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Received for review December 15, 1983. Revised manuscript received May 4, 1984. Accepted May 18, 1984.

Volatile Compounds from Heated Beef Fat and Beef Fat with Glycine

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One hundred forty-three compounds were isolated and identified from the dichloromethane extracts of heated beef fat and heated beef fat with glycine. Beef fat or beef fat with glycine was heated in a pressurized bottle at 200 °C for 4 h. The chemicals isolated from the heated samples using a simultaneous steam distillation/solvent (dichloromethane) extraction apparatus were subjected to gas chromatography and gas chromatography/mass spectrometry. The compounds identified included 15 *n*-alkanes, 12 *n*-alkenes, 13 *n*-aldehydes, 13 2-ketones, 12 *n*-alcohols, 11 *n*-alkylcyclohexanes, and 10 nitrogen-containing compounds. Formation mechanisms of some major products were proposed based on the radicals RCOO·, RCH₂O·, R·, and RO· formed from triglyceride.

Deep-fat frying is one of the most common cooking practices. Fat is used not only for transferring heat to cooking foods but also for giving flavor to foods, so the correct fat must be chosen in order to obtain satisfactory flavor. The fat found in meat seems to play an important role in the formation of cooked meat flavor (Yamamoto et al., 1970). Yamamoto et al. (1970) identified a series of fatty aldehydes in beef fat heated at 150 and 200 °C either under a nitrogen stream or in air. These carbonyl compounds may react with some nitrogen-containing compounds such as an amino acid to produce major cooked flavor chemicals such as heterocyclic compounds (Shibamoto, 1983). Buttery et al. (1977) identified many nitrogen-containing heterocyclic compounds (pyridines and pyrazines) in the volatile oils obtained from roasted lamb fat at 150 °C. These reports indicate that fat plays an important role in the formation of flavors in cooked foods.

Fats used for frying are usually heated over a prolonged period. Occupational cooking in a restaurant may involve the use of the same oil for more than a week. Fats that have been heated to high temperatures for long periods undergo chemical changes, including an increase in formation of carbonyl compounds.

In this study, compounds formed in heated beef fat and beef fat with glycine were isolated and identified in order to investigate a role of fat in flavor formation in cooked foods.

EXPERIMENTAL SECTION

Materials. The beef fat was obtained from the renal periphery of the beef carcass. The fat tissue was ground

with small amounts of dry ice in a blender. The pulverized, solidified fat was placed in a glass container and heated in a water bath at 70–80 °C. The nonfat materials such as blood, muscle, and connective tissue were denatured by the heat and removed from the melted fat by filtration. The refined beef fat was weighed and stored in a freezer (–5 °C). Glycine was purchased from Eastman Kodak Co., Rochester, NY, and used without further purification. Authentic reference compounds were obtained from reliable commercial sources or were donated by Ogawa & Co., Ltd., Tokyo, Japan. 2-Butylpyridine and 2-pentylpyridine were synthesized according to Vogel (1962).

Sample Preparations. Beef fat (10.20 g) and glycine (9.95 g) were mixed in a pressurized bottle. The bottle was placed in an oven and heated at 200 °C for 4 h. The dark brown reaction mixture obtained was subjected to a simultaneous steam distillation (1 L of water) and solvent (dichloromethane, 200 mL) extraction. The dichloromethane extract was dried over anhydrous sodium sulfate. After the solvent was removed by using a Kuderna-Danish evaporative concentrator, the brown oily liquid obtained (381 mg) was analyzed with instrumental techniques. Additional experiments varying the quantities of beef fat and glycine (beef fat/glycine = 10/1 and 2/1 and beef fat alone) were conducted by using the same procedure as above, and the samples obtained from each experiment were also analyzed.

Isolation and Identification of Components in the Heated Samples. All samples were analyzed with Kovats gas chromatographic retention index [*I* (Kovats, 1965)] and gas chromatography/mass spectrometry (GC/MS) techniques as described previously (Yamaguchi and Shibamoto, 1980; Toda et al., 1982). The gas chromatographic retention index (Kovats index) and MS fragmentation pattern

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Table I. Compounds Identified in Extracts of Treated Beef Fat with Glycine

component	beef fat alone		beef fat/glycine (1/1)		ID	
	peak area, %	I_{OV-101}	peak area, %	I_{OV-101}	MS	I
acetaldehyde		<i>d</i>	0.01	389	+	+
acetone	0.06	530	0.02	530	+	+
dichloromethane (solvent)	<i>a</i>	540	<i>a</i>	540	+	+
2-butanone	0.04	543	<i>b</i>	543	+	+
ethyl acetate	0.04	580	<i>b</i>	579	+	+
<i>n</i> -hexane	0.66	600	0.41	600	+	+
3-butenol	0.66	641	<i>c</i>	640	+	+
1-butanol	<i>b</i>	649	0.15	649	+	+
3-methyl-2-butanone	<i>b</i>	650	<i>d</i>		+	+
3-methylbutanolpentanal	0.24	661	<i>b</i>	660	+	+
1-pentanal	<i>b</i>	681	0.35	680	+	+
<i>n</i> -propyl acetate	0.05	694	<i>b</i>	694	+	+
<i>n</i> -heptane	0.29	700	0.09	700	+	+
2-pentanone	0.04	703	<i>b</i>	705	+	+
2-methylbutanol	0.16	726	<i>b</i>	727	+	+
<i>N</i> -methylpyrrole		<i>d</i>	0.82	730	+	+
1-pentanol	0.78	747	1.82	750	+	+
isobutyl acetate	<i>b</i>	758	<i>c</i>	760	+	+
2-hexanone	0.05	775	0.22	768	+	+
1-hexanal	0.58	780	0.24	778	+	+
3-hexanol	<i>c</i>	783	<i>b</i>	785	+	+
<i>n</i> -butyl acetate	<i>b</i>	789	<i>b</i>	788	+	+
2-hexanol	<i>c</i>	790	<i>b</i>	790	+	+
1-octene	0.66	791	<i>c</i>	795	+	+
<i>n</i> -octane	0.11	800	<i>b</i>	800	+	+
<i>n</i> -butyl acetate	0.19	805	<i>c</i>	810	+	+
<i>N,N</i> -dimethylacetamide		<i>d</i>	0.18	820	+	+
4-methylpentanol	<i>c</i>	840	0.12	839	+	+
ethylcyclohexane	0.52	843	<i>b</i>	845	+	+
2-hexenal	0.04	857	1.54	854	+	+
ethylbenzene	<i>b</i>	858	<i>d</i>		+	+
dihydro-2(3 <i>H</i>)-furanone	<i>b</i>	865	0.34	866	+	+
1-hexanol	0.38	868	<i>b</i>	868	+	+
2-heptanone	0.66	870	0.98	870	+	+
1-heptanal	0.90	880	<i>b</i>	880	+	+
3-heptanol	<i>b</i>	882	<i>b</i>	882	+	+
2-heptanol	<i>c</i>	883	0.39	883	+	+
3-hydroxy-2(1 <i>H</i>)-pyridinone		<i>d</i>	0.30	884	+	
1-nonene	0.04	895	<i>b</i>	890	+	+
1-nonane	1.33	900	1.30	900	+	+
<i>n</i> -pentyl acetate	<i>b</i>	903	0.30	905	+	+
methyl <i>n</i> -hexanoate	<i>b</i>	905	<i>c</i>	907	+	+
1,1,3-trimethylcyclopentane	<i>b</i>	908	<i>c</i>	910	+	+
<i>n</i> -propylbenzene	<i>b</i>	929	<i>d</i>		+	+
1-ethyl-3-methylcyclohexane		<i>d</i>	<i>b</i>	931	+	+
<i>n</i> -propylcyclohexane	0.11	931	0.29	935	+	+
3-heptenol	1.09	932	<i>c</i>	940	+	+
2-heptenal	<i>c</i>	951	<i>b</i>	945	+	+
6-methylheptan-2-one	1.53	957	4.41	957	+	+
6-methylheptanol	0.57	960	<i>b</i>	960	+	+
1-octen-3-ol	<i>c</i>	965	<i>b</i>	967	+	+
2-octanone	3.44	980	2.60	978	+	+
3-octanol	<i>b</i>	982	<i>c</i>	980	+	+
3-methylcyclohexanone	<i>b</i>	985	<i>d</i>		+	+
3-octanone	0.82	991	<i>c</i>	988	+	+
1-decene	0.84	992	<i>b</i>	990	+	+
1-octanal	<i>c</i>	995	0.54	994	+	+
<i>n</i> -decane	0.74	1000	0.72	1000	+	+
2-octanol	1.70	1011	1.62	1009	+	+
<i>p</i> -isopropyltoluene (<i>p</i> -cymene)	1.66	1018	0.61	1020	+	+
5-ethylidihydro-2(3 <i>H</i>)-furanone	<i>b</i>	1020	<i>b</i>	1025	+	+
<i>n</i> -butylcyclohexane	<i>b</i>	1032	<i>b</i>	1035	+	+
<i>n</i> -butylcyclohexene	1.82	1034	1.63	1040	+	+
6-methylheptanol	2.26	1055	4.66	1057	+	+
<i>n</i> -butylbenzene	<i>b</i>	1057	<i>d</i>		+	+
1-octanol	0.55	1061	<i>b</i>	1062	+	+
2-nonanone	<i>b</i>	1076	3.80	1073	+	+
1-ethenyl-4-ethylbenzene		<i>d</i>	<i>b</i>	1079	+	
1-nonanal	7.47	1084	0.54	1084	+	+
1-undecene	0.24	1087	<i>c</i>	1090	+	+
<i>n</i> -heptyl acetate	<i>b</i>	1093	0.30	1096	+	+
<i>n</i> -undecane	0.14	1100	1.03	1100	+	+
3-octyl acetate	0.21	1110	0.65	1110	+	+
<i>n</i> -pentylcyclohexane	0.21	1130	<i>b</i>	1134	+	+
7-methyloctanol	3.37	1138	<i>b</i>	1140	+	+

Table I (Continued)

component	beef fat alone		beef fat/glycine (1/1)		ID	
	peak area, %	I_{OV-101}	peak area, %	I_{OV-101}	MS	I
<i>o</i> -ethylbenzaldehyde	0.28	1144		<i>b</i>	+	+
1-nonanol	0.24	1152	0.23	1151	+	+
<i>N</i> -methylacetamide		<i>d</i>	1.42	1158	+	+
2-decanone	1.09	1172	3.00	1173	+	+
2-butylpyridine		<i>d</i>	6.77	1177	+	+
1-decanal	4.16	1182		<i>b</i>	+	+
<i>n</i> -dodecane	1.16	1200	0.88	1200	+	+
methyl <i>n</i> -nonanoate	1.20	1210		<i>c</i>	+	+
5-butylidihydro-2(3 <i>H</i>)-furanone	0.85	1218	0.95	1216	+	+
<i>n</i> -hexylcyclohexane	9.93	1243		<i>b</i>	+	+
1-decanol	0.60	1257	0.18	1257	+	+
2-pentylpyridine		<i>d</i>	3.03	1258	+	+
2-undecanone	<i>b</i>	1270	1.40	1274	+	+
1-undecanal	2.57	1281	2.51	1281	+	+
1-tridecene	1.32	1287	0.49	1288	+	+
<i>n</i> -tridecane	0.23	1300	0.87	1300	+	+
5-pentylidihydro-2(3 <i>H</i>)-furanone	0.48	1331	0.17	1328	+	+
9-methyldecanol	0.56	1337		<i>c</i>	+	+
<i>n</i> -heptylcyclohexane	8.40	1343	1.04	1343	+	+
5-imino-2-methylcyclopenten-1-ol		<i>d</i>	<i>b</i>	1358	+	+
1-undecanol	0.68	1361	2.49	1360	+	+
2-dodecanone	1.26	1371	1.37	1375	+	+
1-dodecanal	<i>b</i>	1383	0.15	1388	+	+
1-tetradecene	<i>c</i>	1390	<i>b</i>	1390	+	+
<i>n</i> -tetradecane	1970	1400	1.51	1400	+	+
methyl <i>n</i> -undecanoate	0.42	1419		<i>b</i>	+	+
<i>n</i> -octylcyclohexane	0.47	1429	0.15	1423	+	+
<i>N</i> -butylacetamide		<i>d</i>	0.23	1433	+	+
10-methylundecanol	0.72	1443	0.23	1447	+	+
N-containing compound, $M^+ = 107$		<i>d</i>	0.21	1449		
5-imino-2-ethylcyclopenten-1-ol		<i>d</i>	<i>b</i>	1461	+	
1-dodecanol	1.03	1465	<i>b</i>	1463	+	+
2-tridecanone	0.99	1478	0.62	1477	+	+
<i>n</i> -undecyl acetate	<i>b</i>	1483	0.16	1486	+	+
1-pentadecene	1.13	1490	1.00	1489	+	+
1-tridecanal	<i>b</i>	1492	<i>b</i>	1494	+	+
<i>n</i> -pentadecane	1.13	1500	1.95	1500	+	+
<i>N</i> -octylformamide		<i>d</i>	<i>b</i>	1505	+	+
2-tridecanol	<i>b</i>	1510	<i>c</i>	1510	+	+
N-containing compound, $M^+ = 181$		<i>d</i>	2.19	1520		
11-methyldodecanol	<i>b</i>	1535	<i>c</i>	1528	+	+
<i>N</i> -pentylacetamide		<i>d</i>	<i>b</i>	1536	+	+
5-heptyldihydro-2(3 <i>H</i>)-furanone	<i>b</i>	1540	0.23	1548	+	+
<i>n</i> -nonylcyclohexane	0.67	1543	<i>b</i>	1543	+	+
5-imino-2-propylcyclopenten-1-ol		<i>d</i>	<i>b</i>	1544	+	
2-tridecenal	<i>c</i>	1553	0.18	1553	+	+
1-tridecanol	1.22	1563	<i>b</i>	1560	+	+
2-tetradecanone	0.21	1580	4.41	1576	+	+
1-hexadecene	0.96	1593	<i>c</i>	1590	+	+
<i>n</i> -hexadecane	0.35	1600	0.32	1600	+	+
methyl <i>n</i> -tridecanoate		<i>d</i>	<i>b</i>	1622	+	+
<i>n</i> -decylcyclohexane	<i>c</i>	1640	<i>b</i>	1642	+	+
N-containing compound, $M^+ = 185$		<i>d</i>	0.33	1665		
<i>N</i> -hexylacetamide		<i>d</i>	0.60	1670	+	+
1-tetradecanol	0.52	1675	2.98	1680	+	+
1-heptadecene	1.28	1692	<i>c</i>	1690	+	+
<i>n</i> -heptadecane	0.96	1700	0.70	1700	+	+
13-methyltetradecanol	0.52	1736	<i>b</i>	1740	+	+
5-octylidihydro-2(3 <i>H</i>)-furanone	<i>c</i>	1760	0.27	1760	+	+
1-pentadecanol	2.23	1768	1.27	1766	+	+
<i>O</i> -methyloxime pentadecanal		<i>d</i>	0.19	1776	+	
2-hexadecanone	0.20	1780	2.73	1782	+	+
1-pentadecanal	<i>c</i>	1785	<i>b</i>	1784	+	+
<i>n</i> -octadecane	0.40	1800	0.55	1800	+	+
<i>n</i> -pentadecyl acetate	0.40	1843	<i>c</i>	1850	+	+
1-hexadecanol	0.30	1850	<i>b</i>	1852	+	+
1-heptadecanal	<i>b</i>	1880	<i>b</i>	1880	+	+
methyl <i>n</i> -pentadecanoate		<i>d</i>	1.35	1881	+	+
2-heptadecanone	2.27	1881	1.00	1883	+	+
1-nonadecene	<i>b</i>	1883	<i>b</i>	1885	+	+
<i>O</i> -methyloxime hexadecanal		<i>d</i>	0.29	1890	+	
<i>n</i> -dodecanamide		<i>d</i>	3.34	1892	+	+
<i>n</i> -nonadecane	2.37	1900	0.76	1900	+	+

Table I (Continued)

component	beef fat alone		beef fat/glycine (1/1)		ID	
	peak area, %	I_{OV-101}	peak area, %	I_{OV-101}	MS	I
N-containing compound, $M^+ = 213$		<i>d</i>	<i>b</i>	1918		
<i>N</i> -nonylacetamide		<i>d</i>	0.71	1934	+	+
<i>N</i> -ethyloctamide		<i>d</i>	<i>b</i>	1945	+	
<i>n</i> -tridecylcyclohexane	<i>b</i>	1949	<i>b</i>	1950	+	+
1-octadecanal	<i>b</i>	1975	0.81	1980	+	+
<i>n</i> -eicosene	<i>b</i>	1986	<i>b</i>	1990	+	+
<i>n</i> -eicosane	2.72	2000	0.93	2000	+	+
<i>n</i> -tetradecylcyclohexane	<i>b</i>	2043	0.81	2036	+	+
1-octadecanol	<i>c</i>	2050	<i>b</i>	2050	+	+
<i>n</i> -heptadecyl acetate	0.32	2093	2.21	2089	+	+

^aThe solvent peak (dichloromethane) was excluded in calculation of peak area percent. ^bPeak area percent was less than 0.01. ^cThe presence of a peak was recognized but mass spectra could not be obtained to confirm the identity due to the lack of quantity. ^dNot found in this sample.

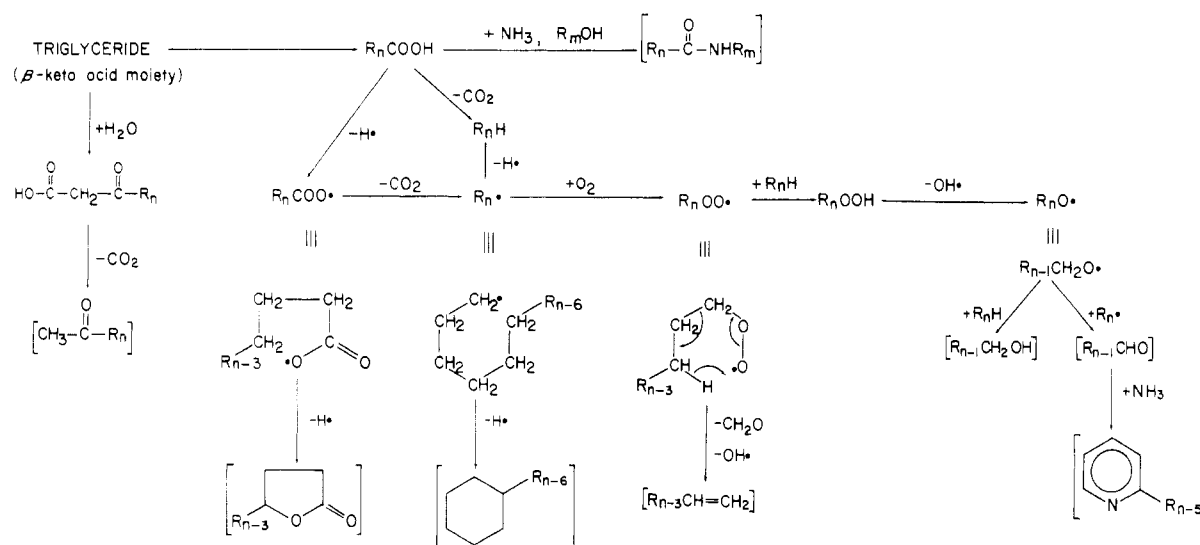


Figure 1. Proposed formation mechanisms of major components (in brackets) identified in heated fat or heated fat with glycine.

of each component were compared with those of the authentic compound to identify the components of the samples. An additional computer search of standard mass spectra to confirm the identity of unknowns was conducted on the "NIH/EPA Chemical Information System" (1978).

Hewlett-Packard Model 5880 and 5790 gas chromatographs equipped with a flame ionization detector (FID) and a flame thermionic detector (FTD) were used. The columns used were 50 m \times 0.23 mm i.d. fused silica capillary columns coated with either Carbowax 20M or OV-101 (donated by Dr. O. Nishimura, Ogawa & Co., Ltd., Tokyo, Japan). Peak areas were integrated by using a Hewlett-Packard Model 3385-A automation system and are reported in Table I. A Finnigan Model 3200 GC/MS equipped with a Finnigan Model 6000 MS data system was used for mass spectral identification of the gas chromatographic components.

RESULTS AND DISCUSSION

The compounds identified in the dichloromethane extracts are listed in Table I in order of elution from the gas chromatographic column OV-101. Table I is prepared by the method reported by Flath et al. (1983). The compounds listed were identified by mass spectra and Kovats indices (I) obtained from two different polarity columns (OV-101 and Carbowax 20M). Some compounds were not identified positively because of the lack of authentic compounds, so the Kovats index could not be compared. The presence of nitrogen in certain unknowns was recognized

in the gas chromatogram monitored with FTD; their mass spectra show a possible molecular ion. These compounds are listed as N-containing compounds in Table I along with their possible molecular ion number.

Among 112 compounds identified in the extract from the beef fat heated alone, the major components were *n*-alkanes, *n*-alcohols, *n*-aldehydes, *n*-alkylcyclohexanes, and 2-ketones. The isolation of *n*-alkanes and *n*-alkenes was consistent with the report of Alencar et al. (1983), who found a series of *n*-alkanes and *n*-alkenes in pyrolyzed oil samples at 300–500 °C. Alencar et al. (1983) identified *n*-alkylcyclohexanes and *n*-alkylcyclohexenes also. We identified 11 *n*-alkylcyclohexanes but found only one *n*-alkylcyclohexene (*n*-butylcyclohexene). *n*-Alkylcyclopentanes, which were also reported in Alencar et al. (1983), were not identified in our samples. This may be due to the use of a somewhat low temperature (200 °C) in our experiments. It has been proposed that methyl ketones form from β-keto acids, which are derived from triglyceride by heat treatment (Schwartz et al., 1966). Shibamoto et al. (1980) observed formation of methyl ketones when milk fat was heated over 100 °C. Twelve methyl ketones were identified in the present study. Yamamoto et al. (1970) reported several fatty aldehydes in beef fat heated at 150 °C. They found longer chain aldehydes when beef fat was heated at 200 °C. *n*-Nonanal, suggested as a possible precursor of 2-butylpyridine (Buttery et al., 1977), was found in large quantity (7.47%) in the present study. Many isoacid derivatives such as isoalcohols were identi-

Table II. Yields of 2-Butyl- and 2-Pentylpyridine in the Beef Fat/Glycine Reaction Systems

reactant	ratio	peak area, % ^a	
		2-butylpyridine	2-pentylpyridine
beef fat/ glycine	1/1	6.77	3.03
	2/1	0.98	0.08
	10/1	0.03	trace

^a Peak area of alkyl pyridine/total peak area of a sample \times 100.

fied. This is reasonable, because isoacids are one of the most abundant acids in many animal fats (Abrahamsson et al., 1963). Homologues of 5-alkyldihydro-2(3*H*)-furanone were tentatively identified. These compounds may be formed from the RCOO· radical via ring closure between the carbonyl radical and γ -carbon atom followed by losing γ -hydrogen as a radical. Formation mechanisms of major components are well explained by the generation of the radicals RCOO·, RCH₂O·, RO·, and R· from the triglyceride molecule, and they are shown in Figure 1. The scheme shown in Figure 1 was made based on those reported in Alencar et al. (1983), Nonhebel et al. (1979), Bell et al. (1953), and Chang and Wong (1947).

Production of nitrogen-containing compounds in a beef fat/glycine system was much less than was expected. The carbonyl compounds formed from beef fat such as aldehydes and ketones were expected to produce some nitrogen-containing aroma compounds. Formation of pyrazines, which are major aroma components in cooked foods, was not observed. The flavor of the reaction mixture was unpleasant rather than the expected cooked flavor. This may be due to the formation of aldehydes and ketones, which are known to cause off-flavor or rancid flavor in fat products. Glycine may not react with these carbonyl compounds to form aroma compounds under the conditions used in this study, although glycine produced many flavor chemicals when it was reacted with a sugar in amino/carbonyl reaction systems (Olsson et al., 1978; Kato et al., 1977; Lipton and Dutky, 1971). On the other hand, it has been proposed that ammonia produced from glycine by the Strecker degradation (Schönberg et al., 1948) is the active form in an amino/carbonyl reaction (Shibamoto and Bernhard, 1978). The Strecker degradation may not have occurred sufficiently under the conditions used in the present study. Acetaldehyde, which is known to be a Strecker degradation product of glycine, was identified in trace quantities. It is, however, difficult to know whether acetaldehyde came from glycine or fat. The amounts of the carbonyl compounds formed from beef fat in the present study was significantly low (ca. 1-nonanal = 17 mg), which may restrict the occurrence of any secondary reactions to form flavor chemicals.

2-Alkylpyridines were proposed to form from the corresponding unsaturated *n*-aldehyde with ammonia upon heat treatment (Buttery et al., 1977). The fact that less 1-nonanal formed in the beef fat/glycine system (see Table I) than in the fat alone indicated that 1-nonanal formed from beef fat underwent secondary reaction with ammonia formed from glycine to give 2-butylpyridine. The same phenomenon was observed in the relation between 1-decanal and 2-pentylpyridine. Another major nitrogen-containing compound in the beef fat/glycine system was *N*-alkylacetamide, for which proposed formation mechanisms are also shown in Figure 1. 5-Imino-2-methylcyclopenten-1-ol was also isolated in trace quantities from the beef fat-glycine system. This compound has been isolated from the reaction mixture of 2-hydroxy-3-methyl-2-cyclopenten-1-one and ammonia (Nishimura et

al., 1980) and reportedly possesses a cooked-rice flavor.

The only obvious differences between the compositions of three samples from three different reactant ratios (beef fat/glycine = 10/1, 2/1, and 1/1) in the beef fat/glycine reaction system were the quantity of 2-alkylpyridines formed. Table II shows the gas chromatographic peak area percentages of 2-butylpyridine and 2-pentylpyridine in the three samples. The beef fat/glycine system did not produce a pleasant cooked flavor compared with a sugar/amino acid system. Yamamoto et al. (1970) proposed that fats must be added in order to obtain a satisfactory meaty aroma in the sugar/amino acid reaction systems. The compounds produced from the beef fat in the present study may contribute to formation of meaty aroma in cooked meats, but further investigation is necessary to understand the role of heated fat in the flavors formed in actual cooked foods.

Registry No. Glycine, 56-40-6; acetaldehyde, 75-07-0; acetone, 67-64-1; dichloromethane, 75-09-2; 2-butanone, 78-93-3; ethyl acetate, 141-78-6; *n*-hexane, 110-54-3; 3-butenol, 627-27-0; 1-butanol, 71-36-3; 3-methyl-2-butanone, 563-80-4; 3-methylbutanal, 590-86-3; pentanal, 110-62-3; *n*-propyl acetate, 109-60-4; *n*-hexane, 142-82-5; 2-pentanone, 107-87-9; 2-methylbutanol, 137-32-6; *N*-methylpyrrole, 96-54-8; 1-pentanol, 71-41-0; isobutyl acetate, 110-19-0; 2-hexanone, 591-78-6; hexanal, 66-25-1; 3-hexanol, 623-37-0; 2-hexanol, 626-93-7; 1-octene, 111-66-0; *n*-octane, 111-65-9; *n*-butyl acetate, 123-86-4; *N,N*-dimethylacetamide, 127-19-5; 4-methylpentanol, 626-89-1; ethylcyclohexane, 1678-91-7; 2-hexenal, 505-57-7; ethylbenzene, 100-41-4; dihydro-2(3*H*)-furanone, 96-48-0; 1-hexanol, 111-27-3; 2-heptanone, 110-43-0; heptanal, 111-71-7; 3-heptanol, 589-82-2; 2-heptanol, 543-49-7; 3-hydroxy-2(1*H*)-pyridinone, 16867-04-2; 1-nonene, 124-11-8; *n*-nonane, 111-84-2; *n*-pentyl acetate, 628-63-7; methyl *n*-hexanoate, 106-70-7; 1,1,3-trimethylcyclopentane, 4516-69-2; *n*-propylbenzene, 103-65-1; 1-ethyl-3-methylcyclohexane, 3728-55-0; *n*-propylcyclohexane, 1678-92-8; octanal, 124-13-0; 3-heptanol, 10606-47-0; 2-heptenal, 2463-63-0; 6-methylheptan-2-one, 928-68-7; 6-methylheptanal, 63885-09-6; 1-octen-3-ol, 3391-86-4; 2-octanone, 111-13-7; 3-octanol, 589-98-0; 3-methylcyclohexanone, 591-24-2; 3-octanone, 106-68-3; 1-decene, 872-05-9; *n*-decane, 124-18-5; 2-octanol, 123-96-6; *p*-cymene, 99-87-6; 5-ethyldihydro-2(3*H*)-furanone, 695-06-7; *n*-butylcyclohexane, 1678-93-9; *n*-butylcyclohexene, 31620-25-4; 6-methylheptanol, 4730-22-7; *n*-butylbenzene, 104-51-8; 1-octanol, 111-87-5; 2-nonanone, 821-55-6; 1-ethenyl-4-ethylbenzene, 3454-07-7; nonanal, 124-19-6; 1-undecene, 821-95-4; *n*-heptyl acetate, 112-06-1; *n*-undecane, 1120-21-4; 3-octyl acetate, 4864-61-3; *n*-pentylcyclohexane, 4292-92-6; 7-methyloctanol, 2430-22-0; *o*-ethylbenzaldehyde, 22927-13-5; 1-nonanol, 143-08-8; *N*-methylacetamide, 79-16-3; 2-decanone, 693-54-9; 2-butylpyridine, 5058-19-5; decanal, 112-31-2; *n*-dodecane, 112-40-3; methyl *n*-nonanoate, 1731-84-6; 5-butyldihydro-2(3*H*)-furanone, 104-50-7; *n*-hexylcyclohexane, 4292-75-5; 1-decanol, 112-30-1; 2-pentylpyridine, 2294-76-0; 2-undecanone, 112-12-9; undecanal, 112-42-5; 1-tridecene, 2437-56-1; *n*-tridecane, 629-50-5; 5-pentylidihydro-2(3*H*)-furanone, 104-61-0; 9-methyldecanol, 55505-28-7; *n*-heptylcyclohexane, 5617-41-4; 5-imino-2-methylcyclopenten-1-ol, 61133-60-6; 1-undecanol, 112-42-5; 2-dodecanone, 6175-49-1; dodecanal, 112-54-9; 1-tetradecene, 1120-36-1; *n*-tetradecane, 629-59-4; methyl *n*-undecanoate, 1731-86-8; *n*-octylcyclohexane, 1795-15-9; *N*-butylacetamide, 1119-49-9; 10-methylundecanol, 20194-45-0; 5-imino-2-ethylcyclopenten-1-ol, 90554-84-0; 1-dodecanol, 112-53-8; 2-tridecanone, 593-08-8; *n*-undecyl acetate, 1731-81-3; 1-pentadecene, 13360-61-7; tridecanal, 10486-19-8; *n*-pentadecane, 629-62-9; *N*-octylformamide, 6282-06-0; 2-tridecanol, 1653-31-2; 11-methyldodecanol, 85763-57-1; *N*-pentylacetamide, 2524-60-9; 5-heptyldihydro-2(3*H*)-furanone, 104-67-6; *n*-nonylcyclohexane, 2883-02-5; 5-imino-2-propylcyclopenten-1-ol, 90554-85-1; 2-tridecanal, 7774-82-5; 1-tridecanol, 112-70-9; 2-tetradecanone, 2345-27-9; 1-hexadecene, 629-73-2; *n*-hexadecane, 544-76-3; methyl *n*-tridecanoate, 1731-88-0; *n*-decylcyclohexane, 1795-16-0; *N*-hexylacetamide, 7501-79-3; 1-tetradecanol, 112-72-1; 1-heptadecene, 6765-39-5; *n*-heptadecane, 629-78-7; 13-methyltetradecanol, 20194-47-2; 5-octylidihydro-2(3*H*)-furanone, 2305-05-7; 1-pentadecanol, 629-76-5; *O*-methyloxime pentadecanal,

90554-86-2; 2-hexadecanone, 18787-63-8; pentadecanal, 2765-11-9; *n*-octadecane, 593-45-3; *n*-pentadecyl acetate, 629-58-3; 1-hexadecanol, 36653-82-4; heptadecanal, 629-90-3; methyl *n*-pentadecanoate, 7132-64-1; 2-heptadecanone, 2922-51-2; 1-nonadecene, 18435-45-5; *O*-methyloxime hexadecanal, 68942-06-3; *n*-dodecanamide, 1120-16-7; *n*-nonadecane, 629-92-5; *N*-nonylacetylamide, 45108-98-3; *N*-ethyloctanamide, 54007-35-1; *n*-tridecylcyclohexane, 6006-33-3; octadecanal, 638-66-4; *n*-eicosene, 27400-78-8; *n*-eicosane, 112-95-8; *n*-tetradecylcyclohexane, 1795-18-2; 1-octadecanol, 112-92-5; *n*-heptadecyl acetate, 822-20-8.

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Received for review January 27, 1984. Accepted May 7, 1984.

L-Ornithyltaurine, a New Salty Peptide

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In a series of studies of the structure-bitter taste relationship of BPIa (H-Arg-Gly-Pro-Pro-Phe-Ile-Val-OH) from casein hydrolysate, the synthesized N-terminus analogues of BPIa, H-L-Orn-Gly-OH-HCl and H-L-Lys-Gly-OH-HCl, were fortuitously discovered to possess an "umami" taste about half as strong as that of monosodium glutamate (MSG), as well as a slightly salty taste. To characterize salty peptides, various analogues in which constituent amino acids were substituted were synthesized and their tastes measured. Among them, H-L-Orn- β -Ala-OH-HCl and H-L-Orn-Tau-HCl (L-ornithyl-2-aminoethanesulfonic acid hydrochloride) exhibited a salty taste equal to or greater than that of sodium chloride (NaCl), and most of the above peptides possessed an umami taste approaching or equal to that of MSG. These salty and umami peptides do not contain sodium ion in the molecules. We expect that these peptides will be useful as a new type of artificial seasoning instead of NaCl and MSG, in which the sodium content could be undesirable for diabetics and hypertensives.

In recent years, many investigators examined the chemical properties of various bitter, sweet, and sour peptides. Those peptides number about 1000. Of these, bitter peptides represent about 80%. Some have been isolated from natural foodstuffs such as cheese (Hamilton et al., 1974), natto (Maekawa and Tamai, 1965), sake (Takahashi et al., 1974), and cocoa (Pickenhangen and Dietrich, 1975). It is well-known that hydrolysis of protein with proteolytic enzymes is usually accompanied by formation of a bitter taste due to the production of bitter peptides (Yamashita et al., 1969; Arai et al., 1970; Matoba et al., 1970; Minamiura et al., 1972a,b). In addition, many workers have synthesized various bitter peptides and reported the relationship between bitter taste and chemical

structure (Shiba and Nunami, 1974; Matoba and Hata, 1972; Okai, 1977; Fukui et al., 1983; Otagiri et al., 1983; Miyake et al., 1983). As for sweet peptides, aspartame (H-Asp-Phe-OMe) and its derivatives have been studied for the development of artificial sweetening agents (Mazur et al., 1969, 1970, 1973; Fujino et al., 1973, 1976; Ariyoshi et al., 1974; Ariyoshi, 1976, 1980; Miyoshi et al., 1978). Aspartame is now being marketed for dietary purposes.

Acidic peptides containing aspartyl and/or glutamyl residues usually possess a sour taste. Sweet and sour peptides account for about 15% of sapid peptides.

Other sapid peptides are rare. Fujimaki et al. (1973) reported some di- or tripeptides possessing an "umami" (MSG-like) taste that were obtained from an enzymatic digest of fish protein. Arai et al. (1973) prepared 12 kinds of dipeptides containing a glutamyl residue and discussed the relationship between umami taste and physicochemical properties. Yamasaki and Maeda (1978) also isolated a delicious peptide from extract of beef treated with papain

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