Studies on Flavor Volatiles of Some Sweet Corn Products

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The volatiles of some canned, frozen, and fresh sweet corn products were analyzed by capillary GLC–MS. Important aroma compounds identified include 2-acetyl-1-pyrroline and 2-acetyl-2-thiazoline. Major volatiles found in all three corn products include dimethyl sulfide, 1-hydroxy-2-propanone, 2-hydroxy-3-butanone, and 2,3-butanediol. Pyridine, pyrazine, alkylpyrazines, and 2-acetylthiazole were additional major components in the canned products but minor components in the fresh and frozen products. Comparison of calculated odor units indicated that the compounds most important to canned sweet corn aroma include dimethyl sulfide, 2-acetyl-1-pyrroline, 2-ethyl-3,6-dimethylpyrazine, acetaldehyde, 3-methylbutanal, 4-vinylguaiacol, and 2-acetylthiazole. The alkylpyrazine and 3-methylbutanal are less important to fresh sweet corn aroma, where 2-acetyl-2-thiazoline also seems to be important.

INTRODUCTION

Sweet corn is marketed in several different forms including the fresh or frozen forms as well as the canned creamed and whole kernel forms. A number of authors had previously carried out studies of the volatile flavor compounds of some of these products. Flora and Wiley (1974), using direct injection of headspace vapors with gas–liquid chromatography (GLC) and mass spectrometry (MS), identified methanethiol (methyl mercaptan), acetaldehyde, ethanol, ethanethiol, and dimethyl sulfide in sweet corn. Sensory studies they carried out indicated that, of these, dimethyl sulfide contributed the most to the aroma. In a study aimed at higher boiling compounds Boyko et al. (1978) isolated the volatiles from sweet corn using dynamic headspace sampling with porous polymer trapping. From GLC–MS analysis they identified the following compounds: diacetyl; 2-methylpropanol; methyl butyrate; acetoin; pyrazine; pyridine; 2-ethylbutanal; methyl-, 2,5-dimethyl-, 2,6-dimethyl-, trimethyl-, 2-ethyl-5-methyl-, 2-ethyl-3,6-dimethyl-, and 2,6-diethyl-3-methyl-substituted pyrazines; furfural; 2-heptanone; hexanol; methional; 2-acetylfuran; dimethyl sulfoxide; 2,5-dimethyldihydro-2*H*-furan-3-one; 2-furfuryl alcohol; dimethyl trisulfide, 2-octanone; 2-methyltetrahydrothiophen-3-one; 2-acetylthiazole; 3-methyl-2-cyclohexenone; dimethyl sulfone; 2-methylfurfural; 2-acetyl-4-methylthiazole; 1,8-cineole; 2-thienyl alcohol; butylbenzene; and 5-methyl- and 5-ethyl-4-oxo-1,3-dithialane. Some of the above were identified only tentatively.

We (Buttery et al., 1978) had previously identified (GC–MS) 2-nonanol, 4-vinylguaiacol, 2-heptanol, (*Z*)-4-hepten-2-ol, 2-acetylthiazole, 2,4,5-trimethylthiazole, and thialdine in the volatile oil from atmospheric steam distillation continuous extraction (SDE) of previously uncooked sweet corn. We had also analyzed volatiles in the vacuum SDE isolated oil from uncooked corn (Buttery et al., 1978) with regard to possible insect attractants.

The present study was aimed at confirming the identification of components in major cooked sweet corn products and obtaining some idea of their concentrations and their probable degree of contribution to the aroma and flavor of sweet corn.

EXPERIMENTAL PROCEDURES

Materials. Samples of sweet corn products (major brands) were obtained from local supermarkets. The exact varieties used were not determined but were common major commercial varieties. Diethyl ether was freshly distilled through a 60 cm long Pyrex helice packed column, stored in the dark and protected by adding ca. 0.001% of Ethyl antioxidant 330.

Authentic samples for reference were obtained from reliable commercial sources or synthesized following established methods. Compounds were purified by preparative GLC separation and their identities verified by spectral (MS or IR) methods.

Isolation of Volatiles. The isolation method used high-flow dynamic headspace sampling with Tenax trapping and was similar to that previously described by us (Buttery et al., 1988) for tomato volatiles.

The fresh corn was cooked by boiling for 10 min with the kernels still on the cob. The kernels were then removed with a knife directly before isolation of volatiles. Frozen corn was heated in a microwave oven following the packet instructions. Canned whole kernel and creamed corns were used directly from the can. The corn sample (100 g) was placed in a blender with 50 mL of water and then blended for 10 s. Sodium chloride (54 g) and sodium carbonate (7.5 g) were then added, and the mixture was blended again for 30 s. The pH of the mixture was 9.4–9.8. For quantitative studies internal standards were also added at this point before blending. The mixture was placed in a 1-L flask containing a large efficient magnetic stirrer. Fitted to the neck of the flask was a suitable head allowing entry of sweep gas directed onto the surface of the vigorously stirred corn mixture and exit to the Tenax trap (14 cm long × 2.2 cm i.d.; ca. 10 g of Tenax). The sweep flow used was purified air at a flow rate of 3 L/min. The isolation was continued for 2 h. The Tenax trap was then removed and the volatiles eluted from the trap with freshly distilled diethyl ether (ca. 50 mL). The ether extract was then concentrated to ca. 50 μL using a warm water bath and a micro-Vigreux distillation column.

Quantitative Analysis of Dimethyl Sulfide and Acetaldehyde. A ground glass stoppered 250-mL Erlenmeyer flask was modified to have a hole (ca. 2-mm diameter) through the side of the flask ca. 6 cm above the base. This hole was covered with three layers of Teflon tape. The corn product (50 g) was placed in the flask together with 50 mL of water and 10.0 mL of an internal standard containing 10.0 ppm of benzene in water. A magnetic stirrer was also added and the flask stoppered. The mixture was stirred at 25 °C for 10 min. A sample of the vapor (3 mL) was removed with a glass syringe through the Teflon tape (acting as a septum) covered hole and injected directly into the GLC instrument. Calibration factors were determined previously by GLC analysis of headspace samples from known concentrations of dimethyl sulfide and acetaldehyde against the benzene internal standard.

Capillary GLC–MS. The capillary GLC column was 60 m long × 0.32 mm i.d. fused silica coated with bonded methylsilicone DB-1. The injector (1/20 split) temperature was 170 °C, and the column was held at 30 °C for the first 25 min and then temperature programmed at 4 °C/min to 200 °C and held at this temperature

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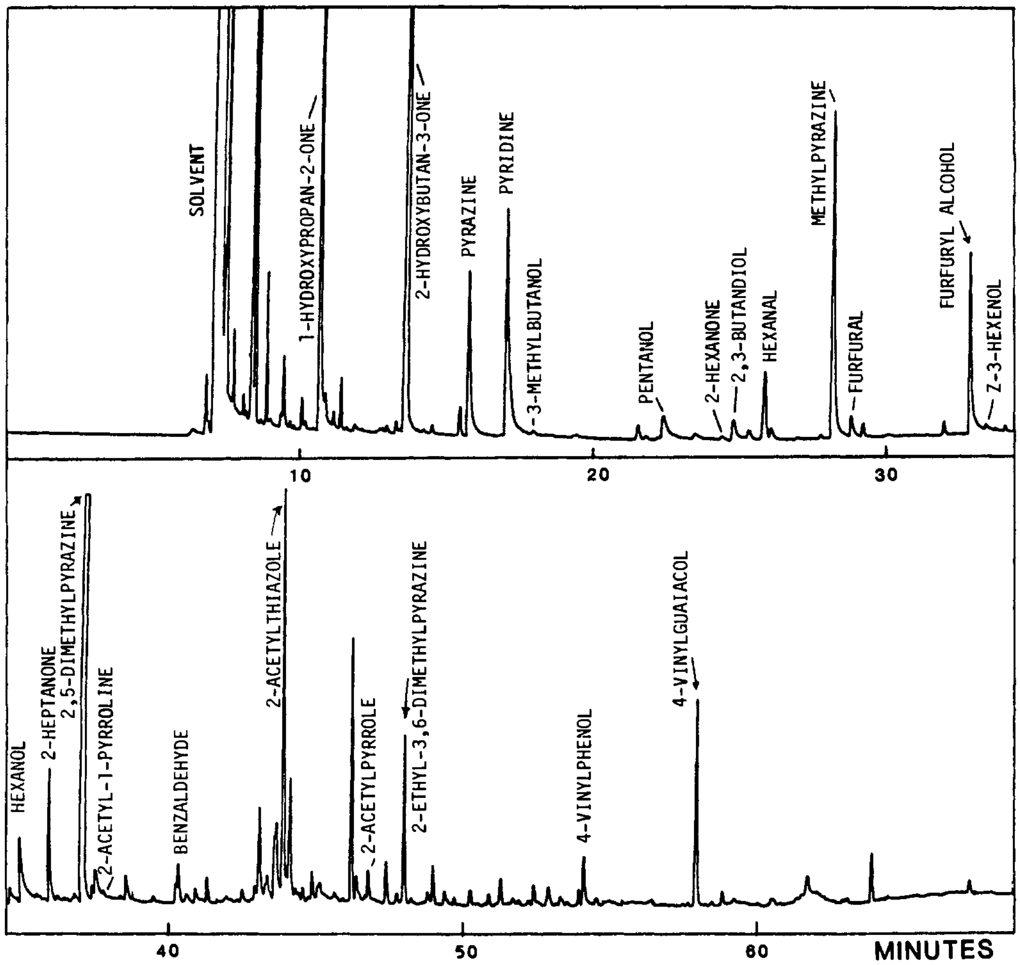

Figure 1. Capillary GLC analysis of volatiles isolated from canned sweet corn (whole kernel style) using a DB-1 fused silica capillary column programmed from 30 to 200 °C.

for a further 20 min. The GLC instrument was a Model HP5890 which was directly coupled to a HP5970 quadrupole mass spectrometer. The average carrier gas (He) flow velocity was 22 cm/s.

Quantitative Analysis. Three compounds, 2-pentanone, 3,5-dimethylpyridine, and quinoline, were used as internal standards. These were dissolved in purified water at a concentration of 20 ppm each to make a stock solution. This was stored in a refrigerator. Normally 1.0 mL of this solution was added during the isolation before trapping of the volatiles. Concentrations were determined by comparison of GLC peak areas with those of the standards. Each standard was used to cover a range of compounds close to its retention time, and combined recovery and response factors were determined for each compound relative to its particular internal standard by using measured amounts of standard solutions of the synthetic forms of the sweet corn volatiles. These factors were found as follows: (1, relative to internal standard 2-pentanone) 1-hydroxy-2-propanone 1%, 3-hydroxy-2-butanone 12%, 2,3-pentanedione 61%, pyrazine 3%; (2, relative to standard 3,5-dimethylpyridine) pyridine 48%, 3-methylbutanol 41%, hexanal 57%, hexanol 43%, methylpyrazine 31%, furfural 34%, furfuryl alcohol 7%, 2-heptanone 75%, heptanal 50%, benzaldehyde 90%, 2-acetylfuran 45%, 2,5-dimethylpyrazine 53%, 2-acetyl-1-pyrroline 29%, 2-acetyl-1,4,5,6-tetrahydropyridine 39%, 2-ethyl-3,6-dimethylpyrazine 82%. Factors for other compounds were assumed to be similar to that of related compounds; e.g., the recoveries of 4-vinylphenol and 4-vinylguaiacol were assumed to be similar to that of 4-ethylphenol (7% relative to quinoline).

RESULTS AND DISCUSSION

Volatiles were isolated from the canned products using the product directly from the can. The fresh corn and frozen corn were first cooked immediately before volatile isolation. Some water was added to make the mixture fluid and then the mixture blended. The mixture was saturated with salt (NaCl) and made slightly alkaline with sodium carbonate to facilitate isolation of some basic compounds (such as the 2-acetyl nitrogen heterocyclic compounds). For the C_4–C_{10} range of compounds the volatiles were isolated from the vigorously stirred blended mixture at room temperature using high-flow dynamic headspace Tenax trapping similar to that previously used by some of the authors for other products [cf. Buttery et al. (1988)]. For the very volatile compounds, dimethyl sulfide and acetaldehyde, analysis was carried out by direct injection of 3 mL of headspace from above the product into the GLC. Capillary GLC–MS analysis was carried out on the concentrated isolate from each of the products. Figure 1 shows the capillary GLC analysis of the volatiles isolated from the canned whole kernel corn. Table 1 lists the compounds identified together with major ions and retention index found. Compounds were identified by comparison with mass spectral and GLC retention data of authentic samples. Table 2 lists concentrations found. These data were obtained by comparing GLC peak areas to that of the three internal standards 2-pentanone, 3,5-dimethylpyridine, and quinoline. Combined recovery–response factors relative to the internal standards were obtained for most compounds. The accuracy of the quantitative data is limited because of the small number of samples analyzed but would be expected to be of the right order of magnitude and give some ideas of the relative concentrations of these compounds in the different products. It can be seen that the major components

Table 1. Mass Spectral and GLC Data Found for Volatile Components Identified in Sweet Corn Products

compound[a]	major MS ions[b]	Kovats index[c]
aliphatic alcohols		
2-methylpropanol	43, 33, *74*, 57	608
1-hydroxy-2-propanone	43, 31, *74*, 59	618
butanol	31, 56, 41	638
3-hydroxy-2-butanone	45, 43, *88*, 73	674
3-methylbutanol	55, 42, 70, 31	714
2-methylbutanol	57, 41, 31, 70	718
pentanol	42, 55, 70, 31	752
2,3-butanediol	45, 57, 31, 75, *90*	768
(*Z*)-3-hexenol	41, 67, 55, 31, 82	834
hexanol	56, 43, 69, 84	848
aliphatic aldehydes and ketones		
2,3-butanedione	43, *86*, 29, 53	558
3-methylbutanal	44, 58, 29, 71, *86*	627
2-methylbutanal	57, 29, 41, *86*, 71	637
2,3-pentandione	43, 29, 57, *100*	664
(*E*)-3-penten-2-one	69, 41, *84*, 55	711
2-hexanone	43, 58, *100*, 71, 85	761
hexanal	44, 56, 29, 72, 82	772
2-heptanone	43, 58, 71, *114*, 99	865
heptanal	44, 70, 55, 29, 86	876
nitrogen compounds		
pyrazine	*80*, 53, 26, 38	706
pyridine	*79*, 52, 39	712
2-methylpyrazine	*94*, 67, 39, 53	796
2,5-dimethylpyrazine	*108*, 42, 81, 52, 66, 93	883
2-acetyl-1-pyrroline	43, 69, 83, *111*, 55	892
2-acetylthiazole	43, *127*, 58, 112, 99, 85	979
2-acetylpyrrole	94, *109*, 66, 39, 53	1024
2-ethyl-3,6-dimethylpyrazine	135, 42, 56, 108, 121, 67	1054
2-acetyl-2-thiazoline	43, 60, *129*, 87, 101	1063
furans and aromatic compounds		
furfural	39, *96*, 29, 67, 50	800
furfuryl alcohol	*98*, 41, 81, 53, 70, 31	827
2-acetylfuran	95, 39, *110*, 68, 51	876
benzaldehyde	77, 105, 51, 39, 63	926
2-pentylfuran	81, 53, *138*, 39, 95, 68	977
benzyl alcohol	79, *108*, 51, 39, 91, 65	1004
acetophenone	105, 77, *120*, 51, 43	1031
benzoic acid	105, *122*, 77, 51, 39, 94	1143
4-vinylphenol	*120*, 91, 39, 65, 51, 79	1190
4-vinylguaiacol	*150*, 135, 39, 77, 51, 107	1280
others		
3-(methylthio)propanal (methional)	48, 29, *104*, 61, 76, 35	856
dimethyl trisulfide	*126*, 45, 79, 64, 111, 32	941
2-thiophenemethanol	*114*, 85, 97, 45, 53, 69	1000
limonene	68, 93, 39, *136*, 53, 79	1020

[a] Mass spectra and GLC retention index consistent with those of authentic samples. [b] Largest ions first with one major ion each 14 mass units and molecular ion in italics if present. [c] Kovats index on DB-1 fused silica capillary.

Table 2. Concentrations of Volatiles Found in the Different Forms of Cooked Sweet Corn in Parts (Milliliters) of Compound per Billion (10^9) Parts (Grams) of Product (ppb)

	concn (ppb)			
compound	can cream	can kernel	frozen kernel	fresh kernel
aliphatic alcohols				
2-methylpropanol	20	17	20	6
1-hydroxy-2-propanone[a]	>5000	>5000	>2000	830
butanol	5	8	20	2
3-hydroxy-2-butanone[a]	>5000	>5000	>4000	>1100
3-methylbutanol	16	4	3	<1
2-methylbutanol	4	1	9	<1
pentanol	7	15	39	5
2,3-butanediol[a]	>5000	>5000	>5000	>3000
(*Z*)-3-hexenol	1	2	<1	<1
hexanol	9	7	1	4
aliphatic aldehydes and ketones				
acetaldehyde	1400	1000	1200	1700
2,3-butanedione	5	5	<2	<2
3-methylbutanal	10	19	7	<4
2-methylbutanal	19	67	14	<4
2,3-pentanedione	9	8	2	<2
(*E*)-3-penten-2-one	6	2	<1	<1
2-hexanone	1	2	<5	<1
hexanal	14	24	5	4
2-heptanone	6	21	3	18
heptanal	6	4	<5	<1
nitrogen compounds				
pyrazine[a]	1700	2000	<5	10
pyridine	630	340	<1	1
2-methylpyrazine	180	160	3	<2
2,5-dimethylpyrazine	880	400	4	30
2-acetyl-1-pyrroline	44	2	3	2
2-acetylthiazole	140	80	5	6
2-acetylpyrrole	8	12	<1	<1
2-ethyl-3,6-dimethylpyrazine	59	22	2	<1
2-acetyl-2-thiazoline	<1	<1	5	6
furans and aromatic compounds				
furfural	8	7	2	<1
furfuryl alcohol[a]	1000	350	8	5
2-acetylfuran	4	2	<1	<1
benzaldehyde	16	5	10	4
benzyl alcohol	8	2	<1	<1
acetophenone	10	5	<1	<1
benzoic acid[a]	10	50	10	<5
4-vinylphenyl[a]	56	320	400	150
4-vinylguaiacol[a]	110	1100	120	110
others				
dimethyl sulfide	6800	9100	590	760
3-(methylthio)propanal	<1	<1	<1	<1
dimethyl trisulfide	<2	2	<2	<2
2-thiophenemethanol	18	7	<2	<2
limonene	<1	<1	10	2

[a] These compounds have very low recovery factors by dynamic headspace sampling and the values are only meant to give an idea of the order of magnitude.

include 3-hydroxy-2-butanone and related compounds such as 1-hydroxy-2-propanone and 2,3-butanediol. It is interesting that many volatiles previously found by some of the authors in fresh uncooked corn (Buttery et al., 1978) such as 2-nonanol and (*Z*)-4-hepten-2-ol were below detection in the cooked corn. These were apparently lost by steam vaporization in the cooking process.

Cracker Aroma Compounds. Compounds identified that possess cracker-like [cf. Teranishi et al. (1975)] or popcorn-like aromas include 2-acetyl-1-pyrroline and related acetyl nitrogen heterocyclic compounds such as 2-acetyl-2-thiazoline and 2-acetylthiazole. 2-Acetylthiazole had been previously reported in sweet corn (Boyko et al., 1978; Buttery et al., 1978). Neither 2-acetyl-1-pyrroline nor 2-acetyl-2-thiazoline (which are the more potent odorants of this group) had been reported in the previous studies on sweet corn volatiles. 2-Acetyl-1-pyrroline had been first found to be important to the flavor of aromatic rices (Buttery et al., 1983). It was later found to be important to the aroma of bread crust (Schieberle and Grosch, 1985) and popcorn (Schieberle, 1991). 2-Acetyl-2-thiazoline was first reported in meat volatiles (Tonsbeek et al., 1971). These probably contribute to the popcorn-like part of the flavor (aroma) of these sweet corn products.

Other related compounds that have been reported in popcorn such as 2-acetylpyrazine (Waldradt et al., 1970; Schieberle, 1991), 2-propionyl-1-pyrroline, 2-acetyl-3,4,5,6-tetrahydropyridine, and 2-acetyl-1,4,5,6-tetrahydropyridine (Schieberle, 1991) could not be detected in the sweet corn products, although they were actively looked for.

Sulfur Compounds. Boyko et al. (1978), besides the better known compounds dimethyl sulfide and 2-acetylthiazole, had also obtained good evidence for the presence of methional, dimethyl trisulfide, dimethyl sulfoxide, and dimethyl sulfone. In addition, they had also obtained tentative evidence for the presence of 2-methyltetrahydrothiophen-3-one, 2-acetyl-4-methylthiazole, 2-thiophenemethanol (thienyl alcohol), 3-methyl-2-thiophenecarboxaldehyde, and 5-methyl- and 5-ethyl-4-oxo-1,3-dithialane. We were able to confirm the well-identified compounds except for the sulfur oxides. We were also able to confirm the presence of 2-thiophenemethanol by direct comparison of MS and GLC data with those of an authentic sample. We were unable to confirm the presence of the other tentatively identified compounds, although a number of other unidentified sulfur compounds were detected at low concentrations (<5 ppb). Flora and Wiley (1974) had identified low concentrations of hydrogen sulfide, methanethiol, and ethanethiol in some samples of cooked sweet corn. These compounds were outside the range of compounds possible to analyze by the dynamic headspace method used by us in the present study and also by the method used by Boyko et al. (1978). Being relatively potent odorants, they very probably contribute to the total odor.

Methional. Methional was detected in all samples of sweet corn, but it is difficult to determine its concentration accurately because it shows a very low recovery (ca. 1%) with dynamic headspace isolation methods such as used in the present study. It also has a low odor threshold (0.2 ppb in water) and very likely contributes to the aroma and flavor of all forms of cooked corn.

2,3-Butanediol and Related Compounds. One group of related compounds that occur in high concentration in all sweet corn products studied includes 2,3-butanedione, 2,3-butanediol, 2-hydroxy-3-butanone, 1-hydroxy-2-propanone, and 2,3-pentandione. The 2,3-butanedione and 2-hydroxy-3-butanone had been reported in previous studies (Boyko et al., 1978). The alcohols in this group are very soluble in water, and it is difficult to isolate them quantitatively by dynamic headspace sampling such as used in this study or by distillation methods such as SDE. Their quantitative analysis is therefore of limited accuracy. They are, however, rather weak odorants and except for 2-hydroxy-3-butanone and 2,3-butanedione are not likely to contribute much to the total odor and flavor.

Differences between Products. In general, the concentrations of volatiles found in the canned products were many times higher than those found in the fresh and frozen products. Boyko et al. (1978) had noticed a similar difference between fresh and canned products. It can be seen that pyrazine itself, pyridine, and the alkylpyrazines are of the order of 10–100 times higher in concentration in the canned product than in the fresh or frozen product. This probably reflects the higher temperature used in can sterilization, etc., which can help produce many of these compounds. Some of the volatile formation might also occur during the normal long storage of canned products at room temperatures. It is interesting that 2-acetyl-2-thiazoline was only found in the fresh corn and the frozen corn and not in the canned corn products. In contrast, the canned products had relatively high concentrations of 2-acetylthiazole. The lignin-related compounds 4-vinylphenol and 4-vinylguaiacol occur in reasonable amounts in all types of sweet corn. They have also been found in popcorn (Schieberle, 1991) and are common in many cooked foods.

Geosmin. Geosmin [(*E*)-1,10-dimethyl-(*E*)-9-decalol] had been identified previously by some of the authors in uncooked fresh sweet corn (Buttery et al., 1978). It was below detection, however, in the present study with cooked sweet corn. Because of its very low odor threshold (0.02 ppb in water), even undetectable concentrations could be effective, however. In the opionion of the authors the odor of geosmin can be detected in some samples of cooked corn, but there is considerable variation between samples which may be due to variety and also the growing conditions.

Table 3. Odor Thresholds of Sweet Corn Components and Calculated Log Odor Unit (OU) Values for Canned Cream Corn and Fresh Corn (Cooked)

compound	threshold[a] (ppb) (H_2O)	log OU cream	log OU fresh
dimethyl sulfide	0.3	4.4	4.5
dimethyl trisulfide	0.01	<2.3	<2
2-acetyl-1-pyrroline	0.1	2.2	1.3
2-ethyl-3,6-dimethylpyrazine	0.4	2.2	<0.4
acetaldehyde	15	2.0	2.1
3-methylbutanal	0.2	1.6	<1.3
4-vinylguaiacol	3	1.6	1.6
2-acetylthiazole	10	1.1	−0.2
3-hydroxy-2-butanone	800	>0.8	>0.14
2-methylbutanal	3	0.8	<0.1
4-vinylphenol	10	0.75	1.2
hexanal	4.5	0.5	−0.05
heptanal	3	0.3	−0.5
2,3-butanedione	3	0.2	<0.2
2-acetyl-2-thiazoline	1	0	0.8
2,5-dimethylpyrazine	1700	−0.3	−1.8
furfuryl alcohol	1900	−0.3	−2.6
2,3-pentanedione	20	−0.35	−1.0
butanol	500	−0.5	−2.4
pyridine	2000	−0.8	−3.3
1-hydroxy-2-propanone	80000	−1.2	−2.0
3-methylbutanol	250	−1.2	<−2.4
benzaldehyde	350	−1.3	−1.9
2-heptanone	140	−1.4	−0.89
(*Z*)-3-hexenol	70	−1.8	<−1.8
pyrazine	180000	−2.0	−2.2
2-methylpyrazine	60000	−2.5	−4.5
furfural	3000	−2.6	−3.5
pentanol	4000	−2.8	−2.9
2-thiophenemethanol	15000	−2.9	<−3.9
acetylfuran	10000	−4.0	−4.0
2-acetylpyrrole	170000	−4.3	−5.2

[a] Threshold concentration in parts (mL) of compound per billion (10^9) parts (mL) of water.

Probable Relative Contribution to Flavor (Aroma). Table 3 lists odor thresholds and calculated $\log_{10}$ odor unit (OU) values which are the log of the ratios of the concentrations found in the corn product divided by the threshold concentration in water. The values for canned creamed corn and fresh corn are compared. This gives some idea of the probable general order of contribution of components to the total odor.

Dimethyl sulfide, 2-acetyl-1-pyrroline, 2-ethyl-3,6-dimethylpyrazine, acetaldehyde, 3-methylbutanal, 4-vinylguaiacol, and 2-acetylthiazole rank the highest for canned cream corn. The data also indicate that 2-ethyl-3,6-dimethylpyrazine and 3-methylbutanal are less important to fresh sweet corn aroma and that 2-acetyl-2-thiazoline (not detected in canned corn) also contributes.

As mentioned above, the importance of methional and geosmin is difficult to evaluate by these methods, but they probably also contribute to the aroma and flavor.

2-Thiophenemethanol proved to be a particularly weak odorant with an odor threshold of 15 000 ppb. The related compound 2-thiophenecarboxaldehyde (only tentatively identified) was similarly weak with a threshold of 5000 ppb.

ACKNOWLEDGMENT

We thank Mark Fraser, Ken Goodnight, and Bob Sacher of Hunt-Wesson, Inc., for helpful discussion and some financial support of this research. We also thank Jean Turnbaugh for help in determining odor thresholds.

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Received for review July 22, 1993. Accepted December 16, 1993.[⊗]

⊗ Abstract published in *Advance ACS Abstracts*, February 1, 1994.