Roasted Chicory Aroma Evaluation by Gas Chromatography/Mass Spectrometry/Olfactometry

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ABSTRACT

Volatiles were isolated from roasted chicory by simultaneous steam distillation-solvent extraction (SDE) and dynamic headspace sampling (DHS). Many volatile components were identified in SDE (92) and DHS (64) extracts. Many pyrazines and N-furfurylpyrroles (N-furfurylpyrrole, N-furfuryl-2-formylpyrrole, and N-furfuryl-2-acetylpyrrole) were identified for the first time in roasted chicory. Aroma extract dilution analysis showed that those extracts from SDE and DHS were similar with respect to predominant aroma-active components. 2-Ethyl-3,5-dimethylpyrazine, 2,3-butanedione, 1-octen-3-one, 3-methylbutanal, and one unknown compound with a chicory- and burnt sugar-like note were the most intense aroma-active components found in roasted chicory.

Key Words: chicory, aroma, volatile flavor

INTRODUCTION

ROASTED CHICORY (*CICHORIUM INTYBUS*, L.) HAS BEEN WIDELY used in coffee blends, since many coffee drinkers prefer the distinct roasted chicory flavor. However, little information is available on the characteristic volatile flavor components of roasted chicory. Tonsbeek et al. (1968) identified 4-hydroxy-5-methyl-3(2*H*)-furanone as having a roasted chicory-like aroma in beef broth. Kawabata and Deki (1977) reported acetophenone as the characteristic component of roasted chicory aroma. Sannai et al. (1982) studied the volatile profiles of roasted chicory root, but sensory evaluation of the compounds was not reported. Holscher (1996) compared the volatile flavor compounds of roasted coffee with those of various coffee surrogates including roasted chicory, and reported that methoxyisoalkylpyrazine and thiols in coffee aroma were not found in roasted chicory.

Aroma extract dilution analysis (AEDA) has been applied for identification of important aroma components (Ullrich and Grosch, 1987; Gasser and Grosch, 1988). AEDA involves analysis of a serially diluted flavor extract by gas chromatography/olfactometry (GC/O) to obtain a flavor dilution factor (FD factor, the highest dilution at which a substance is detected by GC/O) for each aroma-active component. AEDA provides valuable information on characteristics and intensities of important aroma compounds.

The objectives of this study were to analyze volatile flavor components of roasted chicory using simultaneous steam distillation-solvent extraction (SDE) and dynamic headspace sampling (DHS)/gas chromatography/mass spectrometry (GC/MS) and to evaluate characteristic aroma-active components of roasted chicory by AEDA.

MATERIALS & METHODS

Materials

Coarsely ground roasted chicory (*Chicory Leroux*) was obtained from a local coffee company in Baton Rouge, LA. Samples were stored at room temperature until extraction. All standard compounds were purchased from Aldrich Chemical Co. (Milwaukee, WI).

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Simultaneous steam distillation-solvent extraction (SDE)

Roasted chicory (250 g), distilled water (1.5 L), and 810 g of internal standard (I.S., 3-heptanol) were placed in a Likens-Nickerson type SDE apparatus (cat. no. K-523010-0000, Kontes, Vineland, NJ). Redistilled dichloromethane (50 mL) was used as extraction solvent. SDE was carried out for 2.5 h. SDE extracts were kept at -20°C overnight to facilitate water removal. Volume of extract was reduced to 10 mL under a gentle nitrogen stream, and residual moisture was removed by drying over 3 g of anhydrous sodium sulfate. Extract volume was further reduced to 0.5 mL under a nitrogen stream prior to analysis. Extractions were carried out in triplicate. Each extract was analyzed in duplicate.

Dynamic headspace sampling (DHS)

Roasted chicory (10 g) plus 100 mL of distilled water and 810 g of 3-heptanol (I.S.) were placed in a water-jacketed purge and trap apparatus (100 mL, cat. no. 991760, Wheaton, Millville, NJ). Solution at 60°C was stirred using a teflon-coated magnetic stirring bar. Volatiles were purged (50 mL/min) onto a Tenax-TA trap (0.32 g, Chrompack, Raritan, NJ) which had been previously washed with diethyl ether and methanol followed by baking at 340°C for 4 h while purging with helium. After purging, volatiles were eluted from the trap with 5 mL of redistilled diethyl ether. Ether extract was dried over 3 g of anhydrous sodium sulfate and its volume reduced to 100 μL under a gentle stream of nitrogen. Extractions were carried out in triplicate and each extract was analyzed in duplicate.

Gas chromatography/Mass spectrometry (GC/MS)

A Hewlett-Packard GC/mass selective detector (HP 5792A GC/5970B MSD) system was used for analysis of extracts. Each extract (5 μL) was injected in the splitless mode (injector temperature; 155°C, valve delay; 30 s) into a fused silica capillary column (Supelcowax 10, 60 m length × 0.25 mm i.d. × 0.25 μm film thickness, Supelco, Inc., Bellefonte, PA). The linear velocity of the helium carrier gas was 25 cm/s. Oven temperature was programmed from 40°C to 200°C at 3°C/min with initial and final hold times of 5 and 40 min, respectively. MSD conditions were: capillary direct MS interface temperature, 200°C; ion source temperature, 200°C; ionization energy, 70 eV; mass range, 33-300 a.m.u.; and electron multiplier voltage, 2200 V.

Compound identification

Compounds were positively identified by comparing retention indices (RI) (van den Dool and Kratz, 1963) and mass spectra with authentic standards. Tentative identifications were based on matching mass spectra of unknowns with those in the Wiley/NBS mass spectral library (Hewlett-Packard Co., 1988) or published data.

Aroma extract dilution analysis (AEDA)

The gas chromatography/olfactometry (GC/O) system used for AEDA has been described (Baek and Cadwallader, 1997). Serial dilutions (1:2) of SDE and DHS extracts were prepared using dichloromethane and diethyl ether as diluent, respectively. GC conditions were the same as for GC/MS. GC/O was performed by three panel-

ists as described (Baek and Cadwallader, 1997). Flavor dilution (FD) chromatograms were based on aroma-active compounds with $\log_2$FD factors 6 (1:64 dilution) for SDE extracts and 3 (1:8 dilution) for DHS extracts.

RESULTS & DISCUSSION

Volatile profiles of SDE and DHS extracts

SDE and DHS extracts exhibited similar aroma notes typically representing sweet, green, and nutty aromas and were considered representative of roasted chicory aroma. We identified 92 compounds in SDE extracts and 64 compounds in DHS extracts (Table 1). The two extracts had 61 compounds in common. Most compounds we identified are being reported for the first time in roasted chicory.

Thermally generated heterocyclic compounds such as furans, pyrazines, and N-furfurylpyrroles were in high relative abundance in both SDE and DHS extracts. Most pyrazines and N-furfurylpyrroles were identified for the first time in roasted chicory. Pyrazines are important volatile compounds with nutty notes (Maga, 1982). A number of pyrazines were identified in SDE (20) and DHS (14) extracts. Pyrazines (79) have been previously reported in coffee (Flament, 1991). N-Furfurylpyrroles were considered important in cof-

Table 1—Volatile components identified in SDE and DHS extracts of roasted chicory

		Peak area ratio			
		SDE[a]		DHS[b]	
Compound name	**RI[c]**	**Avg[d]**	**S.D.[e]**	**Avg.**	**S.D.**
Aldehydes					
2-Methylbutanal	908	–[f]		0.231	0.055
3-Methylbutanal	914	–		0.088	0.015
3-Methylpentanal*	1029	0.024	0.005	0.037	0.005
2-Butenal	1037	0.013	0.003	–	
Hexanal	1076	0.057	0.013	0.119	0.008
2-Methyl-2-butenal*	1091	0.002	<0.001	0.016	0.004
Nonanal	1394	nd[g]		0.056	0.004
Benzaldehyde	1527	–		–	
Phenylacetaldehyde	1645	–		0.179	0.011
2,4-Nonadienal	1698	–		nd	
(E,Z)-2,4-Decadienal	1760	–		0.025	0.004
α-Ethylidenephenylacetaldehyde*	1936	–		nd	
α-(2-Methylpropylidene)phenyl-acetaldehyde*	1943	0.027	0.023	0.113	0.016
5-Methyl-2-phenyl-2-hexenal*	2078	0.056	0.047	0.151	0.046
2,4-Dimethoxybenzaldehyde*	2350	–		nd	
Ketones					
3-Buten-2-one*	952	0.011	0.003	nd	
2,3-Butanedione (Diacetyl)	982	0.331	0.072	0.050	0.006
4-Methyl-2-pentanone	1008	0.003	0.001	0.006	0.002
2,3-Pentanedione	1055	0.425	0.093	0.714	0.090
3-Penten-2-one	1119	0.012	0.003	0.021	0.001
Cyclopentanone*	1176	0.018	0.004	0.027	0.002
2,4-Pentanedione*	1193	0.009	0.002	nd	
1-Octen-3-one	1291	–			
1-(3 or 2-Pyridinyl) ethanone*	1602	–		nd	
1-(1-ethyl-1*H*-pyrrol-2-yl)-ethanone*	1652	–		nd	
1-Phenyl-2-propanone*	1725	0.018	0.017	nd	
1-(1*H*-pyrrol-2-yl)-ethanone*	1969	0.096	0.085	nd	
4-Hydroxy-3-methylaceto-phenone*	2007	0.023	0.019	0.028	0.006
Furans					
2,5-Dimethylfuran	957	0.024	0.007	0.021	0.007
2-(2-Propenyl)-furan*	1199	0.005	0.001	0.005	0.001
2-Pentylfuran	1222	0.022	0.005	0.025	0.002
2-(Methoxymethyl)furan*	1227	0.024	0.005	0.053	0.002
Dihydro-2-methyl-3(2*H*)-furanone*	1261	1.787	0.403	1.705	0.109
Furfuryl formate	1393	nd		0.155	0.037
5-Methyl-2(3*H*)-furanone*	1433	–		0.152	0.009
2-Furancarboxaldehyde	1483	9.485	2.149	26.226	3.465
3-Methyl-2-furancarboxaldehyde*	1494	–		nd	
1-(2-Furanyl)-ethanone*	1510	1.338	0.397	3.257	0.277
2-Furanmethanol acetate	1526	–		0.974	0.072
5-Methyl-2-furancarboxaldehyde*	1582	2.078	0.879	5.323	0.448
2,2'-Bifuran*	1591	–		0.422	0.069
2,2'-Methylenebisfuran*	1599	–		0.133	0.009
2-Acetyl-5-methylfuran*	1611	0.039	0.028	nd	
Dihydro-2(3*H*)-furanone*	1635	0.067	0.043	nd	
2-Furanmethanol	1648	1.264	0.418	0.813	0.104
2-(2-Furanylmethyl)-5-methylfuran*	1669	0.055	0.040	0.171	0.018
1-(5-Methyl-2-furanyl)-1-propanone*	1677	0.032	0.024	nd	
Furancarboxylic acid*	1706	–		nd	
3,4-Dimethyl-2,5-furandione*	1736	0.031	0.026	nd	
2,2'-Methylenebis-5-methylfuran*	1741	0.046	0.038	0.112	0.012
2-Methyl-3-ß-furylpropenal*	1878	0.017	0.016	0.052	0.004
2,2'-oxybis(methylene)bisfuran*	1979	0.028	0.025	0.115	0.024
5-(2-Furanylmethyl)-2-furancarboxaldehyde*	2328	–		nd	
5-[(5-Methyl-2-furanyl)]methyl-2-furancarboxaldehyde*	2376	0.008	0.004	nd	
5-(Hydroxymethyl)-2-furancarboxaldehyde*	2499	–		nd	
Pyrazines					
Pyrazine	1200	0.002	0.001	0.002	<0.001
Methylpyrazine	1257	0.074	0.016	0.093	0.005
2,5-Dimethylpyrazine	1314	0.187	0.042	0.158	0.010
2,6-Dimethylpyrazine	1320	0.310	0.093	0.249	0.01
Ethylpyrazine	1326	0.028	0.007	0.040	0.004
2,3-Dimethylpyrazine	1339	0.063	0.015	0.015	0.001
2-Ethyl-6-methylpyrazine	1380	0.112	0.041	0.100	0.007
2-Ethyl-5-methylpyrazine	1386	0.015	0.005	0.062	0.005
Trimethylpyrazine	1400	0.062	0.022	0.071	0.005
2-Ethyl-3,6-dimethylpyrazine	1445	0.009	0.003	0.128	0.008
2-Ethyl-3,5-dimethylpyrazine	1465	0.019	0.004	0.013	0.006
Tetramethylpyrazine	1475	–		–	
2-Methyl-5-propylpyrazine*	1489	–		nd	
2-Ethenyl-6-methyl-pyrazine*	1492	–		nd	
A kind of pyrazine*	1494	–		nd	
2,5-Dimethyl-3-(2-methylpropyl) pyrazine*	1520	0.061	0.070	0.086	0.004
2-Methyl-5-(1-propenyl)pyrazine*	1534	–		nd	
2-Methyl-3-propylpyrazine*	1623	–		nd	
2,5-Dimethyl-3-(3-methylbutyl) pyrazine*	1626	–		0.120	0.010
2,5-Dimethyl-3-(3-methylbutyl) pyrazine*	1652	0.073	0.038	nd	
Pyrroles					
N-Furfurylpyrrole	1825	0.015	0.011	0.022	0.002
N-Furfuryl-2-acetylpyrrole*	2254	0.023	0.017	0.044	0.011
N-Furfuryl-2-formylpyrrole*	2271	0.009	0.005	0.017	0.005
Miscellaneous Compounds					
Toluene	1035	0.003	0.001	0.074	0.095
Dimethyl disulfide	1065	nd		0.030	0.004
Pyridine	1172	0.005	0.001	0.017	0.001
1-Methoxy-4-methylbenzene*	1271	0.009	0.003	0.006	<0.001
3-Methyl-butanoic acid*	1657	0.106	0.038	nd	
Methyl phenylacetate*	1755	–		0.057	0.005
2,6-Bis(1,1-dimethylethyl)-4-methylphenol*	1908	0.222	0.154	0.378	0.136
2-Methylphenol	1993	0.016	0.013	0.024	0.004
Phenol	1998	0.017	0.014	0.023	0.003
Octanoic acid*	2051	–		nd	
2,3-Dimethylphenol*	2068	–		0.009	0.002
Tertio butyl hydroxy anisole*	2084	0.042	0.031	0.164	0.056
2-Methoxy-4-(2-propenyl)phenol*	2164	–		nd	
Methyl hexadecanoate*	2207	0.017	0.005	nd	
2-Methoxy-4-(2-propenyl)-phenol*	2345	–		nd	
Hexadecanoic acid*	2458	–		nd	
Methyl 9,12-octadecadienoate*	2485	–		nd	

[a]Simultaneous steam distillation-solvent extraction.
[b]Dynamic headspace sampling.
[c]Retention index.
[d]Average peak area ratio (peak area of compound/peak area of internal standard, n=6).
[e]Standard deviation (n=6).
[f]Peak area not calculated due to either co-elution of several compounds or trace amount.
[g]Not detected.
*Tentatively identified.

fee aroma (Tressl et al., 1981). N-Furfurylpyrrole was described as having a green hay-like aroma contributing to the flavor of popcorn (Walradt et al., 1970). This compound has been identified in roasted coffee (Tressl et al., 1981), roasted peanuts (Walradt et al., 1971), tobacco smoke (Schmeltz and Hoffmann, 1977), and roasted filberts (Kinlin et al., 1972). N-Furfuryl-2-formylpyrrole and N-furfuryl-2-acetylpyrrole were identified from chicory based on published mass spectral data (Tressl et al., 1981; Baltes and Bochmann, 1987). These compounds have been identified in roasted coffee (Tressl et al., 1981) and popcorn (Walradt et al., 1970) and could have been formed via Maillard reaction. However, N-furfurylpyrroles were not indicated as important odorants in coffee (Semmelroch and Grosch, 1996) and popcorn (Schieberle, 1991).

Furans were known to be important in coffee (Flament, 1991). 2-Furancarboxaldehyde (furfural), a caramelization product of sugars, was the most abundant furan compound in both extracts; however, its contribution to overall roasted chicory aroma may be slight because of its high aroma threshold (23 ppm, Buttery et al., 1988). Furfural was probably important as a precursor in the formation of other aroma compounds (Silwar and Tressl, 1989).

Table 2—Aroma-active compounds detected in SDE and DHS extracts

No.[a]	Ri[b]	Compounds name	Aroma description
1	914	3-Methylbutanal	Chocolate
2	929	Unknown	Chocolate, Sweet
3	982	2,3-Butanedione	Buttery
4	1055	2,3-Pentanedione	Buttery
5	1291	1-Octen-3-one	Mushroom
6	1380	2-Ethyl-6-methylpyrazine	Green, nutty, Grassy
7	1445	2-Ethyl-3,6-dimethylpyrazine	Nutty, Potato
8	1457	Unknown	Mushroom
9	1465	2-Ethyl-3,5-dimethylpyrazine	Baked potato, Nutty
10	1522	Unknown	Spicy, Sweet
11	1545	Unknown	Nutty
12	1588	Unknown	Nutty, Mossy
13	1621	Unknown	Floral, Honeysuckle
14	1645	Phenylacetaldehyde	Floral, Honeysuckle
15	1691	Unknown	Sweet
16	1738	Unknown	Minty
17	1771	Unknown	Cotton candy
18	1825	Unknown	Chicory, Burnt sugar
19	1878	Unknown	Candy, Sweet

[a]Numbers correspond to those in figures 1 and 2.
[b]Retention Index.

GC/O evaluation of SDE and DHS extracts

FD chromatograms were compared for roasted chicory volatiles isolated by SDE (Fig.1) and DHS (Fig. 2). Aroma-active compounds detected in SDE and DHS extracts and their aroma descriptions are given in Table 2. Aroma intensities of SDE extracts were higher than those of DHS extracts because of the greater amounts of sample used for extraction as well as the higher extraction recovery by SDE. However, FD chromatograms of both SDE and DHS extracts were similar for predominant aroma-active compounds.

Some aroma-active compounds (13) with $\log_2$FD factors >6 were detected in SDE extracts, while 16 compounds with $\log_2$FD factors >3 were detected in DHS extracts. 2,3-Butanedione (no. 3, buttery; $\log_2$FD = 11) and 2-ethyl-3,5-dimethylpyrazine (no. 9, baked potato-like/nutty; $\log_2$FD = 11) had the highest $\log_2$FD factors in SDE extracts. Next was an unknown compound (no. 18, chicory- and burnt sugar-like; $\log_2$FD = 10), 1-octen-3-one (no. 5, mushroom-like; $\log_2$FD = 10), an unknown compound (no. 2, chocolate-like, sweet; $\log_2$FD = 10), and 3-methylbutanal (no. 1, chocolate-like; $\log_2$FD = 9). 2-Ethyl-3,5-dimethylpyrazine ($\log_2$FD = 8), 1-octen-3-one ($\log_2$FD = 7), 3-methylbutanal ($\log_2$FD = 5), and two unknown compounds (no. 18, $\log_2$FD = 7, no. 10, spicy/sweet; $\log_2$FD = 5) showed high $\log_2$FD factors in DHS extracts.

The most intense compound in both SDE and DHS extracts, 2-ethyl-3,5-dimethylpyrazine, could be formed via the Maillard reaction (Shibamoto and Bernhard, 1977; Amrani-Hemaini et al., 1995) and has been reported to be predominant in some foods (Schieberle and Grosch, 1987; Baek and Cadwallader, 1996). The threshold value of this compound was reported to be 0.04 g/L (Buttery and Ling, 1997). Other pyrazines, such as 2-ethyl-6-methylpyrazine (no. 6) and 2-ethyl-3,6-dimethylpyrazine (no. 7), contributed to the overall aroma of roasted chicory in both SDE and DHS extracts.

2,3-Butanedione had the highest $\log_2$FD in SDE extracts but was not predominant in DHS extracts. Chung and Cadwallader (1994) and Cadwallader et al. (1995) previously observed this and hypothesized that this might be due to the nonpolar Tenax TA adsorbent not effectively adsorbing this polar compound. Although 2,3-butanedione was absent from the FD chromatogram of DHS extracts, this compound was believed to be important in the aroma of roasted chicory. The threshold value of this compound was reported to be 2.6 ppb (Fors, 1983). 2,3-Pentanedione (no. 4), with a similar aroma to 2,3-butanedione, also was detected in both SDE and DHS extracts.

1-Octen-3-one contributes to the aroma of various foodstuffs (Cadwallader et al., 1994, 1995; Schieberle and Grosch, 1987; Gasser

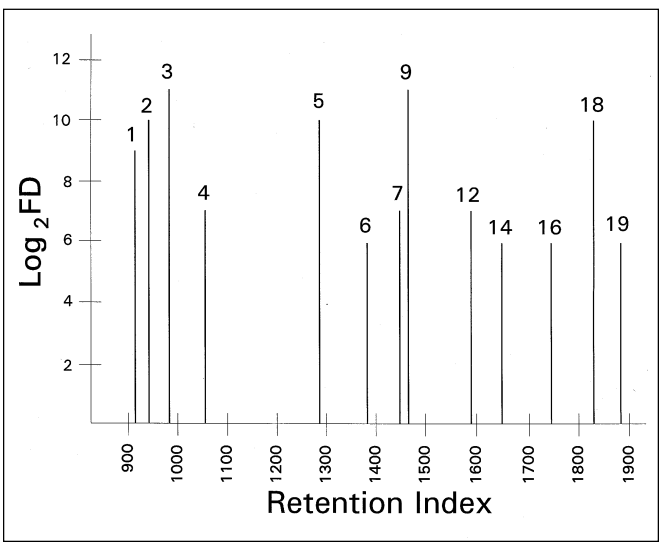

Fig. 1—Flavor dilution chromatogram of volatiles isolated from roasted chicory using simultaneous steam distillation-solvent extraction. Numbers correspond to those in Table 2.

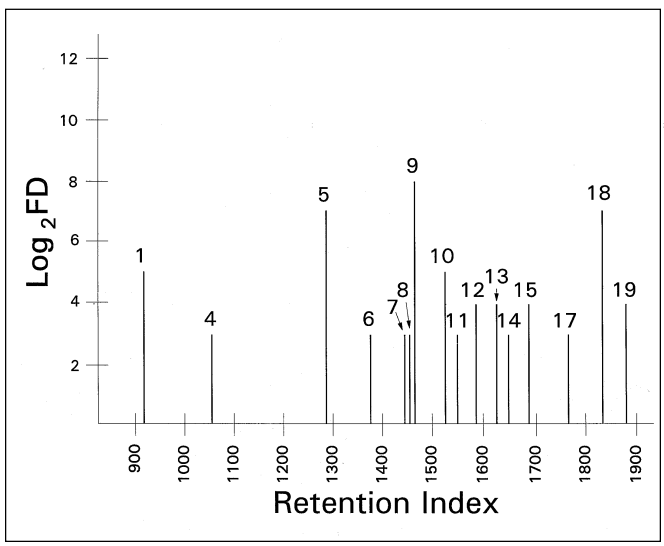

Fig. 2—Flavor dilution chromatogram of volatiles isolated from roasted chicory using dynamic headspace sampling. Numbers correspond to those in Table 2.

and Grosch, 1988). This compound has a mushroom-like aroma note and may be undesirable in chicory since it has been considered an off-flavor in some foods (Whitfield et al., 1982; Cadwallader et al., 1995). It has been shown to be a lipid oxidation product, predominantly from autoxidation of linoleic acid (Ullrich and Grosch, 1987).

3-Methylbutanal, a volatile Strecker aldehyde of leucine (Hurrell, 1982), was detected in both SDE and DHS extracts at high $\log_2$FD factors, suggesting it makes a notable contribution to the aroma of roasted chicory. Its presence might be desirable due to its chocolate-like aroma note. Co-elution with 2-methylbutanal as well as similar chocolate-like aroma notes of both compounds made them difficult to identify. GC/O analyses of authentic standards showed that 3-methylbutanal had almost a 50-fold lower threshold than 2-methylbutanal (data not shown). However, the concentration of 2-methylbutanal was only 3 times higher than 3-methylbutanal. Therefore, we concluded that 3-methylbutanal was primarily responsible for the chocolate-like aroma. Another volatile Strecker aldehyde, phenylacetaldehyde (no. 14), had a floral and honeysuckle note and relatively high $\log_2$FD factors in both SDE and DHS extracts. This suggests that Strecker degradation of some amino acids during roasting of chicory may be important in the aroma of roasted chicory.

In addition, several unknown compounds were detected at relatively high $\log_2$FD factors in roasted chicory. These were from SDE (5) and DHS (9). Of these, no. 18 had a chicory- and burnt sugar-like note and showed a high $\log_2$FD factor in both SDE and DHS extracts. This compound could not be identified because of its low abundance and co-elution with N-furfurylpyrrole. Four other unknowns, having cotton candy-like and nutty notes (no. 11, 12, 17, and 19), were thought to contribute to the overall aroma of roasted chicory because they were desirable and characteristic of roasted chicory. Additional studies using enrichment techniques (Semmelroch and Grosch (1996) might be useful for identifying these unknown components.

Results of AEDA demonstrated that 2-ethyl-3,5-dimethylpyrazine, 2,3-butanedione, 1-octen-3-one, 3-methylbutanal, and 5 unknown compounds contributed to the aroma of roasted chicory. However, techniques based on odor threshold detection such as AEDA have a limitation of a nonlinear relationship between perceived intensity of an odorant and its concentration (Frijters, 1978). Therefore, results of AEDA cannot indicate exactly the degree of importance of each odorant.

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