Aroma Components of Cooked Tail Meat of American Lobster (*Homarus americanus*)

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Key aroma components of cooked tail meat of American lobster (*Homarus americanus*) were studied by gas chromatography−olfactometry (GCO) techniques. Components of low and intermediate volatility were evaluated by aroma extract dilution analysis of solvent extracts prepared by direct solvent extraction−high vacuum distillation and vacuum steam distillation−solvent extraction, whereas headspace volatile components were assessed by GCO of decreasing headspace (static and dynamic modes) samples. Forty-seven odorants were detected by all techniques. 3-Methylbutanal (chocolate, malty), 2,3-butanedione (buttery), 3-(methylthio)propanal (cooked potato), 1-octen-3-one (mushroom), 2-acetyl-1-pyrroline (popcorn), and (E,Z)-2,6-nonadienal (cucumber), were identified as predominant odorants by all four isolation methods. The highly volatile compounds methanethiol (rotten, sulfurous) and dimethyl sulfide (canned corn) were detected by headspace methods only. These eight odorants along with three unknown compounds with crabby, amine, fishy odors were found to predominate in the overall aroma of cooked lobster tail meat.

Keywords: *Lobster; cooked lobster aroma; gas chromatography−olfactometry; aroma extract dilution analysis*

INTRODUCTION

The American lobster, *Homarus americanus*, is one of the most preferred seafoods in world markets and is always traded at a high price (*1*). Similar to other seafood, the desirable flavors of lobster and other crustaceans are formed during cooking via thermal reactions (*2*, *3*). Flavors of seafood are widely defined as complex systems consisting of equivalently significant taste- and aroma-active compounds (*4*). Identification of characteristic and important aroma compounds in cooked meaty flavors of crustaceans has been of interest in numerous studies (*5*−*10*). Recently, Chung (*11*) reported that the unique and desirable aromas of cooked crab (*Charybdis feriatus*) were observed at higher intensities in carapace meat than in claws and body meat. Moreover, Chung (*11*) suggested that the high amounts of aldehydes and lower levels of sulfur-containing compounds present in carapace meat were responsible for desirable crab aromas. In contrast to exhaustive studies on taste-active compounds of lobsters (*1*, *12*−*14*), few reports have been published concerning the aroma-active components of cooked lobsters (*2*, *3*). In addition, other studies mainly focused on determination of compounds imparting off-flavors in lobsters (*15*−*18*). Cadwallader and co-workers (*2*) employed aroma extract dilution analysis (AEDA) to determine potent aroma compounds in cooked Spiny lobster (*Panulirus argus*) tail meat. These compounds included 2-acetyl-1-pyrroline, 2,3-butanedione, 3-(methylthio)-propanal, 2-acetyl-3-methypyrazine, trimethylamine, 1-octen-3-one, (*Z*)-4-heptenal, and two unknown compounds which possessed marine, leather-like, and lobster-like notes. However, the highly volatile headspace aroma components were not addressed in this study. Identification of the aroma-impact components of cooked tail meat of American lobster will substantially aid in the development and production of diverse seafood flavors for formulated and value-added foods, especially surimi-based seafood analogues.

The aim of the present work was to apply AEDA and gas chromatography−olfactometry (GCO) of decreasing headspace (static and dynamic) samples to obtain more complete information on the aroma of cooked tail meat of American lobster. AEDA has been extensively used in the study of food aroma (*19*), including seafood products such as crab (*6*, *7*), crayfish (*8*), and lobster (*2*). GCO of decreasing headspace volumes is similar to AEDA in concept, but odorants detected in the lowest headspace volume are ranked as the most potent (*8*, *20*, *21*).

MATERIALS AND METHODS

Lobsters. Live American lobsters were purchased from a local market (Starkville, MS). All lobsters were transported alive to our laboratory and were beheaded with a stainless steel knife upon arrival. Immediately after removing lobster carapace, the tail meat with attached shell was steam-cooked in a double boiler until the temperature at geometric center of the tail reached 80 °C. Cooked lobster tail was then immediately cooled in an ice-water bath. The tail meat was then removed from the shell and was cut into small cubes (ca. 0.5 cm^3) for aroma extraction.

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Chemicals. Standard compounds listed in Tables 1−5 were purchased from commercial suppliers: **1**−**4, 6**−**11, 15, 19, 23**−**27, 29**−**31, 40**−**41, 43, 45,** and **47** (Aldrich, St. Louis, MO); **17** (Alfa, Ward Hill, MA); and **12** and **20** (Lancaster Synthesis Inc., Windham, NH). Standard **21** was obtained from Dr. R. Buttery (USDA, ARS, WRRC, Albany, CA) and **38** was obtained from Firmenich (Princeton, NJ). Compounds **22** and **32** were synthesized according to Ullrich and Grosch (*22*) and Milo and Grosch (*23*), respectively. Dichloromethane was from Aldrich and was re-distilled prior to use. Other reagents were from commercial sources.

GC−Olfactometry of Decreasing Headspace Samples (GCO−H). GCO−H was conducted according to Cadwallader and Baek (*8*) with some modifications. Freshly cooked lobster tail meat (10 g) was placed into a 250-mL round-bottom flask sealed with a Teflon septum. The flask was incubated in a 60 °C water bath for 20 min before headspace sampling. Headspace volumes of 20, 10, or 5 mL were withdrawn using a preheated (80 °C) gastight syringe and immediately injected (direct injection, 200 °C) into an HP5890 Series II GC (Hewlett-Packard Co., Palo Alto, CA) equipped with either a DB-WAX column (30 m × 0.53 mm i.d. × 1 μm film thickness) or a DB-5MS column (30 m × 0.53 mm i.d. × 1.5 μm film thickness). Both capillary columns were purchased from J&W Scientific (Folsom, CA). A 15-cm section at the head of the column was cooled in liquid nitrogen before injection in order to cryofocus the volatiles. After an injection, the GC was heated rapidly, and the run started when the oven temperature reached 40 °C. Helium was used as the carrier gas (1 mL/min). GC oven temperature was programmed from 40 to 200 °C at a rate of 8 °C/min (DB-WAX) or 6 °C/min (DB-5MS) with initial and final hold times of 5 and 30 min, respectively. Injector temperature was 200 °C. FID and the transfer lines were held at 250 °C. The sniffing port was supplied with humidified air at 30 mL/min. GCO−static headspace samples (SHS) sniffing was performed by two trained panelists for each headspace volume.

GC−Olfactometry of Decreasing Dynamic Headspace Samples (GCO−DHS). GCO−DHS was conducted according to Cadwallader and Baek (*8*) with some modifications. Freshly cooked lobster tail meat (5 g) was placed into a 25-mL headspace sampling tube (15.2 cm × 1.6 cm i.d.) which was then connected to a Tekmar 3000 purge and trap concentrator/cryofocusing module (Tekmar Co., Cincinnati, OH). The sample was preheated for 5 min at 60 °C prior to being purged with ultrahigh purity helium (40 mL/min, 60 °C) for either 6.5, 1.25, or 0.25 min onto a Tenax TA trap (part no. 12-0083-303, Tekmar Co.) maintained at 0 °C. These times corresponded to purge volumes of 250, 50, and 10 mL, respectively. After each sampling, the trap was dry-purged for 5 min to remove traces of moisture, and then the volatiles were desorbed (180 °C for 1 min) and sent to the cryofocusing module where they were cryofocused at −120 °C onto a 15-cm section of 0.53-mm i.d. deactivated fused-silica capillary column. Transfer lines and valves were maintained at a temperature of 175 °C, and the trap pressure control was set at 4 psi during purging. The split/splitless electronic pressure-control pneumatics of the GC were used to control the helium flow during thermal desorption of the Tenax trap (20 mL/min) and the cryofocusing trap (1.4 mL/min). The cryofocused volatiles were desorbed (splitless, 180 °C for 1 min) directly into the GCO system. The GCO system consisted of an HP5890 Series II GC, a flame ionization detector (FID), and an olfactometer (a sniffing port). Separations of the volatile compounds were done on DB-WAX or DB-5MS fused-silica capillary columns (60 m × 0.32 mm i.d. × 0.25 μm film thickness; J&W Scientific). The purge and trap system was cleaned by purging with clean glassware installed, and the Tenax trap was subsequently baked at 225 °C for 10 min. The GC oven temperature was programmed from 40 to 200 °C at a rate of 8 °C/min (DB-WAX) or 6 °C/min (DB-5MS) with initial and final hold times of 5 and 30 min, respectively. The FID and the transfer lines were maintained at 250 °C. The sniffing port was supplied with humidified air (30 mL/min). GCO−DHS sniffing was performed by two trained panelists for each purge volume.

Direct Solvent Extraction (DSE). The procedure for DSE was adapted from Guth and Grosch (*24*). Cooked lobster tail meat (25 g) was frozen in liquid nitrogen prior to grinding into powder. Ground lobster tail meat and anhydrous sodium sulfate (25 g) were mixed and then saturated with dichloromethane. The mixture was kept at 4 °C overnight and then centrifuged (1017*g*, 10 min) before collecting the solvent layer. Extraction was repeated twice as delineated above, except that soaking time was reduced to 1 h. The combined extract was subjected to high vacuum transfer (ca 10^{-5} Torr) at room temperature for 30 min and then at 60 °C for an additional 3.5 h in an apparatus illustrated by Suriyaphan and co-workers (*25*). The distillate was stored at −20 °C overnight and then passed through anhydrous sodium sulfate. The volume of extract was reduced to 30 μL under a gentle stream of nitrogen gas. The concentrated extract was stored at −20 °C in a vial equipped with a Teflon-lined cap until performing AEDA. Two DSE extracts were prepared.

Vacuum Simultaneous Steam Distillation−Solvent Extraction (VSDE). Cooked lobster tail meat (1 kg) plus odorless water (1.5 L) and 2,4,6-trimethyl pyridine (20 μg, TMP, internal standard) was extracted for 4 h with dichloromethane (75 mL) under reduced pressure (ca 25 mTorr) in a modified SDE apparatus as described by Chung and Cadwallader (*6*). The temperature of the sample during distillation was maintained at 55−60 °C. The obtained flavor extract was stored at −20 °C overnight, dried over anhydrous sodium sulfate, and then concentrated to 1 mL under a gentle stream of nitrogen. The concentrated extract was stored at −20 °C in a vial equipped with a Teflon-lined cap until analysis. VDSE extracts were prepared in triplicate.

Aroma Extract Dilution Analysis (AEDA). General procedures for AEDA have been previously described (*19*). Serial dilutions (1:3) of extracts (DSE and VSDE) were prepared using dichloromethane. Each dilution was transferred to a 2-mL amber vial containing a 200-μL glass insert and sealed with a Teflon-lined screw cap. Dilutions were kept at −20 °C until analyzed. DSE dilutions (1 μL) were injected (cool on-column mode) into the GCO system, as described above in GCO−DHS. For DSE, GCO was performed by one trained panelist. For VSDE, the conditions for AEDA and GCO were same as those for DSE except that sniffing was performed by three trained panelists. Each panelist performed GCO on duplicate sets of dilution series. Flavor dilution (FD) factors for each odorant were assigned on the basis of the highest dilution at which it was last detected by GCO. Results are expressed as arithmetic means of $\log_3$ (FD factors) (*6*).

Gas Chromatography−Mass Spectrometry (GC−MS). GC−MS was conducted using an HP5890 Series II GC/HP5972 mass selective detector system (Hewlett-Packard Co., Palo Alto, CA) equipped with a DB-WAX or DB-5MS column (60 m length × 0.25 mm. i.d × 25 μm film thickness; J&W Scientific). GC conditions were the same as those described for AEDA or GCO−DHS except that the oven ramp rate was 2 °C/min. MSD capillary direct-interface temperature was 280 °C, ionization energy was 70 eV, mass range was 33−350 a.m.u, electron multiplier (EM) voltage was 200V above Autotune value, and scan rate was 2.2 scans/s.

Compounds Identification. Compound identification was based on comparison of GC retention indices (RI) (*26*), mass spectra, and odor properties with those of reference compounds under identical analytical conditions. Tentative identifications were based on comparison of RI and odor properties with reference compounds.

Quantitative Analysis. Calibration curves of amount ratios (compound/internal standard) versus peak area ratios (compound/internal standard) were generated under experimental conditions identical to those of VSDE except that the authentic standards were added instead of the sample. Compounds in low abundance were quantified by mass chromatography (*27*).

Table 1. Minimum Headspace Injection Volumes Required for Detection of Aroma-Active Components by Gas Chromatography−Olfactometry of Decreasing Static Headspace Samples

no.[a]	compound	odor quality[b]	RI[c] on DB-WAX	RI[c] on DB-5MS	volume[d] (mL)
1	hydrogen sulfide[e]	rotten egg	<600	<500	5
2	trimethylamine	fish house, crabby	609	<500	5
3	methanethiol	rotten, sulfurous	618	<500	10
4	acetaldehyde	sweet, solvent, ethanolic	655	<500	10
6	dimethyl sulfide	canned corn, sulfurous	703	<500	5
7	2-methylpropanal	dark chocolate, malty	855	537	20
9	3-methylbutanal	dark chocolate, malty	932	651	5
10	2,3-butanedione	buttery	981	606	5
20	1-octen-3-one	mushroom	1296	979	10
21	2-acetyl-1-pyrroline[e]	popcorn	1333	927	10
23	dimethyltrisulfide	cabbage	1381	970	10
26	3-(methylthio)propanal	cooked potato	1456	907	5
30	(*E*,*Z*)−2,6-nonadienal[e]	cucumber	1593	1153	5

[a] Numbers correspond to those in Tables 2−5. [b] Odor quality perceived by panelist during GCO. [c] Retention indices calculated from GCO results. [d] Minimum static headspace volume required for detection by at least one assessor during GCO. [e] MS signal too weak to interpret; compound identified by comparing its RI (or retention time) value on two capillary columns and its aroma properties with those of a reference compound.

RESULTS AND DISCUSSION

GC−Olfactometry of Decreasing Headspace Samples (GCO−H). GCO−H revealed a total of 13 aroma-active compounds in cooked American lobster tail meat (Table 1). These compounds have been previously reported in cooked crustaceans (*2*, *3*, *6*, *8*, *11*, *28*). GCO−H was performed at different headspace injection volumes (20, 10, or 5 mL), and the most intense aroma contributors were those compounds detected at the lowest headspace injection volume. The most potent odorants consisted of hydrogen sulfide (**1**, rotten egg), trimethylamine (**2**, fish house, crabby), dimethyl sulfide (**6**, canned corn), 3-methylbutanal (**9,** dark chocolate, malty), 2,3-butanedione (**10**, buttery), 3-(methylthio)-propanal (**26**, cooked potato) and (*E*,*Z*)-2,6-nonadienal (**30**, cucumber). In addition, methanethiol (**3**, rotten, sulfurous), acetaldehyde (**4**, sweet, solvent, ethanolic), 1-octen-3-one (**20**, mushroom), 2-acetyl-1-pyrroline (**21**, popcorn), and dimethyltrisulfide (**23**, cabbage) were classified as moderately potent odor compounds, while 2-methylpropanal (**7**, dark chocolate, malty) was regarded as a less potent odor compound. Because of their very high volatility, early eluting odorants (**1**−**4**, **6**, **7**, **9**, and **10**) may be especially important in contributing the first recognition of cooked lobster aroma. Other odorants may be more important in the aroma impression perceived during mastication. However, odorants **4**, **7**, **9**,**10**, **21**, and **26** were of particular interest because their odor properties were in agreement with the typical nutty, roasty, popcorn-like aroma of cooked lobster tail meat. 3-Methylbutanal (**9**) was previously reported as a predominant aroma component of cooked crabmeat (*6*). Furthermore, 2,3-butanedione (**10**), 1-octen-3-one (**20**), 2-acetyl-1-pyrroline (**21**), and 3-(methylthio)propanal (**26**) were found to be important aroma constituents in cooked meat from blue crab (*6*), spiny lobster (*2*), and brown shrimp (*28*). In addition, the compound 2,3-butanedione (**10**) was known to contribute buttery notes to the aroma of Louisiana red swamp crayfish tail meat (*5*). 2-Acetyl-1-pyrroline (**21**) has also been identified as a character-impact aroma component of aromatic rice (*29*), and wheat and rye bread crust (*30*). Recently, Kim and co-workers (*10*) reported that 2-acetyl-1-pyrroline (**21**) and 3-(methylthio)propanal (**26**) were the most potent odorants in enzyme-hydrolyzed concentrated oyster-cooker effluent. The presence of 1-octen-3-one (**20**, mushroom) could have a negative impact on the overall aroma of cooked lobster. This compound has been reported to contribute off-flavors to the aroma of fresh whitefish (*31*) and alligator meat (*32*). Additionally, a compound having a similar odor quality, 1-octen-3-ol, was reported by Whitfield and co-workers (*16*) as causing a mushroom, metallic off-flavor in prawns and sand lobsters. The compound 3-(methylthio)propanal (**27**) has been considered to be an important component in basic meat flavor (*5*, *20*).

It is well-documented that Maillard and Strecker degradation reactions play important roles in formation of the meaty aroma of cooked seafood (*4*). Acetadehyde (**4**) could be formed via either Strecker degradation of alanine, cysteine, or cystine (*33*), or via fragmentation of deoxyhexosones through Maillard reaction (*34*). In addition, 2-methylpropanal (**7**) and 3-methylbutanal (**9**) are well-known as Strecker aldehydes and are derived from valine and leucine, respectively (*35*). In addition, Griffith and Hammond (*36*) revealed that Strecker degradation products of methionine included methanethiol (**3**), dimethyl sulfide (**6**), dimethyltrisulfide (**23**), and 3-(methylthio)propanal (**26**). However, in seafood dimethyl sulfide (**6**) is usually formed via thermal degradation of dimethyl-β-propriothetin (*37*). Compounds 2,3-butanedione (**10**) and 2-acetyl-1-pyrroline (**21**) may have been thermally generated through the *Maillard* reaction (*30*, *38*). 1-Octen-3-one (**20**) and (*E*,*Z*)-2,6-nonadienal (**30**) have been shown to be enzymatically derived from omega-3 fatty acids (*39*).

In the present work, several potentially malodorous compounds (**1**, **2**, **3**, **6**, and **23**) were also found. These compounds could be responsible for either characteristic seafood aroma or off-flavors (*40*). Trimethylamine (**2,** fish house**,** crabby) was seemingly an important odorant because at low levels it contributed a typical crab-like note to lobster meat (*2*). This compound (**2**) could be a thermal decomposition product of choline, betaine, methionine (*41*), or most likely, trimethyamine oxide (*42*) during cooking. Methanethiol (**3**) was identified as an odor-active component of stored cod (*21*) and cooked brown shrimp (*28*), as well as stewed beef juice (*20*). Nonetheless, the presence of high levels of odorants possessing putrid notes (e.g., compounds **1** and **3**) generally has a negative impact on foods (*43*). Other sulfur-containing compounds (**6** and **23**) might also negatively impact the aroma of cooked lobster because of their extremely low threshold values (*44*). Both

Table 2. Minimum Purge Gas Volumes Required for Detection of Aroma-Active Components by Gas Chromatography−Olfactometry of Decreasing Dynamic Headspace Samples

no.[a]	compound	odor quality[b]	RI[c] on DB-WAX	RI[c] on DB-5MS	volume[d] (mL)
1	hydrogen sulfide[e]	rotten egg	<600	<500	10
2	trimethylamine	fish house, crabby	609	<500	10
3	methanethiol	rotten, sulfurous	618	<500	10
4	acetaldehyde	sweet, solvent, ethanolic	655	<500	10
5	unknown	rotten, fishy	691	<500	250
6	dimethyl sulfide	canned corn, sulfurous	703	<500	10
7	2-methylpropanal	dark chocolate, malty	855	537	250
9	3-methylbutanal	dark chocolate, malty	932	651	10
10	2,3-butanedione	buttery	981	606	10
11	hexanal	green, grassy	1083	808	50
12	1-hexen-3-one[e]	plastic, water bottle	1096	784	10
17	(*Z*)-4-heptenal	rancid, fishy	1246	900	10
19	octanal	sweet, wine-like	1263	1005	50
20	1-octen-3-one	mushroom	1296	979	10
21	2-acetyl-1-pyrroline[e]	popcorn	1333	927	10
22	(*Z*)-1,5-octadien-3-one[e]	metallic, mushroom	1374	982	10
23	dimethyltrisulfide	cabbage	1381	970	10
25	(*E*)-2-octenal	nutty, raw peanut, stale	1430	1045	50
26	3-(methylthio)propanal	cooked potato	1456	907	10
30	(*E*,*Z*)-2,6-nonadienal[e]	cucumber	1593	1153	250

[a] Numbers correspond to those in Tables 1 and 3−5. [b] Odor quality as perceived by panelist during GCO. [c] Retention indices calculated from GCO results. [d] Minimum purge gas volume required for detection by at least one assessor during GCO. [e] MS signal too weak to interpret; compound identified by comparing its RI or retention time values on two capillary columns and its aroma properties with those of a reference compound.

compounds were found to impart an onion-like off-flavor in prawn (*45*) and spoiled odor in crayfish meat (*5*).

GC−Olfactometry of Dynamic Headspace Samples (GCO−DHS). Aroma-active compounds detected by GCO−DHS are shown in Table 2. Among these, seven odorants (**5**, **11**, **12**, **17**, **19**, **22**, and **25**) were not found during GCO−H indicating that they might be present at low levels. Of these seven, six lipid-derived compounds were identified, including hexanal (**11**, green, grassy), 1-hexen-3-one (**12**, plastic, water bottle), (*Z*)-4-heptenal (**17**, rancid, fishy), octanal (**19**, sweet, wine-like), (*Z*)-1,5-octadien-3-one (**22**, metallic, mushroom), and (*E*)-2-octenal (**25**, nutty, raw peanut, stale). Compound no. **5** (rotten, fish) was not identified. Malodorous compounds **5**, **11**, **12**, **17**, and **22** might have a negative impact on the aroma of cooked American lobster tail meat. For instance, hexanal (**11**) was a major aroma constituent of warmed-over flavor in meats (*46*). 1-Hexen-3-one (**12**) was previously identified in light oxidized milk and imparted a plastic, water bottle-like odor (*47*). In addition, (*Z*)-4-heptenal (**17**) was found to be responsible for an off-odor in cold stored cod (*48*) and in alligator meat (*32*). Moreover, (*Z*)-1,5-octadien-3-one (**22**) provided a metallic off-odor described as very heavy green, geranium leaf-like to seafood and fish (*39*).

The relative potency of each aroma-active compound was determined by GCO−DHS with decreasing purge gas volumes (250, 50, or 10 mL). On the basis of their low purge-gas volume of 10 mL, compounds **1**−**4**, **6**, **9, 10**, **12**, **17**, **20**−**23**, and **26** were considered the most potent aroma components. Similarly, compounds **11**, **19**, and **25** were regarded as moderately potent aroma components, and compounds **5**, **7**, and **30** were less potent aroma components. The ranking of relative potency of these odorants by GCO−DHS and by GCO−H was similar. Hexanal (**11**) has been shown to be an important constituent contributing grassy, apple-like aroma in fresh whitefish (*49*). This compound was derived from n-3 or n-6 polyunsaturated fatty acid by lipoxygenase (*50*). In general, (*Z*)-4-heptenal (**17**) was considered to be a potentially dominating aroma contribution because of its very low odor-detection threshold (0.04 ppb) (*51*). This compound (**17**) was reported as a potent aroma compound in cooked Spiny lobster (*2*). Meanwhile, octanal (**19**) and (*E*)-2-octenal (**25**) were reported as the predominant compounds in steamed clam flavor (*52*). (*E*)-2-Octenal (**25**) also imparted a desirable aroma of cooked tail meat of brown shrimp (*28*). (*Z*)-4-Heptenal (**17**) and (*E*)-2-octenal (**25**) were shown to be readily converted from (*E*,*Z*)-2,6-nonadienal (**30**) and (*E*,*E*)-2,4-decadienal (**40**), respectively, via water-mediated retro-aldol reactions (*53*). The vinyl ketone, (*Z*)-1,5-octadien-3-one (**22**), was generated from unsaturated lipid in fish during heating (*54*).

Aroma Extract Dilution Analysis of Direct Solvent Extracts (AEDA−DSE). To gain a better understanding of contributions made by components of intermediate and low volatility to the typical aroma of cooked lobster meat, we applied aroma extract dilution analysis (AEDA) on DSE extracts (AEDA−DSE). In addition, dilutions were injected in the cool on-column mode to minimize the formation of artifacts, which could occur in a high-temperature injection port. In total, 32 odorants were detected (Table 3). Of these, seven compounds were not identified because their MS responses were too weak or because corresponding MS reference spectra were unavailable. Among the 25 identified compounds, 13 (**2**, **9**−**12**, **17**, **19**−**23**, **26**, and **30**) were previously detected during GCO−DHS. However, several potent aroma compounds found by GCO−H or GCO−DHS were not detected during AEDA−DSE because of their high volatility; for example, hydrogen sulfide (**1**), methanethiol (**3**), and 2,3-butanedione (**4**). These compounds may have been lost during concentration of the extract prior to analysis or possibly coeluted with the solvent during GCO.

On the basis of its highest $\log_3$FD-factor of 6, the most intense odor was (*Z*)-1,5-octadien-3-one (**22**). The compound with the second highest $\log_3$FD-factor of 5 was 3-(methylthio)propanal (**26**). These results were in good

Table 3. Predominant Odorants in Volatile Flavor Extracts Prepared by Direct Solvent Extraction−High Vacuum Distillation

no.[a]	compound	odor quality[b]	RI[c] on DB-WAX	RI[c] on DB-5MS	average log_3FD factor[d]
2	trimethylamine[e]	fish house, crabby	609	<500	1
8	2-methylbutanal	dark chocolate, malty	855	537	1
9	3-methylbutanal	dark chocolate, malty	932	651	1
10	2,3-butanedione	buttery	981	606	1
11	hexanal	green, grassy	1083	808	2
12	1-hexen-3-one[e]	plastic, water bottle	1096	784	1
13	unknown	crabby, amine, fishy	1161	823	1
14	unknown	crabby, amine	1191	884	4
15	2-methyl-3-furanthiol	vitamin, meaty	- -[f]	871	2
16	unknown	crabby, amine, fishy	1234	877	<1
17	(*Z*)-4-heptenal	rancid, fishy	1246	900	2
19	octanal	sweet, wine-like	1263	1005	<1
20	1-octen-3-one	mushroom	1296	979	3
21	2-acetyl-1-pyrroline[e]	popcorn	1333	927	1
22	(*Z*)-1,5-octadien-3-one[e]	metallic, mushroom	1374	982	6
23	dimethyltrisulfide[e]	cabbage	1381	970	2
24	trimethylpyrazine[e]	popcorn, roasted	1421	1020	2
26	3-(methylthio)propanal	cooked potato	1456	907	5
27	(*E*)-2-nonenal[e]	stale, hay	1528	1163	2
28	unknown	potato, musty	1553	1004	<1
29	2-acetylpyridine	popcorn	1586	1046	1
30	(*E*,*Z*)-2,6-nonadienal[e]	cucumber	1593	1153	3
31	2-acetylthiazole	popcorn	1596	1023	1
32	(*Z*,*Z*)-3,6-nonadienal[e]	watermelon	- -	1097	2
33	unknown	burnt sugar	- -	1083	4
35	unknown	fresh fish skin, green	1718	1241	3
36	unknown	crabby, amine, fishy	1742	1158	4
38	beta-damascenone[e]	tea, applesauce	1827	1384	2
39	2-methoxyphenol[e]	smoky	1872	1089	<1
40	(*E*,*E*)-2,4-decadienal[e]	fatty, fried	1883	1275	2
41	benzothiazole	rubbery	1984	1234	1
43	2,5-dimethyl-4-hydroxy-3(2H)-furanone[e]	strawberry, burnt sugar	2060	1061	3

[a] Numbers correspond to those in Tables 1, 2, 4, and 5. [b] Odor quality perceived during GCO. [c] Retention indices calculated from GCO data. [d] Average log_3 flavor dilution factor on DB-5MS column ($n = 2$). [e] MS signal too weak to interpret; compound identified by comparing its RI values on two capillary columns and its aroma properties with those of a reference compound. [f] Compound was not detected on DB-WAX column.

agreement with those of GCO−DHS. A group of odorants with the third highest log_3FD-factors of 4 consisted of three unknowns which possessed crabby, amine, fishy odors (**14 and 36**), and a burnt sugar note (**33**). Odorants having the log_3FD-factors of 3 included 1-octen-3-one (**20**), (*E*,*Z*)-2,6-nonadienal (**30**), unknown (**35**, fresh fish skin, green), and 2,5-dimethyl-4-hydroxy-3(2H)-furanone (DMHF, **43**, strawberry, burnt sugar). DMHF was previous identified as an essential component of beef aroma (*20*, *55*).

Nine odorants (**11**, **15**, **17**, **23**, **24**, **27**, **32**, **38**, and **40**) were detected with log_3FD-factors of 2. Within this group, 2-methyl-3-furanthiol (**15**, meaty, vitamin-like) was regarded as an important aroma contributor providing a desirable meaty note to cooked lobster, since it was reported earlier to impart a strong meaty, beef extract-like odor to roasted shrimp (*56*) and canned tuna (*57*). Gasser and Grosch (*58*) proposed that 2-methyl-3-furanthiol (**15**) was unstable on polar stationary phases, such as DB-WAX. This may explain why this compound (**15**) was detected only when GCO was conducted on the nonpolar DB-5MS column. Trimethylpyrazine (**24**, popcorn, roasted) was regarded as having a positive impact on the overall aroma of cooked lobster tail meat because pyrazines are known to impart desirable aromas (nutty, roasted, toasted-like) to heated processed food products (*43*). Alkylpyrazines are normally generated through Maillard and pyrolysis reactions (*59*). (*E*,*E*)-2,4-Decadienal (**40**, fatty, fried) was an oxidation product of linoleic acid and was also known as a potent aroma compound of deep-fried food products (*60*).

The majority of the odorants detected in DSE extract had log_3FD-factors of 1 (**2**, **8**−**10**, **12**, **13**, **21**, **29**, **31**, and **41**). One unknown odorant (**13**) in this group was described as having a crabby, amine, fishy odor. Although 2-acetylthiazole (**31**, popcorn), and 2-acetylpyridine (**29**, popcorn) were identified in this low intensity group, they were considered important aroma components because of their desirable odor qualities. In addition, 2-acetylthiazole (**31**) was previously identified as an aroma component of cooked meat of Spiny lobster (*2*) and tiger prawn (*40*). This compound was shown to be thermally generated from cysteine (*61*). At a very low concentration, benzothiazole (**41**, rubbery) was reported to impart the desirable flavor of roasted meat (*40*). Zhang and Ho (*62*) postulated that this compound was a Strecker degradation product of cysteine and glucose. The last group of odorants in DSE extracts contained octanal (**19**), 2-methoxyphenol (guaiacol, **39,** smoky), and two unknowns that were detected in the undiluted extract only. Other unidentified compounds imparted crabby, amine, fishy (**16**) and potato, musty (**28**) odors.

Aroma Extract Dilution Analysis of Vacuum Simultaneous Steam Distillation−Solvent Extracts (AEDA−VSDE). Twenty-seven odorants were detected by AEDA of extracts prepared by VSDE (Table 4). Most of these compounds were previously identified during GCO−H, GCO−DHS, and AEDA−DSE. How-

Table 4. Predominant Odorants in Aroma Extracts Prepared by Vacuum Simultaneous Steam Distillation−Solvent Extraction

no.[a]	compound	odor quality[b]	RI[c] on DB-WAX	RI[c] on DB-5MS	average log_3FD-factor[d]
9	3-methylbutanal	dark chocolate, malty	932	651	1
10	2,3-butanedione	buttery	981	606	2
11	hexanal	green, grassy	1083	808	1.3
12	1-hexen-3-one[e]	plastic, water bottle	1096	784	1
13	unknown	crabby, amine, fishy	1161	823	4
14	unknown	crabby, amine	1191	884	6
17	(Z)−4-heptenal	rancid, fishy	1246	900	1
18	unknown	onion, sour, sulfurous	1253	682	1
20	1-octen-3-one	mushroom	1296	979	1
21	2-acetyl-1-pyrroline[e]	popcorn	1333	927	4
22	(Z)−1,5-octadien-3-one[e]	metallic, mushroom	1374	982	1
24	trimethylpyrazine[e]	popcorn, roasted	1421	1020	3
25	(E)−2-octenal	nutty, raw peanut, stale	1430	1045	3
26	3-(methylthio)propanal	cooked potato	1456	907	5.3
28	unknown	potato, musty	1553	1004	4
29	2-acetylpyridine	popcorn	1586	1046	5
30	(E,Z)−2,6-nonadienal[e]	cucumber	1593	1153	2
31	2-acetylthiazole	popcorn	1596	1023	3
34	unknown	crabby, amine, fishy	1683	1204	3
36	unknown	crabby, amine, fishy	1742	1158	4
37	unknown	crab shell, stale, hay, nutty	1775	1137	3
40	(E,E)−2,4-decadienal[e]	fatty, fried	1883	1275	3
42	unknown	crabby, amine, fishy	2000	1283	2
44	unknown	leather, musty, marine	2171	1329	1
45	o-aminoacetophenone[e]	grape, rubbery	2270	1310	1
46	unknown	leather, musty	2347	1493	1
47	3-methylindole	fecal, pungent	2505	1401	4

[a] Numbers correspond to those in Tables 1−3, and 5. [b] Odor quality perceived during GCO. [c] Retention indices calculated from GCO data. [d] Average log_3 flavor dilution factor on DB-WAX column ($n = 6$). [e] MS signal too weak to interpret; compound identified by comparing its RI values on two capillary columns and its aroma properties with those of a reference compound.

Table 5. Concentrations and Odor-Activity Values for Selected Odorants in Flavor Extracts Prepared by Vacuum Simultaneous Steam Distillation−Solvent Extraction

no.[a]	compound	concentration[b] (ng/g)	odor threshold[c] (ng/g)	odor-activity value[d]
10	2,3-butanedione	96 ± 13	2.6[e]	37
11	hexanal	28 ± 8	5[f]	6
17	(Z)−4-heptenal	6 ± 2	0.04[g]	143
26	3-(methylthio)propanal	298 ± 36	0.2[h]	1490
31	2-acetylthiazole	20 ± 2	10[i]	2

[a] Numbers correspond to those in Tables 1−4. [b] Average concentration standard deviation (from VSDE−GC−MS, $n = 3$). [c] Odor threshold in water. [d] Odor-activity value calculated by dividing compound concentration by its odor detection threshold. [e] Ref 65. [f] Ref 29. [g] Ref 48. [h] Ref 66. [i] Ref 67.

ever, there were 8 odorants detected only by AEDA−VSDE including 6 unknowns (**18**, **34**, **37**, **42**, **44**, and **46**), *o*-aminoacetophenone (**45**, grape, rubbery) and 3-methylindole (skatole**, 47**, fecal, pungent). Because of their odor qualities, *o*-aminoacetophenone (**45**) and 3-methylindole (**47**) may have negative affects on the overall aroma of cooked lobster tail meat. *o*-Aminoacetophenone (**45**) contributed a stale flavor in milk products (*63*) and an off-flavor in fermented tuna sauce (*64*).

Results of AEDA−VSDE were slightly different from those of AEDA−DSE. The most potent odorants (average log_3 FD-factor ≥ 5) belonged to an unknown (**14**, crabby, amine), 3-(methylthio)propanal (**26**), and 2-acetylpyridine (**29**). The second most potent group of odorants (average log_3FD-factors of 4) included three unknowns (**13**, **28**, and **36**), 2-acetyl-1-pyrroline (**21**), and 3-methylindole (**47**). Odorants with average log_3FD-factors of 3 consisted of two unknowns (**34** and **37**), as well as trimethypyrazine (**24**), (*E*)-2-octenal (**25**), 2-acetylthiazole (**31**), and (*E*,*E*)-2,4-decadienal (**40**). The other two unknown compounds in this group imparted crabby, amine, fishy (**34**) and crab shell, hay, stale, nutty (**37**) odors. Compounds with average log_3FD-factors close to 2 were 2,3-butanedione (**10**), (*E*,*Z*)-2,6-nonadienal (**30**), and one unknown (**42**). Less potent compounds (average log_3FD-factors close to 1) consisted of odorants **9**, **11**, **12**, **17**, **18**, **20**, **22**, and **44**−**46**.

In summary, a total of 47 odorants were detected by the four isolation methods. These compounds can be categorized by their aroma characteristics into seven groups. The first group was represented by odorants (**13**, **14**, **34**, **36,** and **42**) with crabby, amine, and fishy notes. These unknowns were mostly detected by AEDA−VSDE and AEDA−DSE at high log_3FD factors (Table 4) and are, therefore, believed to be important in the overall aroma of cooked lobster tail meat. A second group consisted of odorants with popcorn and roasted notes (**21**, **24**, **29**, and **31**). Compounds with potato-like notes (**26** and **28**) comprised the third group of odorants. The fourth group was represented by buttery and malty, dark chocolate odors (**7**, **9**, and **10**). The fifth group contained the lipid-derived compounds (**11**, **12**, **19**, **20**, **22**, **25**, **30**, **32**, and **40**) that were present at low or intermediate intensities. A sixth group of malodorous compounds (**1**, **2**, **3**, and **23**), described as rancid, rotten, and cabbage-like, was detected at low or medium odor

intensities. The seventh group consisted of the remaining odorants with widely varying aroma properties.

It is worth mentioning that there were several compounds previously reported as key constituents of seafood flavors that were found at relatively high level in VSDE extracts but were not detected by GCO (data not shown). These included benzaldehyde, 2,3-pentanedione, 1-octen-3-ol, 1H-pyrrole, 2,5-dimethypyrazine, 2,6-dimethylpyrazine, and 2-acetyl-3-methylpyrazine.

Quantification of Selected Odorants. Quantification of selected potent odorants was conducted to obtain a better understanding of the actual contribution of individual compounds to the overall aroma based on odor-activity values. Quantitative analysis was performed by GC−MS of VSDE extracts because this isolation method gave the highest overall recovery of the volatile constituents. Among the quantified odorants 3-(methylthio)propanal (**26**) had the highest odor-activity value (Table 5). This result is supported by the results of AEDA (DSE and VSDE) and GCO−DHS and GCO−H. Despite its lower abundance, (*Z*)-4-heptenal (**17**) had a higher odor-activity value than hexanal (**11**), 2,3-butanedione (**10**), and 2-acetylthiazole (**31**). This can be attributed to the much lower odor threshold of (*Z*)-4-heptenal (**17**) compared with that of these other odorants (Table 5). Nonetheless, we were unable to quantify several key odorants, e.g. 2-acetyl-1-pyrroline (**21**) and 1-octen-3-one (**20**), as they were present at levels below GC−MS detection limits. These data, however, indicated that results of AEDA and of GCO−DHS and GCO−H seem reasonable and comparable.

CONCLUSIONS

GCO results using four complementary isolation methods, namely SHS, DHS, DSE, and VSDE, coupled with dilution analysis techniques provided essential information for the identification of predominant odorants in cooked lobster tail meat. Thirteen and twenty odorants were detected during GCO−H and GCO−DHS, respectively. On the basis of their frequency of detection at high odor intensities, 3-methylbutanal, 2,3-butanedione, (*Z*)-heptenal, 3-(methylthio)propanal, 1-octen-3-one, 2-acetyl-1-pyrroline, (*Z*)-1,5-octadien-3-one, and (*E*,*Z*)-2,6-nonadienal were determined to be key components of the aroma of cooked American lobster tail meat. In addition, several unknown compounds that possessed crabby, amine, fishy notes also should be considered as important aroma constituents because of their high FD-factors.

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