GC/MS Analysis of Flavor-Active Compounds in Cooked Commercial Shrimp Waste

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Crustacean waste generated from the fishing industry represents approximately 70% of the total landings. This abundant waste may pose an environmental hazard due to the ease of deterioration of the fish tissue in the landfill sites; disposing of the waste, therefore, can be achieved at considerable cost to the industry. Alternatively, the waste can be utilized by extracting useful components such as flavor-active compounds and incorporating them in desirable seafood products. The flavor profile of the cooked shrimp waste was determined by GC/MS analysis. The results revealed the presence of 44 compounds of different functional groups including fatty acid esters, long-chain alcohols, aldehydes, and heterocyclic compounds. Twenty-nine compounds were tentatively identified by their mass spectral data, and 15 were identified by both mass spectral data and retention times. Many components provided desirable aroma (nutty, fruity, floral, green woody, meaty), indicating the presence of important flavor compounds in the commercial shrimp waste.

INTRODUCTION

The worldwide production and processing of shrimp represents an industry valued at several hundred millions of dollars. The shellfish industry (shrimp, oyster, and lobster) as a whole is faced with an increasing dilemma due to recent enforcement of pollution laws which now prohibits the disposal of shellfish waste into the ocean or in landfill dumping sites. A better economic use of the shellfish offal would minimize the pollution problem and at the same time maximize the profits of the processor. One method of using shellfish wastes would be its conversion into "valued added" products by extracting "active" components from the waste and using them as useful marketable products. Numerous investigators (Hayashi et al., 1981; Kato et al., 1989; Voight et al., 1990) have identified the primary flavor-active components of shellfish as being umami (free amino acids and sugars, nucleotides, inorganic salts, and betaine). In the same trend, previous work in this laboratory (Mandeville et al., 1991a,b, 1992) permitted the isolation of free amino acids and sugars, lipids, pigments, and flavor-active compounds from raw commercial shrimp waste. The aim of these studies was not only to isolate valuable marketable products but also to identify the Maillard reaction precursors present in the waste. In this study we report the GC/MS analysis of flavor-active compounds found in cooked shrimp waste.

EXPERIMENTAL PROCEDURES

Materials and Methods. Commercial frozen raw shrimp waste was obtained from a local supplier. The shrimp was harvested from the Gulf of Mexico, during the month of July, and the waste was frozen within 20 min of processing the shrimp. The frozen waste was obtained the next day. Proximate analysis (Mandeville et al., 1992) indicated a moisture content of 82.58 ± 0.89% (n = 12) and a high content of arginine, indicating the freshness of the waste.

The samples were thawed followed by removal of any shell-like residue (i.e., tail, claw, etc.) and then cut into small pieces and homogenized using a Restch (Brickman) grinder. The homogenized samples were frozen and kept at −80 °C until further use. All of the chemicals were purchased from Sigma and used without further purification; all of the solvents were of HPLC grade (BDH, Anachemia, or Champlain). Water was obtained from a Milli-Q reagent grade water system (Millipore Corp., Bedford, MA). Fatty acid methyl ester standards were obtained from Nu Chek Prep (Elysian, MN), whereas aliphatic alkane, alcohol, and aldehyde standards were obtained from PolyScience Corp. (Niles, IL). The shrimp waste was cooked by refluxing 500 g of the waste in a 2-L round-bottom flask fitted with a reflux condenser, containing 500 mL of water, for 3 h. After the reflux, the content of the flask was filtered, and the residue and the filtrate were extracted and separated into acidic, basic, and neutral fractions as described previously (Mandeville et al., 1991a). All fractions obtained from the meat residue were termed **methanolic**, whereas those obtained from the filtrate were termed **aqueous**.

Extraction and Separation of the Flavor Compounds into Acidic, Basic, and Neutral Fractions. The thawed shrimp waste samples were mixed with acetone and placed into a homogenizer (Virtis Model 23) set at a speed of 4/10 for 30 s. This procedure was repeated four times until all of the orange pigments were removed. The white residue was then treated with

Table I. Yield and Odor Description of Flavor Extracts from Boiled Commercial Shrimp Waste

fraction	yield mg[a]	yield average[b]	yield %[c]	odor description
whole concentrate	2794[d]		100	
methanolic				
basic	834.5	278.1 ± 22.2	29.9	fresh shrimp, fruity, fatty-like
acidic	138.5	46.1 ± 3.4	4.9	slight boiled, woody, green-like
neutral	834.5	278.2 ± 23.4	29.9	fresh and cooked shrimp, wood-like
aqueous				
basic	436.5	145.5 ± 15.3	15.6	nutty, boiled shrimp, fruity
acidic	75.0	24.9 ± 1.4	2.7	woody, floral, slightly rancid
neutral	475.0	158.3 ± 11.0	17.0	woody, floral

[a] From 1.5 kg of shrimp waste (sum of triplicate extractions). [b] Average of triplicate extractions. [c] From the whole concentrate. [d] 1.10% on a dry weight basis.

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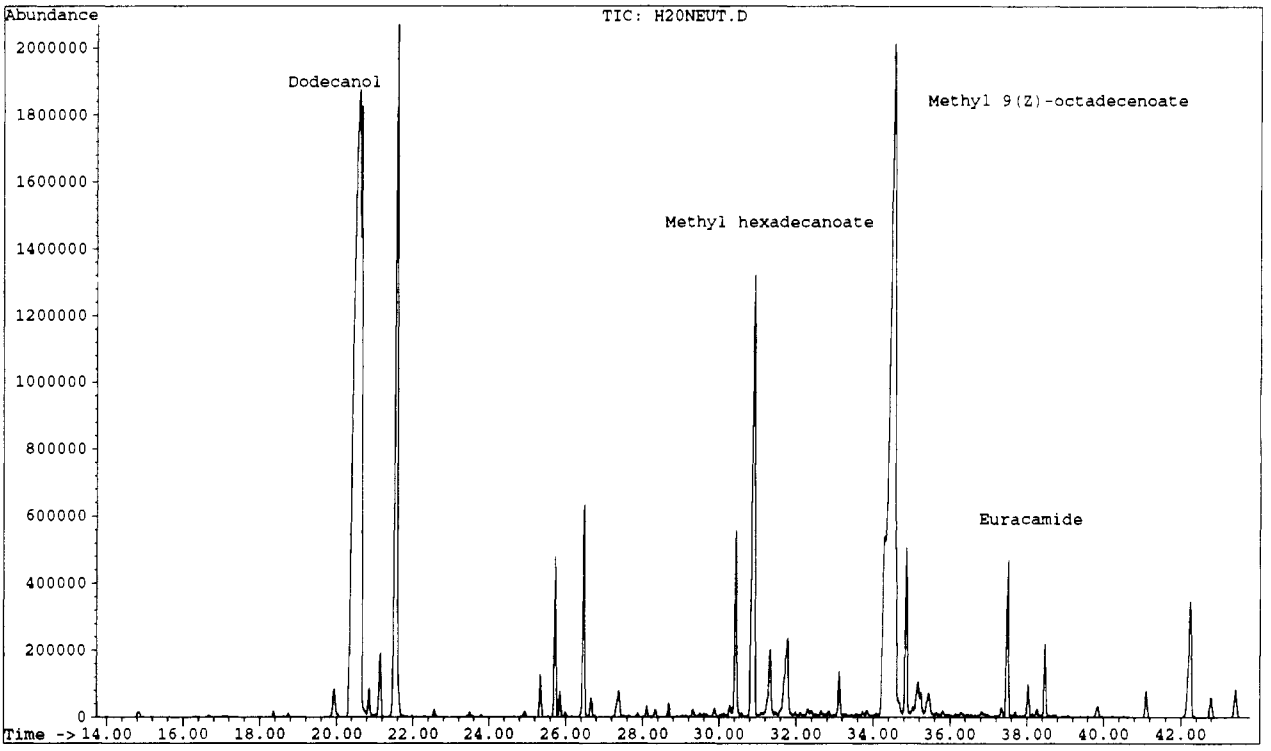

Figure 1. Partial chromatogram of aqueous neutral fraction of the shrimp waste extract injected in a splitless mode into a fused silica capillary column (DB-5, 30 m × 0.32 mm i.d. × 0.25 μm film thickness). The initial column temperature (60 °C) was increased from 60 to 70 °C at a 10 °C/min rate for 1.0 min and maintained at 70 °C for 0.5 min and increased from 70 to 250 °C at a 5 °C/min and maintained at 250 °C for 12 min. Carrier gas (helium) flow rate was 0.85 cm^3/min; injection port temperature was 200 °C.

chloroform/methanol (2:1 v/v) to remove the remaining lipids. The remaining white residue was then extracted with methanol to obtain the flavor compounds.

The methanol extract from the above procedure was concentrated under N_2 at room temperature using a Rotovap evaporator; the residue (intense yellow in color) was then suspended in diethyl ether (75 mL) and extracted with 1 N HCl (75 mL). The diethyl ether layer which contained neutral and acidic components was termed organic fraction 1. The aqueous phase was basified with 2 N NaOH to a pH of 8.5 and then extracted with diethyl ether. The organic fraction 1 was extracted with 6% sodium bicarbonate ($NaHCO_3$) solution and separated into aqueous and organic phases. The organic phase which now contains the neutral compounds was termed organic fraction 2. The aqueous phase was then acidified with 2 N HCl to pH 5.5 and extracted with diethyl ether. The neutral compounds were obtained from the organic fraction 2 after neutralization with 2 N HCl. All of the diethyl ether extracts were concentrated in vacuo and dissolved in a minimum amount of methanol prior to GC/MS analysis. The results of triplicate extractions are shown in Table I.

GC/MS Analysis of Flavor-Active Compounds. A Hewlett-Packard GC/mass selective detector (5890 GC/5971B MSD) was used for the separation and acquisition of the electron impact (70 eV) mass spectra of the compounds. Two microliters of the concentrated extracts (in methanol) were injected in a splitless mode into a fused silica capillary column (DB-5 or DB-1701, 30 m × 0.32 mm i.d. × 0.25 μm film thickness; Chromatographic Specialities). The initial column temperature (60 °C) was increased from 60 to 70 °C at a 10 °C/min rate for 1.0 min and maintained at 70 °C for 0.5 min and increased 70 to 250 °C at 5 °C/min and maintained at 250 °C for 12 min. Carrier gas (helium) flow rate was 0.85 cm^3/min; injection port temperature was 200 °C. Positive identification was confirmed by matching the mass spectra and retention times of unknowns with those of authentic standards under the same GC column (DB-5 or DB-1701) conditions. Tentative identifications were based on fragmentation patterns of unknowns with those in the Wiley/NBS mass spectral library through a PBM library search routine. A representative chromatogram is shown in Figure 1.

RESULTS AND DISCUSSION

Odor Descriptions of Flavor-Active Fractions. Most of the research regarding the flavor compounds found in fish and crustaceans has been directed toward the isolation of volatiles, mainly from cooked fish using Likens–Nickerson type apparatus or dynamic headspace sampling (DHS) equipped with Tenax TA. Very little information, however, is available on the extraction of nonvolatile flavor compounds from marine products either cooked or raw. In this laboratory, an extraction procedure was developed to obtain nonvolatile flavor-active components from raw commercial shrimp waste (Mandeville et al., 1991b). This was accomplished by fractionation of the mixture into acidic, basic, and neutral fractions, thus simplifying the complex mixture prior to GC/MS analysis. In addition, separating the flavor extracts into different fractions also revealed their sensory properties, which may have different applications in different types of food products. The main use of such flavor extracts can be found in the manufacture of surimi type products. The yield and the odor descriptions of the extracted fractions are shown in Table I. The total weight of the flavor-active compounds was 2794 mg, which represents a yield of 1.10% on a dry weight basis. The main components of the whole concentrate were in the basic and neutral methanolic fractions (29.9% each) followed by neutral aqueous fraction (17.0%), basic aqueous fraction (15.6%), acidic methanolic fraction (4.9%), and acidic aqueous fraction (2.7%). Almost 65% of the whole concentrate was obtained from the methanolic portion. Some of the compounds were present in more than one fraction, indicating that the separation procedure was not completely successful. However, the aim of the procedure was to simplify the complexity of flavor extract for the GC/MS analysis. Although the fractions were designated "basic", "acidic", and "neutral", the presence of long-chain compounds might suppress the acid–base properties, rendering them predominantly li-

Table II. Flavor-Active Compounds Identified in Boiled Shrimp Waste

adjusted ret time, min	compound[a]	% chromatographic area in fraction[b]					
		AN	AB	AA	MN	MB	MA
	Fatty Acid Esters						
19.06	dimethyl 3,3-thiobis(propanoate)*	0.9					
25.14	methyl tetradecanoate	0.5	3.2	0.1	3.0	3.8	2.9
29.14	methyl 9(*Z*)-hexadecenoate	2.5			5.6	7.7	5.2
29.63	methyl hexadecanoate	8.1	10.1		22.2	28.3	23.1
26.01	dimethyl nonanedionate*		1.3				
28.95	methyl 15-(acetylhydroxy)hexadecanoate*		1.9				
32.94	methyl 9,12(*Z*)-octadecadienoate	3.3	2.3	0.2	2.4	1.6	4.7
33.13	methyl 9(*Z*)-octadecenoate	25.3	14.9	0.5		20.7	26.4
33.22	methyl 12(*Z*)-octadecenoate		4.0		5.5	7.8	6.5
33.52	methyl octadecanoate	2.1	2.0		2.4	2.6	3.3
36.41	ethyl 2-(dimethylamino)pentadecanoate*				1.3	1.8	
36.75	methyl 5,8,11,14(*Z*)-eicosatetranoate				2.3	3.8	1.7
40.66	methyl 13(*Z*)-docosenoate	2.4					
40.91	methyl docosanoate	0.3			1.1		
	Alcohols						
19.02	dodecanol	27.4	0.6				1.9
19.06	2-methyldodecanol*	0.3					
20.67	2-(dimethylamino)cyclohexanol*		0.5				
24.69	1,14-tetradecanediol*	2.7					
28.95	hexadecanol			0.1			
29.53	heptadecanol	1.1					
33.17	5,8,11(*Z*)-heptadecatrienol*	0.7					
33.81	3,7,11-trimethyldodecan-3-ol*					1.4	
33.90	octadecanol					2.8	
	Ketones						
13.24	2,3,3,4-tetramethylcyclobutanone*		7.6				
14.80	1,3-cyclopentanedione*		8.3				
15.06	3(*R*)-methylcyclohexanone*		5.7				
33.80	2-methylcyclooctanone*				0.8		
	Carboxylic Acids						
30.42	hexadecanoic acid*	2.0		8.3	3.7	2.4	
34.04	8-heptadecenoic acid*			3.2			
	Aldehydes						
26.17	pentadecanal			1.0			
36.21	8-octadecenal*	0.4			11.4		16.8
36.25	9,17-octadecadienal*					0.8	
	Heterocyclics						
2.55	2,5-dimethyl-1*H*-pyrrole*		24.4				
10.97	6-methyl-3-pyridinol*		1.2				
14.93	pyridine*			7.7			
17.22	5-methyl-2(3*H*)-furanone*		3.8				
18.14	2,2′-bipyridine*		0.8				
23.59	4-ethylpyrido[2,3-*d*]pyrimidine*		1.4				
29.90	3,9-diazatricyclo[7.3.0.0]dodecane-2,11-dione*		1.5	0.7			
34.34	3-pyridinecarboxylic acid*			0.5			
	Miscellaneous						
23.94	cyclododecane*	1.9					
35.70	euracamide*	2.0					
35.99	9(*Z*)-octadecenamide*		1.8				
39.83	1,4,6-cyclooctatriene*				7.3		

[a] Unmarked compounds are identified by their mass spectra and by their retention times using standards under the same GC column (DB-5) conditions. Those compounds marked with an asterisk are tentatively identified according to their mass spectral fragmentations. [b] MB, methanolic basic function; MA, methanolic acidic fraction; MN, methanolic neutral fraction; AB, aqueous basic fraction; AA, aqueous acidic fraction; AN, aqueous neutral fraction.

pophilic. The term acidic fraction does not necessarily mean that it contains only acidic compounds but simply that it was extracted with a base.

Characterization of Flavor-Active Components from Cooked Shrimp Waste. *Methanolic Basic (MB) Fraction.* The extracted amount of the basic methanolic fraction was 834.5 mg and had a stimulating fresh shrimp, fruity, and fatty-like aroma (Table I). This fraction was composed of nine fatty acid esters (FAE) of C_{14}–C_{20} in length (Table II) and occupied 78.1% of the total GC chromatogram area. The most abundant compounds were methyl hexadecanoate (28.3%) and methyl 9(*Z*)-octadecenoate (20.7%). The fruity character detected could be associated with the presence of these FAE, which are known to produce fruity notes. Kubota et al. (1986, 1991), Tanchotikul and Hsieh (1989), and Kawai et al. (1991) reported the presence of similar long-chain fatty acid esters in the boiled shrimp, crayfish waste, and dried squid, respectively. However, these authors did not indicate if these fatty acid esters contribute to the aroma. The origin of these FAEs is not known; however, it might be due to the presence of esterases in the shrimp, since the same FAEs were also identified in the raw shrimp. The acid- or base-catalyzed transesterification or acid-catalyzed esterification during the extraction process is unlikely since this type of reaction requires much higher temperatures.

The remaining flavor components of this fraction were 3,7,11-trimethyl-3-dodecanol and octadecanol. In addition, hexadecanoic acid and 9,17-octadecadienal were also present.

Methanolic Neutral (MN) Fraction. The yield obtained from the neutral methanolic fraction was 138.5 mg and had a slight cooked as well as a woody-green aroma. Dodecanol and 8-octadecenal (11.4%) were two new compounds associated with this fraction. The latter aldehyde could be responsible for the woody-green aroma perceived in this fraction since it is present in high concentrations. Dodecanol was also identified by Tanchotikul and Hsieh (1991) as a component of steamed rangia clam; this compound could be generated through the fatty acid oxidation pathway. Methyl hexadecanoate (22.2%) was the most abundant FAE.

Methanolic Acidic (MA) Fraction. The odor perception for this fraction (834.5 mg) was quite intriguing since it was reminiscent of cooked as well as fresh shrimp aroma and had a woody-green note (Table I). In this fraction, 2-methylcyclooctanone, methyl docosanoate, and 1,4,6-cyclooctadecatriene were the three new components not identified in the previous fractions. 8-Octadecenal, due to its high relative abundance (16.8%), could contribute to the woody-green perception noted in this fraction.

Aqueous Fraction. The compounds representing this group were obtained from the filtrate of the meat sample after cooking. In this group, some components were also identified in the methanolic fraction. Their presence in the aqueous fraction is probably the result of leaching during cooking. Nevertheless, the aqueous fraction contained heterocyclic compounds which are formed through Maillard-type reactions. However, the percentage of the heterocyclic compounds formed was rather minimal when compared to heterocyclic volatile compounds obtained using Lickens–Nickerson apparatus or DHS. The reason for such a low yield of heterocyclic compounds is related to the high volume of water used during cooking. Kubota et al. (1986) demonstrated the contrast between volatile formation in boiled and cooked shrimp aroma; they noted that the difference of the amounts of volatiles produced was directly related to the water content used to cook the shrimp.

Aqueous Basic (AB) Fraction. The flavor profile of the aqueous basic fraction (436.5 mg) had very strong nutty, fruity, as well as boiled shrimp character. Eighteen constituents were identified, and 12 of them were not present in the methanolic extract; 2 were FAEs (dimethyl nonanedionate and methyl 15-(acetylhydroxy)hexadecanoate), and 3 were cyclic ketones [2,3,3,4-tetramethylcyclobutanone (7.6%), 3-methylcyclohexanone (5.7%), and 1,3-cyclopentadione (8.3%)]. The high percentage of these ketones could impart the fruity note present in this fraction. Five heterocyclic components were also identified; among them the 2,5-dimethyl-1*H*-pyrrole (24.4%) was the most abundant. Kawai et al (1991) reported the presence 2,5-dimethyl-1*H*-pyrrole in the flavor components of dried squid. In addition, 5-methyl-2(3*H*)-furanone was another flavor-active heterocyclic compound present in this fraction.

Aqueous Acidic (AA) Fraction. This fraction (75 mg) had a woody, floral, and slightly rancid aroma. Six components were identified which were not present in the previous fractions; among them were pentadecanal, hexadecanol, and 8-heptadeceneoic acid. Hexadecanol has been identified in the steamed rangia clam (Tanchotikul and Hsieh, 1991). Long-chain aliphatic aldehydes were also identified in the volatiles of short-necked clam (Kubota et al., 1991). The other components contributing to the flavor profile of this fraction were two heterocyclic compounds (pyridine and 3-pyridinecarboxylic acid). Pyridine was also reported as a flavor constituent of dried squid (Kawai et al., 1991) and in thermally processed crayfish and blue crab (Hsieh et al., 1989), respectively. Pyridine was also reported by Kubota et al. (1982) to be present in the cooked odor of Antarctic krill as well as in roasted and boiled shrimp (Kubota et al., 1986, 1988).

Table III. Mass Spectral Data of Heterocyclic Compounds Found in Shrimp Waste

compound	*m/z* (relative intensity)
2,5-dimethyl-1*H*-pyrrole	96 (5), 95 (43), 94 (100), 93 (15), 92 (3), 80 (18), 67 (9), 66 (4), 53 (16), 52 (8), 51 (8)
6-methyl-3-pyridinol	111 (2), 109 (100), 108 (2), 94 (9), 81 (51), 80 (49), 68 (6), 54 (12), 53 (15), 52 (3), 51 (11)
pyridine	81 (4), 80 (8), 79 (100), 78 (13), 77 (5), 70 (8), 53 (10), 52 (68), 51 (33)
5-methyl-2(3*H*)-furanone	99 (7), 98 (100), 97 (9), 83 (11), 70 (12), 69 (9), 68 (8), 56 (22), 51 (6)
2,2′-bipyridine	157 (12), 156 (100), 155 (40), 130 (9), 128 (29), 78 (21), 52 (11)
4-ethylpyrido[2,3-*d*]pyrimidine	159 (80), 158 (100), 131 (24), 105 (9), 104 (9), 78 (9)
3,9-diazatricyclo[7.3.0.0]-dodecane-2,11-dione	194 (31), 138 (5), 124 (8), 111 (4), 110 (6), 97 (8), 96 (12), 71 (6), 70 (100), 69 (16), 68 (12), 55 (13)
3-pyridinecarboxylic acid	124 (8), 123 (100), 106 (5), 105 (59), 79 (6), 77 (23), 51 (6)

Aqueous Neutral (AN) Fraction. The aqueous neutral fraction (475 mg) had an analogous odor to that of the AA fraction with the addition of a rancid note. A total of 20 peaks (see Figure 1) were identified, and 8 of them were not detected in the other fractions. The floral note is most likely related to dodecanol since it occupies 27.4% of the total GC area. Alcohols usually do not contribute to the flavor profile unless they are present in high concentration (Heath and Reineccius, 1986). In addition, four other alcohols were also present in this fraction. The remaining compounds were methyl 13(*Z*)-docosenoate, dimethyl 3,3′-thiobis(propanoate), and euracamide.

Heterocyclic Compounds Identified in Shrimp Waste. The mass spectral data of the heterocyclic compounds identified in shrimp waste are shown in Table III. The importance of these compounds in food products is primarily due to their exceptional sensory properties and to their low threshold values. Because of their properties, they are considered as character-impact compounds in food. Very few heterocyclic compounds are produced by enzymatic action; the majority are produced through thermal degradation of carbohydrates or other precursors or via the Maillard reaction. The formation of 5-methyl-2(3*H*)-furanone, for example, results from the thermal degradation of carbohydrates through the intermediacy of 5-(hydroxymethyl)-2-furancarboxaldehyde (Manley et al., 1974). 2,5-Dimethylpyrrole arises via Maillard or Strecker reaction between sugars and amino acids during thermal treatment (Hodge, 1953). Pyridines arise from the thermal degradation of sulfur-containing amino acids (Kato et al., 1973) alone or in the presence of glucose (Kato et al., 1969). Another possibility is from the interaction of 1,5-dicarbonyl compounds with ammonia or amino acids.

Conclusion. Information obtained through this study and others on the recovery of flavorants from shellfish

offal may facilitate the selection and recovery of important natural flavor-active components from the abundant shrimp waste generated each year and at the same time enhance its economic value. Furthermore, knowledge of the precursors present in the raw shrimp and the products formed in the cooked waste will facilitate the understanding of the pathways for the formation of volatile components generated during cooking through Maillard-type reactions.

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LITERATURE CITED

Hayashi, T.; Yamaguchi, K.; Konosu, K. Sensory analysis of taste-active components in the extract of boiled snow crab meat. *J. Food Sci.* **1981**, *46*, 479–83.

Heath, H. B.; Reineccius, G. *Flavor Chemistry and Technology*; AVI Publishing: Wesport, CT, 1986.

Hodge, J. E. The Amadori rearrangement. *Adv. Carbohydr. Chem.* **1953**, *10*, 169–206.

Hsieh, T. C.-Y.; Vejaphan, W.; Williams, S. S.; Matiella, J. E. Volatile flavor components in thermally processed Louisiana red swamp crayfish and blue crab. In *Thermal generation of aromas*; Parliment, T. H., McGorrin, R. J., Eds.; ACS Symposium Series 409; American Chemical Society: Washington, DC, 1989; Chapter 36.

Kato, H.; Rhue, M. R.; Nishimura, T. Role of free amino acid and peptides in food taste. In *Flavor Chemistry: Trends and Developments*; Terashini, R., Buttery, R. G., Shahidi, F., Eds.; ACS Symposium Series 388; American Chemical Society: Washington, DC, 1989; Chapter 13.

Kato, S.; Fujimaki, M.; Kurata, T. Pyrolysis of sulfur-containing amino acids; *Agric. Biol. Chem.* **1969**, *33* (8), 1144–51.

Kato, S.; Kurata, T.; Ishiguro, S.; Fujimaki, M. Additional volatile compounds produced by pyrolysis of sulfur-containing amino acids. *Agric. Biol. Chem.* **1973**, *37* (7), 1759–61.

Kawai, T.; Ishida, Y.; Kakiuchi, H.; Ikeda, N.; Higashida, T.; Nakamura, S. Flavor components of dried squid. *J. Agric. Food Chem.* **1991**, *39*, 770–7.

Kubota, K.; Kobayashi, A.; Yamanishi, T. Basic and neutral compounds in the cooked odor from Antarctic krill. *Agric. Biol. Chem.* **1982**, *46* (11), 2835–9.

Kubota, K.; Shijimaya, H.; Kobayashi, A. Volatile components of roasted shrimp. *Agric. Biol. Chem.* **1986**, *50* (11), 2867–73.

Kubota, K.; Watanabe, K.; Kobayashi, A. Novel dithiazine compounds in volatile components from cooked Sakuraebi (*Sergia Lucens* Hansen). *Agric. Biol. Chem.* **1988**, *52* (6), 1537–40.

Kubota, K.; Nakamoto, A.; Moriguchi, M.; Kobayashi, A.; Ishii, H. Formation of pyrrolidino[1,2-e]-4H-2,4-dimethyl-1,3,5-dithiazine in the volatiles of boiled short-necked clam, clam and corbicula. *J. Agric. Food Chem.* **1991**, *39*, 1127–30.

Mandeville, S.; Yaylayan, V.; Simpson, B.; Ramaswamy, H. Isolation and identification of carotenoids pigments, lipids and flavor active components from raw commercial shrimp waste. *Food Biotechnol.* **1991a**, *5*, 185–95.

Mandeville, S.; Yaylayan, V.; Simpson, B.; Paré, J. GC-MS analysis of flavor active compounds from raw commercial shrimp waste. *Spectrosc: Int. J.* **1991b**, *9*, 61–72.

Mandeville, S.; Yaylayan, V.; Simpson, B. Proximate analysis, isolation of free amino acids and sugars from commercial shrimp waste. *Food Biotechnol.* **1992**, *6* (1), 51–64.

Manley, C. H.; Vallon, P. P.; Erickson, R. E. Some aroma components of roasted sesame seed (*sesasum indicum* L.). *J. Food Sci.* **1974**, *39*, 73–6.

Tanchotikul, U.; Hsieh, T. C.-Y. Volatile flavor components in crayfish waste. *J. Food Sci.* **1989**, *54* (6), 1515–20.

Tanchotikul, U.; Hsieh, T. C.-Y. Analysis of volatile flavor components in steamed Rangia clam by dynamic head space sampling and simultaneous distillation and extraction. *J. Food Sci.* **1991**, *56* (2), 327–31.

Voight, M. N.; Osborne, R.; Hall, D. E. Profile of taste active components in crab products/by-products and in cod surimi. In *Advances in fisheries technology and biotechnology for increased profitability*; Voight, M. N., Botta, J. R., Eds.; Technomic Publishing: Lancaster, PA, 1990; pp 453–68.

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Registry No. $MeOC(O)(CH_2)_2SS(CH_2)_2CO_2Me$, 15441-06-2; $CH_3(CH_2)_{12}CO_2Me$, 124-10-7; (*Z*)-$CH_3(CH_2)_5CH{=}CH(CH_2)_7CO_2Me$, 1120-25-8; $CH_3(CH_2)_{14}CO_2Me$, 112-39-0; $CH_3CH(OAc)(CH_2)_{13}CO_2Me$, 88167-68-4; (*Z*,*Z*)-$CH_3(CH_2)_4CH{=}CHCH_2CH{=}CH(CH_2)_7CO_2Me$, 112-63-0; (*Z*)-$CH_3(CH_2)_7CH{=}CH(CH_2)_7CO_2Me$, 112-62-9; (*Z*)-$CH_3(CH_2)_4CH{=}CH(CH_2)_{130}CO_2Me$, 2733-86-0; $CH_3(CH_2)_{16}CO_2Me$, 112-61-8; $CH_3(CH_2)_{12}CH(NMe_2)CO_2Et$, 141850-73-9; (*Z*,*Z*,*Z*,*Z*)-$CH_3(CH_2)_4CH{=}CHCH_2CH{=}CHCH_2CH{=}CHCH_2CH{=}CH(CH_2)_3CO_2Me$, 2566-89-4; (*Z*)-$CH_3(CH_2)_7CH{=}CH(CH_2)_{131}CO_2Me$, 1120-34-9; $CH_3(CH_2)_{20}CO_2Me$, 929-77-1; $CH_3(CH_2)_{11}OH$, 112-53-8; $CH_3(CH_2)_9CH(CH_3)CH_2OH$, 22663-61-2; $OH(CH_2)_{14}OH$, 19812-64-7; $CH_3(CH_2)_{15}OH$, 36653-82-4; $CH_3(CH_2)_{16}OH$, 1454-85-9; $CH_3(CH_2)_4CH{=}CHCH_2CH{=}CHCH_2CH{=}CH(CH_2)_4Oh$, 22117-09-5; $(CH_3)_2CH(CH_2)_3CH(CH_3)(CH_2)_3CH(OH)(CH_3)CH_2CH_3$, 83377-04-2; $CH_3(CH_2)_{17}OH$, 112-92-5; $CH_3(CH_2)_{14}CO_2H$, 57-10-3; $CH_3(CH_2)_7CH{=}CH(CH_2)_6CO_2H$, 1975-86-6; $CH_3(CH_2)_{13}CHO$, 2765-11-9; $CH_3(CH_2)_8{-}CH{=}CH(CH_2)_6CHO$, 56554-94-0; $CH_2{=}CH(CH_2)_6CH{=}CH(CH_2)_7CHO$, 85263-73-6; (*Z*)-$CH_3(CH_2)_7{-}CH{=}CH(CH_2)_7CONH_2$, 301-02-0; dimethyl nonanedionate, 141850-72-8; 2-(dimethylamino)cyclohexanol, 30727-29-8; 2,3,3,4-tetramethylcyclobutanone, 53907-62-3; 1,2-cyclopentanedione, 3859-41-4; (*R*)-3-methylcyclohexanone, 13368-65-5; 2-methylcyclooctanone, 10363-27-6; 2,5-dimethyl-1*H*-pyrrole, 625-84-3; 6-methyl-3-pyridinol, 1121-78-4; pyridine, 110-86-1; 5-methyl-2(3*H*)-furanone, 591-12-8; 2,2′-bipyridine, 366-18-7; 4-ethylpyrido[2,3-*d*]pyrimidine, 28732-68-5; cyclododecane, 294-62-2; 1,4,6-cyclooctatriene, 3725-30-2; 3-pyridinecarboxylic acid, 59-67-6.