Additional Studies on Flavor Components of Corn Tortilla Chips

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Volatile components from tortilla chips were isolated by high flow dynamic headspace sampling using a closed loop system with excess anhydrous sodium sulfate and trapping the volatiles on Tenax. Capillary GC-MS was used to identify the components. Major volatiles identified included 2-methylpyrazine, 1-hydroxy-2-propanone, 4-vinylguaiacol, 2-acetyltetrahydropyridine, (*E*,*E*)- and (*E*,*Z*)-2,4-decadienal, acetic acid, 3-methylbutanal, γ -butyrolactone, furfuryl alcohol, and 2,5-dimethylpyrazine. Concentration/threshold ratios indicated that the components that have the highest probability to contribute to the aroma and flavor include (*E*,*E*)- and (*E*,*Z*)-2,4-decadienal, 3-methylbutanal, 2-furfurylthiol, 2-acetyl-1-pyrroline, 2-acetyltetrahydropyridine, (*E*,*E*)-2,4-decadienal, (*E*,*E*)-2,4-decadienal, 3-methylbutanal, 2-furfurylthiol, 2-acetyl-1-pyrroline, 2-acetyltetrahydropyridine, (*E*,*E*)-2,4-decadienal, (*E*,*E*)-2,4-decadienal, 3-methylbutanal, 2-furfurylthiol, 2-acetyl-1-pyrroline, 2-acetyltetrahydropyridine, (*E*,*E*)-2,4-decadienal, (*E*,*E*)-2,4-decadienal, 3-methylbutanal, 2-furfurylthiol, 2-acetyl-1-pyrroline, 2-acetyltetrahydropyridine, 2-acetyltetrahydropyridine, 2-acetyltetrahydropyridine, (*E*)-2-decenal, methional, and 2-ethyl-3,5-dimethylpyrazine.

Keywords: Corn; tortilla chips; volatiles; flavor; identification; concentrations; odor thresholds

INTRODUCTION

Corn tortilla chips form a substantial part of the market for corn products. Knowledge of the important aroma and flavor components involved can be useful to the industry in quality control. The authors had previously carried out a preliminary study of the flavor components of corn tortilla chips as part of a general study of tortilla-related products (Buttery and Ling, 1995). The present study extended the earlier investigation and concentrated solely on tortilla chips.

EXPERIMENTAL PROCEDURES

Materials. The tortilla chips were major brands purchased from local supermarkets. Three different brands were examined. These were all of the deep fried type and were of the "Mexican" or "restaurant" style used well before their expiration dates. They contained 25-30% vegetable oil (corn, canola, soybean, or sunflower oil). These were stored at room temperature in the dark and used within a few days. Anhydrous sodium sulfate and sodium dihydrogen phosphate (both >99% pure) were heated at 150 °C for several hours to remove possible trace volatiles. Diethyl ether was freshly distilled, stored in the dark, and contained ca. 1-2 ppm Ethyl Corp. antioxidant 330. Authentic compounds were obtained from reliable commercial sources or synthesized by established methods.

Isolation of Volatiles Using Excess Na₂SO₄. The general method used for the major part of the work had been previously described (Buttery and Ling, 1997a). Tortilla chips (30 g) were placed in a Pyrex blending jar and blended to a coarse powder. Water (30 mL) was then added, and the mixture was blended again. Sodium sulfate(100 g) was next added and the mixture blended. For approximate neutral conditions (pH ca. 7.4), NaHCO₃ (2.0 g) was added at this point. For slightly acid conditions (pH ca. 4.5), NaH₂PO₄ (2.0 g) was added instead. The mixture was then transferred to a 1 L beaker containing an additional 140 g of sodium sulfate and mixed thoroughly with a glass rod. This mixture was then placed in a Pyrex glass column 36 mm o.d. \times 350 mm long containing a coarse fritted disk at the lower end and ground ball-and-socket joints for connection to the closed loop system. The lower end of the column was connected to a large Tenax trap (ca. 10 g of Tenax). The column and trap were connected to an all Teflon diaphragm pump that recirculated nitrogen around the loop at ca. 3-6 L/min for 3 h. It was necessary throughout to keep the sodium sulfate mixture at less than ca. 25 °C because Na₂SO₄·10H₂O decomposes at 32 °C. After the isolation, the Tenax trap was removed from the loop and extracted with freshly distilled ether (50–100 mL). The ether extract was concentrated to ca. 50 μ L using a warm (50–60 °C) water bath and micro Vigreux distillation column.

Isolation of Volatiles Using an Aqueous System. Tortilla chips (30 g) were added to 150 mL of volatile free distilled water, 54 g of sodium chloride, and 7.5 g of sodium bicarbonate in a Pyrex Waring blender and blended for 30 s. The mixture was poured into a 1 L flask, a suitable Pyrex head was attached that allowed entry of sweep gas over the solution and exit through a large Tenax trap. The flask, head, and trap were connected in a closed loop system similar to that used for the sodium sulfate method. Nitrogen was pumped around the loop for 3 h. Again, extraction of the trap and concentration as above gave the concentrate for analysis.

Basic and Nonbasic Fractions. The ether extract (ca. 50 mL), from extraction of the Tenax trap using the sodium sulfate method above, was placed in a separatory funnel and extracted with 3 N HCl (3 \times 25 mL). The upper ether layer, containing the nonbasic components, was washed with NaClsaturated water (25 mL), dried over sodium sulfate, and concentrated to give the nonbasic fraction. The combined HCl extract was washed with ether (50 mL) and then placed in a wide-neck Erlenmeyer flask and covered with ether (75 mL). The flask was cooled externally with an ice bath, and excess sodium carbonate was added to neutralize the acid. The mixture was then transferred to a separatory funnel and shaken with the ether layer. The aqueous layer was extracted with more ether (2 \times 50 mL); the combined ether extracts were dried over sodium sulfate and concentrated to ca. 50 μ L to give the basic fraction.

Quantitative Analysis. For quantitative studies, 1 mL of a water solution of internal standards was added during the blending process. For the approximate neutral conditions with sodium bicarbonate, the internal standard solution consisted of $20 \,\mu$ L/L each of 2-pentanone, 3,5-dimethylpyridine, and quinoline. For the slightly acidic conditions with NaH₂-PO₄, the solution consisted of 20 μ L/L each of 2-pentanone, 6-methyl-5-hepten-2-one, and 4-phenyl-2-butanone.

Isolation of Volatiles from Dry Tortilla Chips. Tortilla chips (70 g) were ground up in a blender to a coarse powder

(ca. 40 mesh). This powder was packed into a Pyrex glass column similar to that described above for the sodium sulfate mixture. The lower end of the column was attached to a Tenax trap and connected in the closed loop system as above, and nitrogen was recirculated around the loop for 3 h at a flow rate of 3-6 L/min. Extraction of the trap and concentration as above gave the concentrate used for GC and GC-MS analyses.

Capillary GC–MS. A HP 5890 GC instrument was used coupled to a HP 5971 quadrupole mass spectrometer. The capillary columns were fused silica, 60 m long \times 0.25 mm i.d. wall coated with DB-WAX or DB-1. The DB-WAX column was held at 30 °C for the first 4 min after injection and then heated at 2 °C/min to 170 °C and held a further 30 min. The injector temperature was 170 °C and used a 1/20 split. The DB-1 column was held at 30 °C for the first 25 min and then heated at 4 °C/min until 200 °C and held a further 30 min.

Recovery of Volatiles Using the Na_2SO_4 **Method.** Absolute recoveries of compounds were determined by making standard solutions (usually 10.0 ppm) of pure samples in water and blending a measured amount (1.00 mL) with the sodium sulfate and taking it through the isolation process using the closed loop method described above. A measured amount of an internal standard (2-octanone) was added to the ether extract at the end of the isolation before concentration.

Odor Thresholds. These were determined as described previously (Buttery and Ling, 1997b) or were values determined in previous work by the authors for a variety of food products (cf. Buttery and Ling, 1995).

RESULTS AND DISCUSSION

Isolation Methods. Several different isolation methods were used, some offering advantages over others for specific components. Previously (Buttery and Ling, 1995) the authors had isolated the volatiles from tortilla chips blended with an excess of water saturated with NaCl. This method had the advantage that it was relatively easy to carry out, but such aqueous method gives very poor or no significant recovery of watersoluble compounds. In the present study, the primary method was one that used an excess of anhydrous sodium sulfate and had been previously described by the authors for other products (Buttery and Ling, 1997a). With this method, only a small amount of water was blended with the chips, then anhydrous sodium sulfate was used to bring the food back to a dry state to allow isolation of highly water-soluble volatiles together with the more easily isolated poorly water-soluble volatiles.

Two main pH conditions were used to isolate the volatiles. Sodium bicarbonate (dry) was added to the dry sodium sulfate mixture to give a pH of ca. 7.4, close to neutral conditions. Monosodium dihydrogen phosphate (NaH₂PO₄) was added to give slightly acidic conditions (pH ca. 4.5). In all cases, volatiles were swept to a large Tenax trap with nitrogen in a high flow closed loop dynamic headspace system. The volatile isolate concentrate was analyzed by GC–MS.

The isolate from a separate experiment was also divided into basic and nonbasic fractions, and GC-MS analyses were carried out on these. This had the advantage of giving simpler chromatograms with less peak overlap with other components.

Volatiles were also isolated from the ground (ca. 40 mesh) dry chips packed into a Pyrex column and sweeping the chips with nitrogen in the closed loop arrangement with a Tenax trap. The total volatile concentrate isolated with this dry method was considerably less than that obtained when water was added and consisted mainly of the aliphatic aldehydes and alkyl-

pyrazines, although these too were in lesser concentration than that obtained from wetted chips.

Volatiles Identified. Table 1 lists compounds identified that had not been previously reported by the authors (Buttery and Ling, 1995) or other workers. The mass spectra and GC retention data found were consistent with those of authentic samples. For a few compounds, authentic samples were not available, but the mass spectra matched that of published data; these compounds are listed in parentheses and considered only tentatively identified. Listed in Table 2 are GC retention indices for all compounds on DB-WAX, together with concentrations found using internal standards added to the chips at the beginning of the isolation. The quantitative data are the means from the separate isolation and analysis of three different samples (same brand) of tortilla chips. Although the data were measured as carefully as possible, they are only intended to give some idea of the order of magnitude of the actual concentrations. It is difficult to obtain very accurate quantitative analyses on such a large number of food volatiles. In addition, the concentrations of components varied considerably with different brands of tortilla chips. Also listed in Table 2 are the odor thresholds of the identified components in water solution and the log of the ratio concentrated/threshold.

2-Acetyl and (2-Propionyl) Nitrogen Heterocyclic Compounds. Members of this group give an important "cracker" or "popcorn" character to tortilla chips. Most of them are the same as those that have been identified in popcorn by Schieberle (1991). The 2-acetyltetrahydropyridine tautomers (1,4,5,6 and 3,4,5,6 forms) together occur at a concentration of 1100 μ g/kg, considerably greater than any of the other members of the group. The tautomers form a single broad peak on the DB-WAX capillary GC column but give sharp wellseparated peaks on the low polarity DB-1 capillary. The reason for this, as with other tautomers, is that polar solvents (in this case stationary phase) enhance the tautomerism. The interchange between the two forms apparently occurs to an appreciable extent during the chromatography on DB-WAX.

2-Acetylpyridine occurs in the next highest concentration of this group at 120 μ g/kg but is a relatively weak odorant. 2-Acetyl-1-pyrroline and 2-propionyl-1-pyrroline occur at less than 1/10th the concentration of the 2-acetyltetrahydropyridines, but their odors can be detected at ca. 20 times lower concentration (i.e., odor threshold), which compensates for their lower weight concentrations. The weakest odorant of this group is 2-acetylpyrrole, whose odor threshold is more than a million times higher than that of 2-acetyl-1-pyrroline, despite the chemical and structural similarity of these two compounds.

None of this group were found in the volatiles isolated from dry tortilla chips. They were only found in the chips that were moistened at some point in the volatile isolation, indicating that they are released in some way by the moisture, possibly by displacement of the volatiles from their adsorption sites by water, by breakdown of starch walls that have encapsulated volatiles, or by hydrolysis of some precursors. The likely origins of these acetyl nitrogen heterocyclic compounds have been studied by Schieberle (e.g., Hofmann and Schieberle, 1998).

2-Aminoacetophenone. This compound had been previously found by the authors to be important to the

Table 1. Mass Spectral Data Found for Additional Volatiles Identified in Corn Tortilla Chips

compound ^a	major MS ions ^b	compound ^a	major MS ions ^b							
Aliphatic Alcohols										
butanol	45, 31, 59, 29	2,3-butanediol	45, 57, 31, 75, 90							
1-hydroxy-2-propanone	43, 31, 74, 59	meso-2,3-butanediol	45, 57, 31, 75, <i>90</i>							
Aliphatic Aldehydes and Ketones										
(E)-2-hexenal	41, 55, 69, 83, <i>98</i>	(\vec{E}) -2-decenal	43, 55, 70, 83, 97, 110							
2-octanone	43, 58, 71, 128, 85, 113	(E,E)-2,4-nonadienal	81, 41, 67, 53, 95, 138							
octanal	43, 57, 84, 29, 69, 100	(E)-2-undecenal	41, 57, 70, 83, 97, 111							
(E,E)-2,4-heptadienal	81, 39, 29, 53, 67, <i>110</i>	"4,5-epoxy-2-nonenal"	68, 41, 55, 81, 95, 138							
1-octen-3-one	43, 55, 70, 83, 97	4,5-epoxy-(<i>E</i>)-2-decenal (isomer A)	68, 39, 55, 81, 95, 139							
(E)-2-octenal	41, 55, 70, 83, 97, <i>126</i>	4,5-epoxy-(E)-2-decenal (isomer B)	68, 39, 55, 81, 95, 139							
Aliphatic Acids										
acetic acid	43, 45, <i>60</i> , 29	3-methylbutyric acid	60, 43, 87, 73							
propionic acid	45, 74, 57, 60	pentanoic acid	60, 73, 41, 87							
isobutyric acid	43, 73, 55, <i>88</i> , 60	hexanoic acid	60, 73, 41, 87, 98							
butyric acid	60, 73, 41, <i>88</i>	heptanoic acid	60, 73, 41, 87, 101							
Aromatic and Furan Compounds										
2-pentylfuran	81, 53, <i>138</i> , 39, 95, 68	2-methyl-3-hydroxy-4-pyranone (maltol)	<i>126</i> , 71, 43, 55, 97, 111							
2-methoxyphenol (guaiacol)	109, <i>124</i> , 81, 53, 39, 65	2,5-dimethyl-4-hydroxy-3(2 <i>H</i>)-furanone (Furaneol)	43, <i>128</i> , 57, 29, 85, 72							
(Ful alicol)										
nyridine	79 52 51 50 39	"2-methyl-6-vinylnyrazine"	120 52 39 94 66 79							
2 6-dimethylpyrazine	<i>108 1</i> 2 67 81 93 52	"2-isopropenylpyrazine"	110 67 52 39 79 94							
2-ethyl-6-methylpyrazine	121 39 94 56 66 80	"2 5-dimethyl-3-vinylnyrazine"	133 42 54 66 81 106							
2-ethyl-3-methylpyrazine	121, 67, 39, 94, 81, 51	2-acetylpyridine	79, 43, 121, 52, 93, 106							
2.3.5-trimethylpyrazine	42. 122. 81. 54. 107. 67	"N-methyl-2-pyrrolidone"	<i>109</i> , 53, 39, 80							
"2-propionyl-1-pyrroline"	57. 41. 97. 69. 125. 83	2-acetylpyrrole	94, 109, 66, 39, 53, 80							
2-ethyl-3,5-dimethylpyrazine	135, 56, 39, 108, 67, 80	515	- ,							
Lactorias										
v-butvrolactone	41, 42, 56, 86, 85	v-octalactone	85, 41, 56, 100, 70, 114							
dimethyl disulfide	91 15 79 61 32	dimathyl trisulfida	126 15 79 61 111 32							
limonene	68 93 39 <i>136</i> 53 79	2-furfurvlthiol	81 53 <i>114</i> 45 69							
minomente	00, 00, 00, 100, 00, 10	~ mining minor	01, 00, 111, 10, 00							

^{*a*} Mass spectrum and GC Kovats retention index found are consistent with those of an authentic sample except for those listed in quotes where the mass spectrum was consistent with published spectra but no authentic sample was available. ^{*b*} One major ion each 14 mass units in order of their intensities with most intense ions first. Molecular ion if present is italicized.

character of many tortilla-related products (Buttery and Ling, 1994, 1995). Its concentration varied considerably with different commercial sources of tortilla chips. It is apparently formed during the commercial preparation of these products when the corn is heated briefly to boiling with water containing lime (Ca(OH)₂) and then allowed to steep in this aqueous alkaline mixture for 8-24 h. It has long been known by the tortilla processors that this alkaline treatment is important to produce the traditional desired flavor of tortilla products, although the mechanism was not suspected until suggested by the authors (Buttery and Ling, 1994).

In studies related to the stability of tryptophan, Tabone et al. (1951) had shown (Figure 1) that alkaline conditions catalyzed air oxidation of the amino acid tryptophan to the compound kynurenine, which they showed was degraded to 2-aminoacetophenone by alkali (cf. also Spacek, 1954). It is easy to demonstrate this in the laboratory by taking 1 g of L-tryptophan in 50 mL of water and 5 g of Ca(OH)₂ in an open 150 mL Erlenmeyer flask, bringing the mixture to a boil, and then allowing it to cool in the uncovered flask. Within 1 h, there is a strong odor of 2-aminoacetophenone. Isolation of the volatiles after 24 h gave 2.5 μ g of 2-aminoacetophenone.

Aliphatic Aldehydes. This is another group that gives an important character to the aroma and flavor of tortilla chips. In this case a "deep fried" character. These compounds are responsible for a similar character in many other fried products and most likely originate primarily from the cooking oils. (E,E)-2,4-Decadienal and (E,Z)-2,4-decadienal together were found at a high

concentration in the chips. These isomers are also very potent odorants, often described as having a fatty, deep fried character. The C₇ and C₉ dienals and the C₆-C₁₁ 2-enals also found are additional common components of fried foods.

Small concentrations of 4,5-epoxy-(E)-2-decenal (two isomers) and 4,5-epoxy-(E)-2-nonenal were identified. These compounds have been reported in a number of foods by Schieberle, Grosch, and co-workers (e.g., Gassenmeier and Schieberle, 1994; Kerler and Grosch, 1996).

The aliphatic aldehydes were the main group of compounds isolated from the dry tortilla chips where no water was used in the isolation. Greater concentrations were found though after addition of water.

Alkylpyrazines. The alkylpyrazines found are typical of those that occur in foods that are heated to relatively high temperatures such as in frying. The separation of a basic fraction from the total isolated volatiles facilitated the identification of the pyrazines. At 1830 μ g/kg, 2-methylpyrazine occurs at the highest concentration of this group but is an extremely weak odorant. More important to the aroma and flavor are 2-ethyl-3,5-dimethylpyrazine (odor threshold 0.04 μ g/ L), 2-ethyl-3,6-dimethylpyrazine (odor threshold 8.6 μ g/ L) and 2,3,5-trimethylpyrazine (odor threshold 23 μ g/ L). In their previous work (Buttery and Ling, 1995) had mistakenly attributed the odor of the ethyldimethylpyrazine peak to the major 2-ethyl-3,6-dimethylpyrazine isomer but later realized that the odor of this peak is mostly due to the isomeric and very much more potent odorant 2-ethyl-3,5-dimethylpyrazine. These isomers

Table 2. GC Data, Concentrations Found, Odor Thresholds in Water Solution, and Ratio of Concentration/Threshold (in Log₁₀ Form) for Volatile Compounds Identified in Corn Tortilla Chips

	KI ^a	concn ^b	threshold	log ₁₀ concn/		KI ^a	concn ^b	threshold	log ₁₀ concn/		
compound	DB-WAX	(µg/kg)	(nL/L)	threshold	compound	DB-WAX	(µg/kg)	(nL/L)	threshold		
Aliphatic Alcohols											
butanol	1138	25	500	-1.3	1-hydroxy-2-propanone	1295	2000	105	-1.8		
1-penten-3-ol	1152	28	400	-1.2	1-octen-3-ol	1448	57	1	1.8		
pentanol	1246	89	4000	-1.7	2,3-butanediol	1539	21	>105	<-3.7		
3-hydroxy-2-butanone (acetoin)	1278	200	8000	-1.6	meso-2,3-butanediol	1576	76	>10 ³	<-3.1		
Aliphatic Aldehydes and Ketones											
2-methylbutanal	912	630	3	2.3	nonanal	1389	110	1	2.0		
3-methylbutanal	914	680	0.2	3.5	(E)-2-nonenal	1532	21	0.08	2.4		
2,3-butanedione	966	190	3	1.8	(E,E)-3,5-octadien-2-one	1566	75	150	-0.3		
2,3-pentanedione	1050	200	20	1.0	(E)-2-decenal	1641	45	0.4	2.1		
hexanal	1077	160	4.5	1.6	(E,E)-2,4-nonadienal	1698	39	0.01	3.6		
2-heptanone	11/8	34	140	-0.6	(E)-2-undecenal	1749	160				
(T) 2 housed	1180	39	3	1.1	(E,Z)-2,4-decadienal	1/00	270	0.07	4.9		
(E)-2-nexenan	1214	17	17	-0.4	(<i>E</i> , <i>E</i>)-2,4-decadienal	1800	980	0.07	4.2		
2 hontonal	1201	17	12	-0.5	"4.5 opena 2 poponal"	1033	3	00	-1.1		
2-neptenal	1320	12	13	-0.03	4.5 apovy 2 decenal (isomor A)	1075	10				
1 octor 3 oro	1204	3	0.7	1.1	4.5 epoxy 2 decenal (isomer R)	1000	25				
$(F)_{2}$ octornal	1425	12	0.05	1.0	4,5-epoxy-2-decentar (Isoiner D)	1990	20				
(<i>L</i>)-2-0000111	1425	12	5	0.0	Aliphatic Acids						
acetic acid	1440	700	22000	-1.5	3-methylbutyric acid	1665	61	250	-0.6		
propionic acid	1520	110	2000	-1.3	pentanoic acid	1730	55	3000 (lit.)	-1.7		
isobutyric acid	1560	100	50	0.3	hexanoic acid	1825	130	3000 (lit.)	-1.4		
butyric acid	1625	98	240	-0.4	heptanoic acid	1925	28	3000 (lit.)	-2.0		
				Aromatic	and Furan Compounds						
2-pentylfuran	1226	43	6	0.9	2-methoxyphenol (guaiacol)	1855	30	3	1.0		
furfural	1456	92	3000	-1.5	2-methyl-3-hydroxy-4-pyranone (maltol)	1960	200	2500	-1.1		
2-acetylfuran	1500	2	104	-3.7	2,5-dimethyl-4-hydroxy-3(2H)-	2030	260	60	0.64		
benzaldehyde	1516	4	350	-1.9	furanone (Furaneol)						
phenylacetaldehyde	1636	250	4	1.8	4-vinylphenol	2170	60	10	0.78		
furfuryl alcohol	1656	540	2000	-0.6	4-vinyl-2-methoxyphenol	2180	1500	3	2.7		
				N1:4	(4-vinylguaiacol)						
nyridine	1181	30	2000	-1.8	"2-propionyl-1-pyrroline"	1/15	15	0.1.(lit)	22		
pyrazine	1207	260	18×10^{4}	-2.8	2-pt byl-3.6-dimethylpyrazine	1415	13	8.6	0.15		
2-methylpyrazine	1262	1830	6×10^4	-1.5	2-vinylpyrazine	1435	6	700	-2.1		
2.5-dimethylpyrazine	1320	500	1700	-0.5	2-ethyl-3 5-dimethylpyrazine	1455	4	0.04	2.1		
2.6-dimethylpyrazine	1326	200	1500	-0.9	"2-methyl-6-vinylpyrazine"	1485	4	0.04	2.0		
2-ethylpyrazine	1331	260	6000	-1.4	"2-isopropenylpyrazine"	1492	5				
2-acetyl-1-pyrroline	1335	57	0.1	2.8	pyrrole	1507	120				
2.3-dimethylpyrazine	1344	290	2500	-0.9	"2.5-dimethyl-3-vinylpyrazine"	1535	8				
2-ethyl-6-methylpyrazine	1380	80			2-acetyltetrahydropyridine (1,4,5,6 and 3,4,5,6 isomers)	1585	1100	2	2.7		
2-ethyl-5-methylpyrazine	1385	61	100	-0.2	2-acetylpyridine	1597	120	19	0.8		
2-ethyl-3-methylpyrazine	1402	12			2-acetylpyrrole	1970	30	$17 imes 10^4$	-3.8		
2,3,5-trimethylpyrazine	1404	170	23	0.9	2-aminoacetophenone	2210	100	0.2	2.7		
J 1 J					Lactones						
v-butyrolactone	1623	620	>103	<-0.2	v-octalactone	1916	36	8	07		
, sucronactoric	1000	020	10	0.2	Othons	1010	00	0	0.7		
dimethyl diaulfide	1070	E	19	0.4	Others 2 funfumilthial	1495	- E	0.000	~ 9.0		
limonene	11070	5 97	300	-0.4	2-mathylthionronanal (mathional)	1420	~ 5 20	0.000	~2.9		
dimethyl trisulfide	1971	~1 <9	0.01		o mempimopropanai (memionai)	1440	20	0.2	2.0		
unically i disulluc	1014	· 60	0.01	·							

^a Kovats retention index found on the DB-WAX capillary GC column. ^b Probably only accurate to the right order of magnitude. ^c Odor threshold measured in water solution and represented as nL of compound/L of water (ppb).

are not separated using a DB-1 capillary GC column but are well separated using a DB-WAX column. The large difference between the odor potencies of these two isomers was first pointed out to the authors by Prof. W. Grosch (1995) and subsequently confirmed by the authors (Buttery and Ling, 1997b).

Furans. These also are fairly typical of furans generally found in cooked foods. The 2-pentylfuran found probably originates from the cooking oils used in deep frying. Being relatively potent and occurring in moderate concentrations, it is a likely contributor to the total aroma and flavor. Also found in moderate amounts was 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone (furaneol), which has been identified previously by the authors in a number of cooked corn products (Buttery and Ling, 1997a).

Sulfur Compounds. Schieberle (1991) had identified the very potent odorant 2-furfurylthiol in popcorn, and this was later confirmed by us (Buttery et al., 1997). This compound was also identified in the corn tortilla chips in the present study. As with popcorn, the authors had difficulty in determining its concentration with any certainty because of its very low concentration and its readiness to oxidize with air.

Methional (3-methylthiopropanal) was found in moderate concentration in tortilla chips and has been known (Guadagni and Buttery, 1974) to be an important contributor to the aroma and flavor of fried foods. It was found in a high enough concentration, considering its potency, to contribute significantly to the aroma and flavor of tortilla chips. Dimethyl disulfide and trisulfide



KYNURENINE

Figure 1. 2-Aminoacetophenone probably results from the base-catalyzed oxidation of tryptophan in the treatment of corn with lime in the first step of the tortilla and tortilla chip process. This type of degradation of tryptophan had been first reported by Tabone et al. (1951) who showed that kynurenine was an intermediate.

were also identified, but only the trisulfide was found above its water odor threshold concentration.

Benzene Derivatives. Again these are compounds commonly found in cooked foods. Probably the most important are 4-vinylguiacol (2-methoxy-4-vinylphenol), one of the major volatiles, and phenylacetaldehyde, both are reasonably potent odorants. Also included in this group are benzaldehyde, guaiacol, and 4-vinylphenol.

Free Aliphatic Acids. The staight chain C_2-C_7 aliphatic acids were found together with isobutyric and 3-methylbutyric acids. These are generally relatively weak odorants, and only isobutyric acid has a high enough concentration/threshold ratio to have a chance to contribute to the aroma and flavor.

Recoveries Using Isolation Method. Recoveries of compounds were determined by making up known concentrations of authentic compounds in water, adding a known amount to the weighed amount of sodium sulfate, carrying them through the isolation process (used for the blended moistened tortilla chips as described in the experimental), and determining the absolute recovery using an internal standard added to the isolated concentrate. Average recoveries were in general reasonable for most compounds such as 81% for 2-aminoacetophenone, 70% for (E, E)-2,4-decadienal, 31% for nonanal, 62% for 2-phenylethanol, 68% for 2,3butandiol, 40% for 1-octen-3-ol, 27% for pentanol, 42% for trimethylpyrazine, 62% for 3-methylbutyric acid, 52% for γ -butyrolactone, and 33% for guaiacol.

The recovery of some compounds varied with the pH conditions. Methional gave an average recovery of 38% with the slightly acidic (pH 4.5) conditions but only 9% with the close to neutral (pH 7.4) conditions. The lower recovery of some more volatile compounds such as pentanol may be due to loss into the atmosphere during the mixing with the sodium sulfate.

Probable Contribution to Aroma. Odor thresholds in water solution and concentration/threshold ratios for many tortilla chip components have been previously reported for the related taco shells in the preliminary study by the authors (Buttery and Ling, 1995) and are included in Table 2 for tortilla chips together with those of the additionally identified compounds. The log₁₀ of the ratio concentrated/threshold is also listed (here the

assumption is made that most compounds have densities close to 1).

As discussed in the previous paper, because fried products such as tortilla chips contain only very low concentrations of water, odor thresholds measured in water are not strictly applicable as they are with mostly aqueous foods. The liquid medium in tortilla chips is primarily vegetable oil, and it would seem desirable to determine the thresholds in vegetable oil as was done in a study of potato chip volatiles carried out at this laboratory some years ago (Guadagni et al., 1972). This is very difficult to do in practice because of the background odors usually present in vegetable oils. When the chips are chewed in the mouth, the saliva gives a mostly water medium together with the chips oil medium, resulting in control of the atmospheric concentration of volatiles somewhat between that of oil and water. This effect was discussed by us in our recent more general paper on tortilla products (Buttery and Ling, 1995).

GC odor descriptions were also obtained by sniffing the DB-WAX capillary GC effluent during the separtion of volatiles from the tortilla chip volatile isolate. Such methods have been used very effectively by Grosch and co-workers (cf. Grosch, 1994). In the present work, no thorough AEDA method was used, but the evaluation was carried out at high dilution where only the most potent odorants could be detected. Recognized odor characters were found for biacetyl, 3-methylbutanal, 2,3pentadione, hexanal, 2-pentylfuran, 1-octen-3-one, 2-acetyl-1-pyrroline, dimethyltrisulfide, 2-propionyl-1pyrroline, methional, 2-ethyl-3,5-dimethylpyrazine, 2-acetyltetrahydropyridine, (E)-2-nonenal, (E,E)-2,4decadienal, furaneol, 4-vinylguaiacol, and 2-aminoacetophenone.

If we make the assumption that when tortilla chips are chewed in the mouth they are in a predominately water medium from saliva, then the compounds with high probabilities of contributing to the flavor are those with high concentrated/threshold ratios. These include (E,E)- and (E,Z)-2,4-decadienal, (E,E)-2,4-nonadienal, 3-methylbutanal, 2-furfurylthiol, 2-acetyl-1-pyrroline, 2-acetyltetrahydropyridine, 2-aminoacetophenone, 4-vinylguaiacol, (E)-2-nonenal, 2-methylbutanal, 2-propionyl-1-pyrroline, (E)-2-decenal, methional, and 2-ethyl-3,5-dimethylpyrazine.

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