odors), aldehydes ("greenish, synthetic, deep-fried, herblike" smells), esters ("fruity, candy" odors), and ketones ("fruity, cheesy") (Berdague et al., 1993; Stahnke, 1994; Stahnke, 1995).

The present paper reports identities for the major volatile components of experimentally prepared frankfurters and compares them with those detected in the meat, spice, or smoke ingredients of these sausages. Identities for volatile aroma compounds responsible for the characteristic flavor of these frankfurters are suggested. The key odors for these frankfurters are compared with those obtained for two brands of commercial frankfurters.

MATERIALS AND METHODS

Materials. *Frankfurters.* Experimentally prepared frankfurters were manufactured in conjunction with the National Food Centre (Dublin, Ireland). The main ingredients used for the formulation of these frankfurters are summarized in Table 1.

Frankfurters were prepared as described by Hughes et al. (1997) and cooked in a Kerres Showsmoker (CS 350 EL, Kerres Gmbn & Co., KG, Sulzbach/Murr, Germany) for 1 h at 80 °C. The cooked frankfurters were showered in cold water and kept overnight at 4 °C.

To study the volatile composition of basic meat ingredients from the above frankfurters, sausages were prepared with the same ingredients but lacking the spices, smoke, and sodium ascorbate. After stuffing, these sausages were cooked in a water bath at 80 °C for 1 h, then placed in cold water just after cooking, and kept overnight at 4 °C. For both types of frankfurters (with or without spices and smoke), casings were removed the day after cooking and the products vacuum-packed and frozen at -20 °C until required.

Commercial frankfurters were purchased from local supermarkets and were a German make (Konecke, imported by Chineham International Ltd., Basingstoke, U.K.) and a French

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Table 1. Composition of Experimentally Prepared Frankfurters

ingredients	source
fresh beef ^a (27/28/29)	The National Food Centre abattoir, Dublin, Ireland
pork ^a (25/26/27)	Dairygold meat processing plant, Michelstown, Ireland
pork backfat ^a (34/13/4)	Dairygold meat processing plant, Michelstown, Ireland
distilled water ^a (11/29/37)	Distilled in a Aquatron A4D, J.Bibby Science Products, France
1.5% nitrite salt	99.4% sodium chloride with 0.6% sodium nitrite (E250), Colorozout, Degens, Holland
0.25% sodium tripolyphosphate	E450b, Albright & Wilson Ltd., England
0.05% sodium ascorbate	E301, Dalgety Food Ingredients, Dublin, Ireland
0.5% spice mix	spice mix (mostly pepper, mint, ginger, nutmeg, and cardamon) Vienna Gold, Rasp & Co., Kulmbach, Germany
0.05% smoke flavoring	hickory smoke flavoring D402V, Dalgety Food Ingredients, Dublin, Ireland

^a The quantity of these ingredients was such that, in the final frankfurter, the total protein content was 12.5% and the fat content was 30, 12, or 5%. The numbers in parentheses give the percentage (approximated to the closest whole number) used for the formulation of the 30, 12, and 5% fat frankfurters, respectively.

Table 2. Composition of Commercial Frankfurters As Listed by the Manufacturers

Konecke	Herta
minimum of 70% meat (pork and beef), water, salt, sodium nitrite E250, milk protein, spices, sugar, stabilizer E331, natural flavor enhancer, smoke	minimum of 75% meat (pork and beef), water, salt, whey protein albumen, polyphosphates, hydrolyzed vegetable protein, spices, antioxidant (sodium ascorbate), preservatives (sodium nitrite, potassium nitrate), garlic

make (Herta, imported by Nestle U.K. Ltd., Surrey, U.K.). Their stated compositions are given in Table 2.

Solutions of Spices and Smoke. Solutions of spices and smoke were obtained by diluting 1.25 g of spice mix or 0.125 g of liquid smoke in 250 mL of distilled water, so that their final concentration was the same as in the frankfurters.

Standards. Standard solutions of volatile compounds were prepared at a concentration of ~ 10 ng μL^{-1} of authentic compound (Aldrich, Sigma, or Fluka, Dorset, U.K.) in hexane (glass-distilled grade, Rathburn Chemical Limited, Walkerburn, Scotland) or ethanol (A.R. 99.7%, Hayman Limited, Essex, U.K.).

Isolation of Headspace Volatiles. The headspace volatiles from frankfurters were collected onto a conditioned glass-lined stainless steel trap (95 \times 0.8 mm i.d.) containing 2.6 mg of Tenax GC (Scientific Glass Engineering Ltd., Milton Keynes, U.K.), using either a static or a dynamic headspace concentration method.

The static headspace collection method was used to collect odor compounds from frankfurters prior to GC/odor assessment. Commercial frankfurters or experimentally prepared frankfurters with spices and smoke (20 g) were chopped into 1 cm slices and then into 4 sectors and placed in a bottle (100 mL; Duran, Davidson and Hardy Ltd., Belfast, U.K.) sealed with a screw cap with PTFE protective seal and fitted with two bulkhead unions ($1/16 \times 1/16$ in., Scientific Glass Engineering Ltd.) into which a Tenax trap and a gastight syringe (10 mL; Series II, Scientific Glass Engineering Ltd.) could be fitted. A short length of stainless steel tubing (1 cm length $\times 1/16$ in. external diameter) joined the union fitted with the syringe with a 6.5 cm long PTFE tubing ($1/16$ in. i.d.) so that the flow of air from the syringe would be directed to just above the food sample. The bottle was placed in a water bath at 70 °C and the syringe fitted. The other union remained open, avoiding a pressure buildup in the system. After 20 min, a conditioned trap (preconditioned at 250 °C with a flow of helium at ~ 1 mL min^{-1} for 20 min) was fitted to the second union and 10 mL of air was injected through the syringe into the bottle, displacing the same volume of headspace onto the trap. After 5 min, the trap was removed and purged for 5 min with nitrogen (50 mL min^{-1}) to eliminate moisture from the trap. This method gave better reproducibility than an earlier variation of the method, which was used for the collection of volatiles from frankfurters without spices and smoke, as indicated by the coefficient of variance (CV) for peak areas (CV = 21 and 58%, respectively). In this earlier method, ~ 5 g of sausages (prepared as described above) was placed in a 40 mL vial (Pierce and Warriner Ltd., Chester, U.K.), fitted with an open-topped cap and a PTFE-coated septum, and were heated for 30 min in a water bath at 70 °C. Headspace volatiles (5

mL) were withdrawn by inserting the needle of a warmed (60 °C) gastight syringe (5 mL; Series II, Scientific Glass Engineering Ltd.) through the septum. The sample was injected onto a Tenax trap that had been attached to a water vacuum pump such that a gentle flow of air (~ 20 mL min^{-1}) drew the sample onto the trap. Residual water was removed from the trap with a flow of nitrogen (50 mL min^{-1} for 5 min).

A dynamic headspace collection method was used to collect volatiles prior to analysis by GC/MS. The volatiles released from 50 g of frankfurters during 30 min at 70 °C were swept onto a Tenax trap using a stream of nitrogen (50 mL min^{-1}) (Whitfield et al., 1988). Residual water was removed from the trap with a flow of nitrogen (50 mL min^{-1} for 5 min). Volatiles from the headspace of aqueous solutions of spices or smoke were also collected using this method.

Authentic standards (~ 10 ng of standard in 1 μL of hexane or ethanol) were injected onto Tenax traps using a syringe (5 μL , Scientific Glass Engineering Ltd.) while a slight vacuum was applied to one end of the trap using a water pump (flow rate of ~ 20 mL min^{-1}). Excess ethanol was removed by passage of nitrogen (50 mL min^{-1} for 5 min).

Capillary Gas Chromatography and Odor Assessment (GC/OA). Gas chromatography was performed using an HP 5890 Series II gas chromatograph (Hewlett-Packard, Wokingham, Berks, U.K.), fitted with a Unijector (Scientific Glass Engineering Ltd.). The collected volatiles were thermally desorbed at 250 °C for 5 min and refocused onto a 10 cm region at the front of a fused silica capillary column that had been precooled for 5 min with solid CO_2 . After desorption, the oven was heated rapidly to 60 °C and maintained at this temperature for 5 min before the temperature was increased at 4 °C min^{-1} to 220 °C (30 min). The effluent from the column was split between a flame ionization detector and an odor port to allow olfactory assessment of the separated volatiles. The odor port comprised a heated (220 °C) uncoated fused silica transfer line fitted with PTFE cup and an auxiliary flow of moist air (~ 15 mL/min). Most of the separations were performed on a CPWax 52CB capillary column (50 m \times 0.32 mm i.d., Chrompack Ltd., London, U.K.). Some analyses were performed on a CPSil 8CB capillary column (50 m \times 0.32 mm i.d., Chrompack Ltd.) to aid the confirmation of identities. GC/OA were conducted by four assessors on each type of frankfurter.

Capillary Gas Chromatography/Mass Spectrometry (GC/MS). The volatiles collected from the frankfurters with spices and smoke and from the solutions of spices or smoke were analyzed using an HP 5890A gas chromatograph connected to an HP 5970 mass selective detector (Hewlett-Packard). Those from the frankfurters without spices and smoke were analyzed using a Kratos MS25 RFA mass spec-

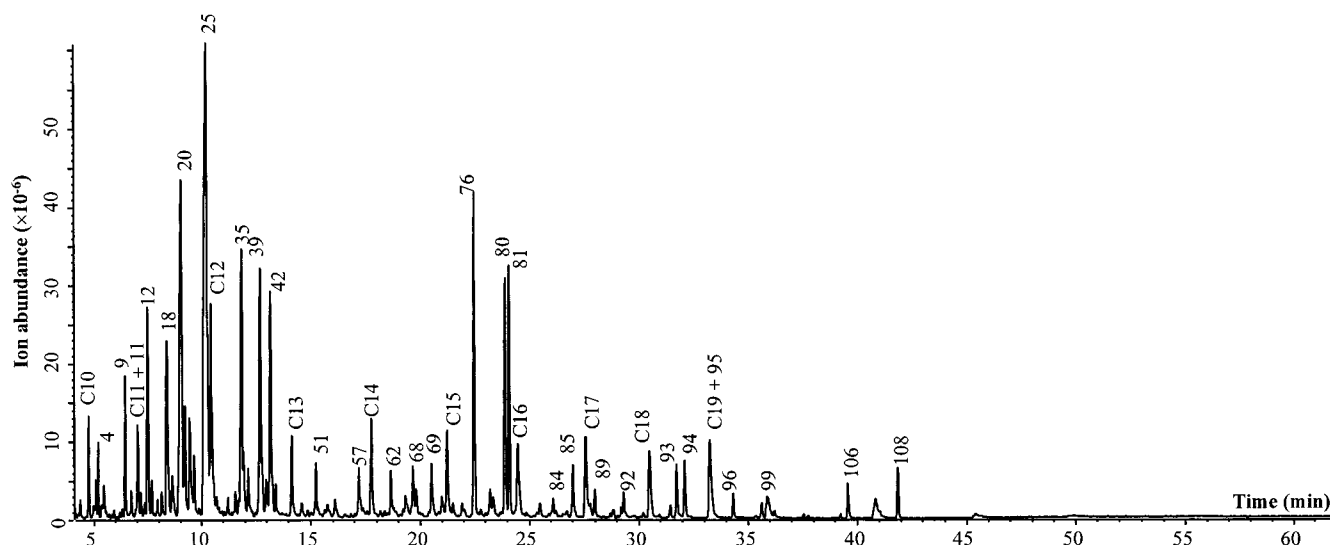


Figure 1. Ion chromatogram of the volatiles obtained by dynamic headspace concentration from frankfurters (with added alkanes, shown as C10, C11, etc.). The numbers correspond to the compounds listed in Table 3.

trometer (Kratos Analytical Ltd., Manchester, U.K.) connected to a Carlo Erba MFC 500 gas chromatograph (Manchester, U.K.). Both instruments were operated at 70 eV in the EI mode over the range 35–450 amu. Authentic standards were analyzed on both instruments. The injection technique and oven program were as described for GC/OA.

Positive identification of volatile compounds was confirmed by matching sample linear retention indices (LRI) on the two capillary columns, CPWax 52CB and CPSil 8CB, calculated according to the method of Van den Dool and Kratz (1963), and mass spectra with those of authentic standards under comparable conditions. Tentative identifications were based mainly on comparisons of the mass spectra of unknown compounds with published mass spectra.

RESULTS AND DISCUSSION

The principal volatile components of the headspace of frankfurters are given in order of elution on a CPWax 52CB capillary column in Table 3. Also listed are the volatile components identified in the headspace of frankfurters prepared without spices and smoke and of the solutions of spices and smoke. The identities of 85 of the compounds listed have been established by comparison of their linear retention indices (LRI) and mass spectral data with those of authentic substances, and probable identities have been suggested for a further 23 compounds. A typical chromatogram of the volatile profile is shown in Figure 1.

The key odors detected in frankfurters (with and without spices and smoke) are listed in Table 4 in order of elution on the CPWax 52CB capillary column and compared with those detected in commercial frankfurters. For each odor, an indication of the frequency of detection is reported, together with the LRI of the odor. In total, 40 odors were detected in the different types of frankfurters, 16 of which were detected in the frankfurters without spices and smoke; 29 were detected in the frankfurters with spices and liquid smoke, whereas 24 and 27 were detected in the commercial frankfurters, Herta and Konecke, respectively. Fourteen of the odors listed have been positively identified by comparison with odors and LRI values of authentic compounds on capillary columns of two different polarities. Possible identities have been suggested for a further 10 compounds. The 16 remaining unidentified

odors were often weak in intensity and not detected by all of the assessors (Table 4).

During the period of this study, the static headspace collection method was developed to improve reproducibility. Furthermore, two different GC/MS instruments were employed. For these reasons, a direct comparison of quantities of volatiles or odors detected is not possible. Nevertheless, an evaluation of the relative abundances of different compounds and the relative importance of the odors allows interesting conclusions to be drawn. Comparison of the headspace volatiles from the frankfurters with spices and liquid smoke with those detected in the separate ingredients (spices or smoke) or sausages without spices or smoke suggests that most abundant headspace compounds from frankfurters originated from the spices, with smaller quantities derived from the meat or smoke ingredients (Table 3). The volatile compounds and odors identified in these frankfurters will be discussed according to their apparent origin. Odor compounds from these experimental frankfurters will be then compared with those from two commercial brands of frankfurters.

Volatile Compounds Derived from the Meat Ingredients. Frankfurters without spices and smoke were dominated by lipid oxidation products. Among the 47 major volatile compounds that were positively identified in these sausages, 10 aldehydes, 9 alcohols, 5 ketones, 8 alkanes, 10 aromatic hydrocarbons, and 5 compounds from other classes were detected (Table 3). Most of these compounds have been reported previously as volatile components of cooked beef and pork (Maarse and Visscher, 1989). In a study comparing the flavor volatiles from cooked beef and pork meat systems, Mottram et al. (1982) demonstrated that the major volatiles from both of these meats were aldehydes and alcohols. It is, therefore, not surprising to find a majority of these compounds in these sausages, of which the main ingredients were beef, pork, and pork backfat. These oxygenated compounds arise principally from the thermal oxidative decomposition of unsaturated fatty acids and lipids (Forss, 1972). The relative contribution of the aliphatic and aromatic hydrocarbons to the headspace of these sausages was less than that of the aldehydes, alcohols, and ketones. These compounds are also likely

Table 3. Comparison of Headspace Volatile Components (Dynamic Headspace) from Frankfurters with or without Spices or Smoke and from Solutions of Spices and Smoke

no.	compound ^a	LRI values ^b		indication of relative abundance ^c				ID method ^e	additional information ^f
		CPWax 52CB	CPSil 8CB	frankfurters HPMSD ^d	frankfurters without spices or smoke Kratos ^d	aqueous solution of spices HPMSD ^d	aqueous solution of smoke HPMSD ^d		
1	pentanal	982		--				MS + LRI	
2	decane	1000	1000	----	----		--	MS + LRI	
3	α -pinene	1014	933	--		--		MS + LRI	
4	α -thujene	1018	926	--		--		MS + lri	CBWax, 1003 (ten Noever de Brauw et al., 1980)
5	methylbenzene	1031	760	--		--		MS + LRI	
6	camphene	1047		--				MS + LRI	
7	2,3-pentanedione	1063		--				MS + LRI	
8	dimethyl disulfide	1065		tr				MS + lri	CBWax, 1077 (Kennet et al., 1982)
9	hexanal	1083	797	----	-----	--	--	MS + LRI	
10	undecane	1100	1100	--	----	--	--	MS + LRI	
11	β -pinene	1102	975	--		--		MS + LRI	
12	sabinene	1110	973	-----		--		MS + LRI	
13	ethylbenzene	1115	855	--	--			MS + LRI	
14	1,4-dimethylbenzene	1123	865	--	--			MS + LRI	
15	1,3-dimethylbenzene	1129	862	--	----			MS + LRI	
16	1-butanol	1130		--	--			MS + LRIe	LRIe (CPWax 52CB) = 1135
17	3-heptanone	1134		--	--			MS + LRIe	LRIe (CPWax 52CB) = 1140
18	3-carene	1135	1010	----		--		MS + LRI	
19	α -phellandrene	1155	1003	-----		--		MS + LRI	
20	β -myrcene	1157	991	-----		-----		MS + LRI	
21	α -terpinene	1168	1016	--		-----		MS + LRI	
22	1,2-dimethylbenzene	1175	890	--	--			MS + LRI	
23	2-heptanone	1180	887	--	----			MS + LRI	
24	heptanal	1181	898	--	-----			MS + LRI	
25	limonene	1192	1031	-----	-----			MS + LRI	
26	1,8-cineole	1196		----		----		MS + LRI	
27	dodecane	1200	1200	--	--			MS + LRI	
28	β -phellandrene	1205		-----		-----		MS + lri	CBWax 20M, 1208 (Nykanen, 1986)
29	2-methylpyridine	1206	811	--				MS + LRI	
30	3-methyl-1-butanol	1215		--	--			MS + LRI	
31	1-ethyl-4-methylbenzene	1216		--	--		tr	MS	
32	2-hexenal	1216		--	----			MS + LRI	
33	2-pentylfuran	1224		--	--			MS + LRI	
34	<i>cis</i> - β -ocimene	1229	1037	--	--	--		MS + LRI	
35	γ -terpinene	1237	1059	-----		-----		MS + LRI	
36	<i>trans</i> - β -ocimene	1239	1048	--	--	----		MS + LRI	
37	styrene	1244		--	--			MS + LRI	
38	1-pentanol	1245	758	--	----			MS + LRI	
39	<i>p</i> -cymene	1261	1024	-----	--	-----		MS + LRI	
40	α -terpinolene isomer	1267		--	--	----		MS	
41	methylpyrazine	1267		--	--			MS + LRI	CPWax 57CB, 1272
42	α -terpinolene	1273	1088	-----		-----		MS + LRI	
43	cyclohexanone	1280		--	--			MS + LRI	
44	octanal	1280		--	-----		--	MS + LRI	
45	3-hydroxy-2-butanone	1282	707	--	--			MS + LRI	
46	tridecane	1300	1300	--	--			MS + LRI	
47	2-heptenal	1320	952	--	----			MS + LRI	
48	2,3-octanedione	1322	981	--	--			MS + LRIe	LRIe (CPWax 52CB) = 1325
49	1,2,3-trimethylbenzene	1324		--	--			MS + LRI	
50	6-methyl-5-hepten-2-one	1328	984	--	--	--		MS + LRI	
51	bromobenzene*	1330	926	--	--			MS + LRI	
52	2-cyclopenten-1-one	1344		--	--		--	MS + LRI	
53	1-ethyl-3,5-dimethylbenzene	1347		--	--			MS	
54	1-hexanol	1351		--	--			MS + LRI	
55	2-methyl-2-cyclopenten-1-one	1355	901	--	--		--	MS + LRI	
56	dimethyl trisulfide	1359	963	tr	tr			MS + LRI	
57	nonanal	1383	1102	--	----	--	--	MS + LRI	
58	2-butoxyethanol	1395	904	--	--			MS + LRI	
59	tetradecane	1400	1400	--	--			MS + LRI	
60	3-ethyl-2-methyl-1,3-hexadiene or 5-ethylcyclopenten-1-carbaldehyde	1404		--	--			MS	Werkhoff et al. (1993) for 5-ethyl-cyclopenten-1-carbaldehyde
61	2-octenal	1421		--	--			MS + LRI	
62	a methyl(1-methylethenylbenzene)	1423		--	--	--		MS	
63	a dimethyl-2-cyclopenten-1-one or a methylcyclopenten-1-carbaldehyde	1432	940	--	--		--	MS	67 (100), 110 (66), 95 (32), 55 (23), 81 (22), 67 (21), 53 (20), 99 (15)
64	methional	1443		tr	tr			MS + LRI	

Table 3 (Continued)

no.	compound ^a	LRI values ^b		indication of relative abundance ^c				ID method ^e	additional information ^f
		CPWax 52CB	CPSil 8CB	frankfurters HPMSD ^d	frankfurters without spices or smoke Kratos ^d	aqueous solution of spices HPMSD ^d	aqueous solution of smoke HPMSD ^d		
65	1-octen-3-ol	1447		tr	----			MS + LRI	
66	acetic acid	1450		--		--		MS + LRI	
67	1-heptanol	1450			--			MS + LRI	
68	2-furfural	1460	826	--			----	MS + LRI	
69	α -copaene	1479	1382	--		--		MS + lri	DBWax, 1488; DB1, 1373 (Binder and Flath, 1989)
70	3-methyl-2-furfural	1488		--			--	MS + lri	CBWax20M, 1475 (Baltes and Bochman, 1984)
71	2-ethyl-1-hexanol	1490			--			MS + LRI	
72	pentadecane	1500	1500		--			MS + LRI	
73	benzaldehyde	1508	955	--			--	MS + LRI	
74	2,3-dimethyl-2-cyclopenten-1-one	1521	1034	--			--	MS + LRI	
75	2-nonenal	1540			--			MS + LRI	
76	linalool	1546	1098	-----		-----		MS + LRI	
77	1-octanol	1549		--	--			MS + LRI	
78	5-methylfurfural	1563	958	--			--	MS + LRI	
79	isobornyl acetate	1565		--				MS + lri	CBWax20M, 1577 (Nykanen, 1986)
80	β -caryophyllene	1582	1428	-----		-----		MS + LRI	
81	terpinen-4-ol	1588	1176	-----		-----		MS + LRI	
82	hexadecane	1600			--			MS + LRI	
83	2-decenal	1631			--			MS + LRI	
84	α -caryophyllene	1640		--		--		MS + LRI	
85	α -terpineol	1689	1098	--	--			MS + LRI	
86	heptadecane	1700	1700	--	--			MS + LRI	
87	α -zingibirene	1702	1497	--			-----	MS + lri	Supelcowax, 1728; SE-30, 1486 (Ramaswami et al., 1986)
88	β -bizabolene	1708	1512	--			-----	MS + lri	Supelcowax, 1735; SE-30, 1501 (Ramaswami et al., 1986)
89	l-carvone	1714	1240	--				MS + LRI	
90	δ -cadinene	1731		--		--		MS + lri	DBWax, 1742; DB1, 1495 (Binder and Flath, 1989)
91	α -farnesene	1737	1436	--			-----	MS + lri	Supelcowax, 1722 (Ramaswami et al., 1986)
92	ar-curcumene	1752	1485	--			-----	MS + lri	Supelcowax, 1777 (Ramaswami et al., 1986)
93	guaiacol	1849	1086	--			-----	MS + LRI	
94	safrole	1853	1288	--			-----	MS + LRI	
95	butylated hydroxytoluene	1898	1515	--	----			MS + LRI	
96	2-methoxy-4-methylphenol	1945	1187	--			-----	MS + LRI	
97	2-methylphenol	2000	1054	--			--	MS + LRI	
98	phenol	2000	977	--			--	MS + LRI	
99	cis-methylisoeugenol	2008		--			-----	MS + LRI	
100	4-ethylguaiacol	2020	1275	--			-----	MS + LRI	
101	2-ethylphenol	2069		--			--	MS + LRI	
102	2,5-dimethylphenol	2074		tr			--	MS + LRI	
103	2,4-dimethylphenol	2078		tr			--	MS + LRI	
104	a methylphenol	2085		tr			--	MS	
105	2-methoxy-4-propylphenol	2103		tr			----	MS + LRI	
106	eugenol	2155	1357	--			-----	MS + LRI	
107	elemicin	2219	1553	--			-----	MS + lri	DB1, 1530 (Shiota, 1993)
108	myristicin	2247	1522	--			-----	MS + lri	DB5, 1526 (ten Noever de Brauw et al., 1980)

^a Compounds are presented in the order of elution on a CPWax52 CB capillary column. ^b LRI values: linear retention indices (LRI) are given for two different polarity capillary columns, when applicable. Most of the analyses have been conducted on the polar CPWax52CB column, whereas a smaller number of analyses of frankfurters with liquid smoke have been performed on the nonpolar CPSil8CB capillary column. ^c Headspace volatiles were collected by dynamic headspace concentration. As different mass spectrometers were used for the analyses, absolute ion counts are not comparable between types of frankfurters. However, the relative abundances of different compounds in the different frankfurters are comparable. Relative sizes of peaks in GC chromatograms (full-fat frankfurters) are indicated by the following: -----, very large (peak height $>5 \times 10^7$ ions); -----, large (peak height between 2.5×10^7 and 5×10^7 ions); ----, medium (peak height between 1.25×10^7 and 2.5×10^7 ions); --, small (peak height $<1.25 \times 10^7$ ions); tr, trace; no mark, compound not detected. ^d Instrument of analysis: These analyses have been conducted on different MS instruments; a Kratos MS25RFA and a HP5970 mass selective detector. ^e Method of identification: MS + LRI, mass spectrum and LRI agree with those of authentic compound; MS + LRIe, mass spectrum agrees with literature spectrum, and LRI has been estimated from authentic standard of the same homologous series; MS + lri, mass spectrum agrees with literature spectrum and LRI agrees with literature data; MS, mass spectrum agrees with literature spectrum. ^f Additional information: Mass spectral data are given where not previously reported in the literature. The eight most abundant ions are cited in order of decreasing relative intensity. The molecular ion is underscored. For the other mass spectra, the literature reference is given by the *Wiley Registry of Mass Spectral Data* (Palisade Corp., Ivybridge, Devon, U.K.) unless otherwise stated. When an authentic compound was not available, LRI data have been compared with literature data. An indication of the LRI values obtained by these references is given.

Table 4. Key Odor Impact Compounds Detected in Frankfurters

LRI values ^a		odors ^b	detection of the odor ^c				possible compounds	ID method ^d	possible origin ^e
CPWax 52CB	CPSil 8CB		frankfurters	frankfurters without spices/smoke	frankfurters Herta	frankfurters Konecke			
943	-	caramel, fudge, vanilla	nd	nd	***	*	unknown		
982	618	fudge, buttery	***	**	***	***	2,3-butanedione	MS + LRI _{1,2} + odor	meat/smoke
1026	928	spices, green, pine needles	**	nd	*	**	α-pinene	MS + LRI _{1,2} + odor	spices
1083	-	green	nd	**	nd	nd	hexanal	MS + LRI ₁ + odor	meat
1114		stale, rubbery, green	*	nd	nd	*	unknown		
1164		biscuity, sweet, meaty; potato tubers (Konecke); fresh, sweet (Herta)	*	*	*	*	unknown		
1195	-	metallic, plastic	nd	**	nd	nd	an unsaturated alcohol	MS	meat
1205	1032	medicinal, cough syrup	**	nd	*	**	1,8-cineole	MS + LRI _{1,2} + odor	spices
1241	647	green, meaty, rubbery, not pleasant	*	nd	nd	*	unknown		
1294		potato, soil, musty	*	nd	nd	nd	unknown		
1302	968	mushrooms, green	*	***	***	***	1-octen-3-one	LRI _{1,2} + odor	meat
1314	863	meaty, roasty, biscuity	***	nd	**	**	2-methyl-3-furanthiol	LRI _{1,2} + odor	meat
1330	910	roasted meat, grilled fat, popcorn	***	*	**	**	2-methyl-3-methylthiofuran	<i>LRI₁ + odor</i>	meat
1376	964	old cooking smell, gassy, stale	***	*	***	***	dimethyl trisulfide	LRI _{1,2} + odor	meat
1396	-	mushroom	nd	**	nd	nd	1-nonen-3-one	LRI ₁ + odor	meat
1430		roasted, coffee beans, burnt potatoes; old, musty (Herta)	*	***	***	***	2-furanmethanethiol	LRI ₁ + odor	meat
1453	898	potatoes, biscuity, roasted meat; meaty, fatty (without spices)	***	*	***	***	methional + unknown	MS + LRI _{1,2} + odor	meat
1506	-	raw potatoes; stale, metallic	***	**	**	***	2-acetylfuran + unknown	MS + LRI ₁ + odor	meat
1525	-	burnt, rubber	nd	*	nd	nd	unknown		
1541	1097	flowers, carnation	**	nd	***	***	linalool	MS + LRI _{1,2} + odor	spices
1565		roasty, biscuity (only in 5% fat)	*	nd	nd	nd	unknown		
1587	-	meaty not pleasant, metallic	nd	nd	***	***	unknown		
1603		rubber, inflatable boats (only in 5% fat frankfurter)	*	nd	nd	nd	unknown		
1615	-	burnt, meaty	nd	*	nd	nd	unknown		
1627		vomit, sickly	*	nd	*	*	unknown		
1663		potatoes, warm, meaty (only in 5% fat sample), meaty sweet (frankfurters without spices)	*	**	nd	nd	unknown		
1666	1100	meaty, roasty, biscuity	***	nd	**	***	2-methyl-3-methyl-dithiofuran	LRI ₂ + odor	meat
1688	-	meaty, fatty	nd	*	nd	nd	2-furfuryl methyl disulfide	LRI ₁ + odor	meat
1733	1069	medicinal, plastic	***	nd	***	***	unknown		
1754	1101	roasted meat, biscuity, popcorn	***	nd	***	***	2-acetylthiazoline	MS + LRI _{1,2} + odor	meat
1800	-	unpleasant, stale, meaty, oily	nd	**	nd	nd	2,4-decadienal	MS + LRI ₁ + odor	meat
1833	-	brown sugar, sweet	nd	nd	***	***	2-hydroxy-3-methyl-2-cyclopenten-1-one (cycloten)	MS + LRI ₁ + odor	smoke
1856	1087	smoky, frankfurter, plastic	***	nd	***	***	2-methoxyphenol	MS + LRI _{1,2} + odor	smoke
1938	1185	smoky	***	nd	***	***	2-methoxy-4-methylphenol	MS + LRI _{1,2} + odor	smoke
1969	1392	roasty, meaty	*	nd	nd	***	2-methyl-3-methyl-trithiofuran	<i>LRI₂ + odor</i>	meat
1969	1080	smoky, frankfurters	nd	nd	nd	***	2-methylphenol	MS + LRI _{1,2} + odor	smoke
2084	1357	stale, gassy, medicinal, phenolic	***	nd	**	***	2-methoxy-4-propylphenol	MS + LRI _{1,2} + odor	smoke
2156		sausage meat, spicy, stock cube	*	nd	*	nd	unknown		
2222	1651	mushrooms	*	nd	nd	nd	unknown		
2264	1349	smoky, frankfurter	**	nd	***	***	2,6-dimethoxyphenol	MS + LRI _{1,2} + odor	smoke

^a LRI values: Linear retention indices (LRI) are given on two different polarity capillary columns, when applicable. Values in italics correspond to odors whose correspondence with the odor detected on the CPWax 52CB column is uncertain. A blank means that it has not been possible to assign the LRI value of the odor on the CPSil 8CB capillary column; a hyphen means that the odor was not detected at all by GC/odor assessment conducted on this column. ^b Odors are presented in order of elution on the CPWax 52CB column. ^c Detection of the odors: ***, odor detected in at least three-fourths of the assessments; **, odor detected at least half of the assessments; *, odor detected in less than half of the assessments; nd, odor not detected. Volatiles from frankfurters without spices and smoke were assessed by four assessors in duplicate. Volatiles from the frankfurters with spices and smoke and from the commercial frankfurters Herta and Konecke were evaluated by four assessors at least once. ^d Method of identification: MS + LRI + odor, mass spectrum, LRI, and description of odor agree with those of authentic compound; LRI + odor, compound not identified by GC/MS, but whose odor and LRI correspond to the odor found by GC/odor assessment; MS + LRI, compound identified by GC/MS at similar LRI as the odor; *LRI, odor (italics)*, LRI and/or description of odor agree with literature data; LRI₁ corresponds to identification on a CPWax 52CB capillary column; LRI₂ corresponds to identification on a CPSil 8CB capillary column. ^e Possible origin: meat, compound derived from the basic meat fraction; spices, compound derived from spices incorporated in frankfurters; smoke, compound derived from smoke incorporated in frankfurters.

to be derived from the thermal oxidation of lipids (Min et al., 1977).

Few of the lipid oxidation products listed in Table 3 contributed to the overall aroma of frankfurters, but some aldehydes, ketones, and alcohols, not necessarily detectable by GC/MS, are probably responsible for certain of the individual odors presented in Table 4. Hexanal may be responsible for the "green" odor at LRI 1083, 2,4-decadienal for the "unpleasant, stale, meaty, oily" odor at LRI 1800, 1-octen-3-one and 1-nonen-3-one for the "mushroom" odors at LRI 1302 and 1396, and an unsaturated alcohol (not identified) for the "metallic, plastic" odor at LRI 1195. Among these compounds, hexanal, 1-octen-3-one, and 2,4-decadienal have been reported as potent odorants in roasted beef (Cerny and Grosch, 1992).

Some butylated hydroxytoluene (BHT; 2,6-di-*tert*-butyl-4-methylphenol) was detected in the headspace of these products. This compound possibly originates from the fat fraction, to which it is generally added for its antioxidant effect, or from the pork fraction, through animal feed (Berdague et al., 1991).

Compared with the frankfurters prepared without spices or smoke, most of the above compounds either were present in reduced amounts or were not detectable in the frankfurters with spices and smoke. It is possible that these lipid oxidation products were suppressed by an antioxidative effect of added ingredients to these frankfurters. The ingredients responsible may include the nitrites, ascorbic acid, or smoke or spice components. Shiratsuchi et al. (1993) also found that lipid oxidation products were hardly detected in nonfermented coarse-cut sausages prepared with spices and liquid smoke. Other workers showed a decrease of lipid oxidation products in cured meats compared with noncured meats (Ramarathnam et al., 1991; Cross and Ziegler, 1965); this was attributed to the antioxidant effect of nitrite, used during the curing process. Ascorbic acid is well-known for its antioxidative properties (Bauernfiend and Pinkert, 1970), whereas the antioxidant activity of spices or smoke is generally considered to be due to the presence of high-boiling phenols and related substances (Daun, 1979; Sethi and Aggarwal, 1957). The headspace of the solution of smoke used in the present frankfurters was characterized by phenolic compounds, whereas that of the solution of spices contained related compounds, such as eugenol and isoeugenol. Such compounds have been proven to act as antioxidants (Sethi and Aggarwal, 1957) and could, therefore, contribute to the reduction in quantities of aldehydes, alcohols, and ketones in these frankfurters. In the present study, although nitrite salt was added to both types of frankfurters, sodium ascorbate, spices, and smoke were included only in the frankfurters with spices and smoke. The observed antioxidative effect is, therefore, probably due to the combined effect of ascorbic acid and phenolic compounds derived from the spices or smoke.

Other compounds derived from the meat fraction of frankfurters included Maillard formation products. Two straight-chain sulfur-containing compounds, dimethyl trisulfide, responsible for the "old cooking smell, metallic, gassy, stale" odor at LRI 1376, and methional, responsible for the "potato" odor at LRI 1453, were detected by GC/MS in trace quantities (Table 3). Other Maillard products detected in frankfurters included furan thiols and sulfides; these compounds were not detected by GC/MS (Table 3) but seemed to play a

significant role for the overall aroma of frankfurters, as determined by GC/OA (Table 4). The odor description and LRI value of authentic 2-furanmethanethiol fitted well with the "roasted, coffee beans" odor at LRI 1430, whereas 2-methyl-3-methylthiofuran is likely to be responsible for the "roasted meat, grilled fat, popcorn" odor at LRI 1330, which was detected at a similar LRI value by other workers with a "meaty, sulfurous" odor (Gasser and Grosch, 1988). Some of these odor compounds, such as 2-furanmethanethiol and 2-methyl-3-methylthiofuran, were detected in all types of frankfurters. However, other "meaty" odor compounds that might have been expected in all of the frankfurters were detected only in one type of frankfurter (Table 4). For example, the odor at LRI 1688 ("meaty, fatty") was detected only in the frankfurters without spices and smoke. This odor is likely to be due to 2-furfurylmethyl disulfide, because GC/OA of the authentic compound gave an odor at LRI 1690 with a "meaty, roasty, biscuity" odor. Other meaty odors were detected only in the fully formulated frankfurters; for instance, odors were detected at LRI 1314 ("meaty, roasty, biscuity"), at LRI 1666 ("meaty, roasty, biscuity"), at LRI 1754 ("roasted meat, biscuity, popcorn"), and at LRI 1969 ("roasty, meaty"), which are likely to be caused by 2-methyl-3-furanthiol, 2-methyl-3-methyldithiofuran, 2-acetylthiazoline, and 2-methyl-3-methyltrithiofuran, respectively. The first three of these compounds eluted at a similar LRI values and with the same odor character as the authentic standards. 2-Methyl-3-methyltrithiofuran has been found by other workers (Mottram and Madruga, 1994) at an LRI similar to that detected on the CPSil 8CB capillary column and with an odor description similar to those obtained in this study. It was surprising that these sulfur-substituted furans were not detected in the frankfurters without spices and smoke. However, this may be due to differences in experimental procedure; the frankfurters without spices and smoke were cooked in a water bath, whereas those with spices and smoke were cooked in an oven, which may provide higher temperature at the surface of the sausage and lower moisture levels, both factors favoring the Maillard reaction (van den Ouweland et al., 1978). It is also possible that the labile sulfur compounds react on the steel needle in the syringe used to sample the headspace volatiles from frankfurter without spices and smoke. For the other types of frankfurters, the volatiles were withdrawn directly from the headspace to the Tenax trap, thus avoiding contact with the metal surface.

Volatile Compounds Derived from the Spices. The early part of the chromatogram obtained for frankfurters with spices and smoke (LRI = 1000–~1280 on CPWax 52CB column) was characterized by the abundance of monoterpene hydrocarbons and alcohols. Sesquiterpene hydrocarbons, of high molecular weight, eluted mostly in the early second part of the chromatogram (LRI = 1450–1764). The identities of most of the sesquiterpene hydrocarbons remain quite uncertain due to the unavailability of authentic standards. Identification for these compounds relies mainly on mass spectral information from the *Wiley Registry of Mass Spectral Data* (Palisade Corp., Ivybridge, Devon, U.K.) and on LRI values obtained from the literature (Ramaswami et al., 1986). The last part of the chromatogram (LRI = 1656–2340 on CPWax 52CB column) was dominated by

phenyl propanoids, such as estragole, *trans*-anethole, safrole, *cis*-methylisoeugenol, eugenol, elemicin, and myristicin.

The terpenes and phenyl propanoids were also major components in the headspace from the solution of spices (Table 3) and, thus, are clearly derived from this source. These compounds have been widely reported in individual spices and condiments, such as pepper, mint, ginger, nutmeg, and cardamon, which were the major spices incorporated in these frankfurters. For example, the major volatile components in black pepper (Lawrence, 1981) comprised mostly monoterpene hydrocarbons, such as sabinene (19% of the total volatile composition), limonene (17%), and β -pinene (10%), and also a large proportion (14%) of β -caryophyllene (sesquiterpene hydrocarbon) and 4% of 1,8-cineole (terpene alcohol). Nutmeg oil has also been characterized by a large proportion of monoterpene hydrocarbons (e.g., sabinene, α -pinene, and β -pinene) but may also account for the presence in frankfurters of phenyl propanoids (e.g., myristicin and safrole). The relatively large quantity of 1,8-cineole and α -terpinyl acetate in frankfurters is likely to be due to the presence of cardamon, in which these components comprise from 26 to 45% and from 29 to 34% of the total cardamon oil composition, respectively (Pieribattesti et al., 1988). The sesquiterpenes α -zingiberene and *ar*-curcumene probably originated from ginger, from which they have been extracted in large quantities (approximately 38 and 17% of the total ginger oil composition, respectively; Vernin and Parkanyi, 1994). Some of these compounds have also been identified in other meat products containing spices, such as nonfermented sausages (Shiratsuchi et al., 1993) and dry salami (Berger et al., 1990).

Three of the above compounds, α -pinene, 1,8-cineole, and linalool, contributed to the overall aroma of frankfurters. These compounds, readily identified by GC/MS and having odor descriptions matching those of the authentic standards, were responsible for the individual odors "spices, green, pine needles" at LRI 1026, "medicinal, cough syrup" at LRI 1205, and "flowers, carnation" at LRI 1541, respectively (Table 4).

Volatile Compounds Derived from the Liquid Smoke. The major contribution from the liquid smoke to the headspace of frankfurters was the addition of phenols, cyclopentenones, and furans (Table 3). These compounds have been widely reported in smoke or smoked meat products (Fiddler et al., 1966; Bratzler et al., 1969; Knowles et al., 1975; Guillen et al., 1995). Phenol and methoxyphenol derivatives originate mainly from the pyrolysis of lignin (Kim et al., 1974), which accounts for ~25% of the composition of wood (Goos, 1952). Cyclopentanones may arise from corresponding dicarboxylic acids by decarboxylation and ring formation (Toth and Potthast, 1984). The formation of furans most probably results from the degradation of glucose, which is an intermediate product of the thermal degradation of cellulose. Other furans, such as 2-furfural, can be formed from pentoses, which are degradation products of hemicellulose (Toth and Potthast, 1984).

Several smoky odors (LRI 1856, 1938, 2084, and 2264) were detected by GC/OA of frankfurters (Table 4). Comparison with the dynamic headspace analyses and an assessment of the odor and LRI of authentic compounds indicated that these odors were caused by 2-methoxyphenol (guaiacol), 2-methoxy-4-methylphenol (4-methylguaiacol), 2-methoxy-4-propylphenol (4-propyl-

ylguaiacol), and 2,6-dimethoxyphenol (syringol), respectively. Previous studies have shown that phenols appear to be the principal contributors to smoke flavor and aroma (Bratzler et al., 1969; Fiddler et al., 1970). Among the phenols found in these frankfurters, guaiacol, phenol, and 4-methylguaiacol gave the largest peaks. It is interesting to note that these compounds were also among the major components found in wood smoke vapor (Kornreich and Issenberg, 1972). They were also found as the major constituents in the phenolic fraction of cooked bacon (Knowles et al., 1975; Ho et al., 1983; Shu and Mookherjee, 1985).

A number of fatty acids have also been reported in smoke extracts or smoked meat products (Fiddler et al., 1966; Ho et al., 1983; Shu and Mookherjee, 1985). Despite the fact that they do not contribute to the smoky flavor (Fiddler et al., 1970), the acids may be important in the smoking process for other reasons such as the denaturation of the meat protein to form a "skin", increasing the peelability of the casing of sausage products, aiding surface color development, or serving as a solvent for smoke components (Fiddler et al., 1970). Acetic acid (LRI 1450) was the only acid detected in the headspace of the present frankfurters. Other acids, such as tetradecanoic acid, hexadecanoic acid, and octadecanoic acid were collected from these frankfurters using a Likens-Nickerson extraction method (Chevance, 1998). However, separate analyses of the solutions of spices and smoke indicated that these acids originated from the spice solution rather than the liquid smoke (Chevance, 1998).

Comparison of Odors with Commercial Frankfurters. In general, very similar odors were detected in the frankfurters with spices and smoke and the commercial brands, with a few exceptions (Table 4). Three odors appeared only in the commercial products; these odors included "caramel, fudge, vanilla" at LRI 943, "meaty, unpleasant, metallic" at LRI 1587, and "brown sugar, sweet" at LRI 1833. Of these odors, the third one could be due to the presence of 2-hydroxy-3-methyl-2-cyclopenten-1-one (cycloten) in the commercial products. GC/OA of the authentic compound gave a description of "brown sugar" at an LRI of 1830, which agrees with the description and LRI of the odor detected in the frankfurters. Cycloten has been reported as one of the key odor compounds in smoke (Guillen et al., 1995). It was not detected by GC/MS in the experimental frankfurters prepared with spices and liquid smoke but was easily detected in frankfurters that were traditionally smoked (Chevance, 1998) and in the commercial frankfurters. The absence of this odor in these experimental frankfurters (Table 4) is possibly due to the absence or very low quantities of cycloten in the liquid smoke used for these sausages. One odor, "mushrooms", at LRI 2222, occurred only in the experimental frankfurters. The compound responsible for this odor has not been identified.

With the exception of the above, the odor profile of all three types of frankfurters is dominated by a similar range of compounds; a range of sulfur-containing Maillard products contribute to "meaty", "roasty", "biscuity", "potato", and "gassy" odors, whereas lipid oxidation products provide "mushroom", "stale", "oily", and "green" notes. Linalool, 1,8-cineole, and α -pinene provide "flowery", "medicinal", and "spicy" odors, while a range of phenols from the smoke are responsible for the characteristic "smoky" aroma and flavor of frankfurters.

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