Effect of Beef Packaging Method on Volatile Compounds Developed by Oven Roasting or Microwave Cooking

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The objectives of this study were to identify and quantify volatile compounds in beef that was dry aged or vacuum packaged and then oven roasted or microwave cooked. Supercritical CO_2 was used to extract the volatile compounds, and gas chromatography coupled with mass spectrometry or flame ionization detection was used for analyses. Classes of compounds isolated and identified included hydrocarbons (alkanes, alkenes, aromatic), alcohols, aldehydes, acids, ketones, esters, lactones, thiazoles, pyrazines, phenols, and furans. Differences in relative proportions of certain hydrocarbons, terpenoids, aldehydes, and ketones were observed for individual combinations of cooking × packaging interactions, some of which were directly attributed to autoxidation. Results indicate that quantifying single compounds was not sufficiently accurate. Instead, using total weight percentages of chemical classes was preferable.

INTRODUCTION

Vacuum packaging, microwave heating, and warmed-over flavor are important issues relating to development of new markets and new added-value beef products. Beef flavor is one of the most important qualities of beef, and a number of review papers have been written on this subject (Wasserman, 1979; MacLeod and Seyyedain-Ardebili, 1981; Gray and Pearson, 1984; Shahidi et al., 1986; Gray and Weiss, 1988; Rubin and Shahidi, 1988). Raw beef has little odor and only a blood-like taste, whereas cooking develops flavor and aroma, primarily by development of volatile compounds. However, these are extremely complex sets of compounds, and they have not been completely defined or produced in the laboratory. Furthermore, the subtle differences in flavor caused by various cooking and packaging methods are even more of a mystery. Consequently, there is strong motivation for fundamental chemical studies on the nature of volatile compounds in beef and on the variation arising from cooking and packaging methods.

A previous study (King et al., 1993) concerned the volatile composition of raw beef as determined by supercritical CO_2 extraction, followed by gas chromatography/mass spectroscopy. The purpose of the present work was to apply similar methodology to determine the volatile composition of cooked beef products. Two packaging treatments (vacuum packaging versus dry aging) and two cooking methods (oven roasting versus microwave cooking) were used. Results provide insight into the effects of both packaging and cooking methods and the interaction between the two on volatile compounds, many of which are likely involved in beef flavor and aroma. Results from this study should help to
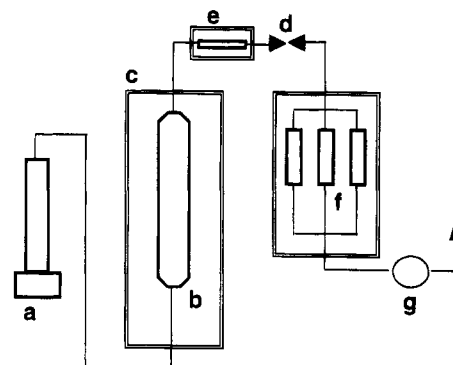
Figure 1. Supercritical extraction apparatus: a, syringe pump for supercritical carbon dioxide; b, extraction vessel; c, forced-air circulation bath; d, heated restrictor; e, lipid collection trap; f, Tenax adsorption traps; g, gas flow totalizer.

elucidate the chemical changes that occur upon cooking and add to our understanding of the chemistry and mechanisms forming beef aroma and flavor compounds.

MATERIALS AND METHODS

Sample Preparation. Although variation from muscle location may exist, only the longissimus muscle was used because this one represents cuts of most value to the meat producer and has been studied most in other research on meat quality. Cross sections of longissimus muscle from both 8th and 12th rib sections from each of two commercially produced, choice grade beef steers were removed 24 h postmortem and subjected to a 2 × 2 factorial design in which packaging method and cooking method were evaluated. Right-side rib sections were vacuum-packaged and held at 4 °C for 28 days. The left-side rib sections were wrapped in freezer paper and held at 4 °C to dry-age for 14 days. Aging times for vacuum-packaged and dry-aged beef were consistent with commercial practice. Portions of each rib section were oven-roasted at 175 °C to an internal temperature of 70 °C or microwave-cooked to the same internal temperature. Samples of cooked beef were taken from a core of each roast after approximately 2 cm was trimmed from all surfaces. Immediately following cooking and trimming, cores were frozen in liquid nitrogen, pulverized in a Waring Blendor, vacuum-packaged in oxygen-impermeable bags, and stored at −70 °C until extraction.

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Table 1. Weight Percentages of Volatile Compounds Extracted from Cooked Beef with Supercritical CO_2[a]

				oven roasted		microwave cooked	
peak no.	Kovats index[b]	retention time (min)	compound	dry aged, mean (SD)	vacuum pack, mean (SD)	dry aged, mean (SD)	vacuum pack, mean (SD)
			Hydrocarbons				
8	700	9.16	heptane	18.82 (6.03)[d]	13.70 (5.24)[c]	10.42 (1.19)[b]	4.77 (1.23)[a]
15	800	13.52	octane	0.00 (0.00)	0.18 (0.18)	0.17 (0.30)	0.00 (0.00)
18	899	17.56	1-nonene	0.00 (0.00)	0.00 (0.00)	0.01 (0.02)	0.06 (0.06)
27	997	23.06	1-decene	0.80 (0.19)	1.48 (0.51)	0.72 (0.48)	1.69 (0.11)
28	1000	23.28	decane	0.65 (0.07)	0.96 (0.27)	0.72 (0.10)	0.48 (0.21)
39	1100	28.08	undecane	0.26 (0.01)	0.20 (0.20)	0.35 (0.03)	0.22 (0.05)
44	1187	30.56	1-dodecene	0.00 (0.00)[a]	0.83 (0.17)[b]	0.07 (0.11)[a]	1.44 (0.17)[b]
47	1200	32.16	dodecane	1.65 (0.15)	1.27 (0.26)	2.18 (0.50)	1.00 (0.13)
56	1300	37.10	tridecane	0.16 (0.13)	0.28 (0.04)	0.28 (0.07)	0.29 (0.29)
58	1336	38.36	1-tetradecene	0.54 (0.46)[b]	0.18 (0.01)[ab]	0.12 (0.16)[a]	0.23 (0.00)[ab]
63	1400	40.08	tetradecane	1.81 (0.34)[a]	3.20 (0.21)[b]	3.51 (0.96)[b]	1.87 (0.30)[a]
67	1500	44.06	pentadecane	0.09 (0.09)[a]	0.38 (0.05)[b]	0.23 (0.19)[ab]	0.74 (0.07)[c]
68	1506	44.24	2,4-dimethyl-1-decene	0.22 (0.09)[b]	0.00 (0.00)[a]	0.20 (0.07)[b]	0.00 (0.00)[a]
73	1551	45.44	1-hexadecene	0.01 (0.01)[a]	0.39 (0.05)[bc]	0.48 (0.17)[c]	0.26 (0.09)[b]
74	1570	46.22	2,6,10-trimethyldodecane	0.14 (0.02)[ab]	0.07 (0.02)[a]	0.29 (0.15)[b]	0.14 (0.01)[ab]
76	1600	47.08	hexadecane	1.70 (0.56)[a]	3.44 (0.47)[ab]	4.31 (2.16)[b]	2.42 (0.30)[ab]
79	1700	49.48	1-heptadecene	0.31 (0.09)	0.37 (0.08)	0.23 (0.17)	0.46 (0.01)
80	1751	50.00	heptadecane	0.29 (0.10)[a]	0.54 (0.23)[ab]	0.48 (0.10)[ab]	1.10 (0.12)[b]
85	1757	51.50	1-octadecene	0.30 (0.07)[ab]	0.40 (0.05)[bc]	0.22 (0.08)[a]	0.47 (0.14)[c]
90	1800	53.36	octadecane	1.17 (0.40)[a]	1.92 (0.33)[b]	2.41 (0.22)[c]	2.35 (0.64)[c]
97	1900	56.26	nonadecane	0.54 (0.15)[a]	0.56 (0.04)[a]	0.58 (0.21)[a]	0.97 (0.20)[b]
101	1942	57.54	1-eicosene	0.39 (0.18)[a]	0.34 (0.07)[a]	0.64 (0.16)[b]	0.51 (0.17)[ab]
103	2000	59.36	eicosane	3.74 (2.26)	1.27 (0.34)	1.35 (0.70)	3.07 (0.53)
107	2100	63.06	henicosane	0.44 (0.09)[b]	0.04 (0.01)[a]	0.34 (0.15)[b]	0.13 (0.01)[a]
			total	**34.04 (2.53)[b]**	**31.99 (3.23)[b]**	**30.30 (5.17)[b]**	**24.68 (2.08)[a]**
			Aldehydes				
9	703	9.24	pentanal	5.56 (1.07)[b]	3.26 (0.12)[ab]	2.95 (2.07)[ab]	0.58 (0.58)[a]
14	791	13.10	hexanal	6.88 (1.65)[b]	3.76 (0.60)[a]	2.36 (0.87)[a]	3.11 (1.68)[a]
19	900	18.38	heptanal	1.00 (0.33)[a]	2.77 (0.76)[b]	0.92 (0.64)[a]	2.12 (0.28)[ab]
23	963	21.16	2-heptenal	0.03 (0.05)[a]	0.38 (0.16)[c]	0.04 (0.07)[ab]	0.17 (0.17)[b]
29	1003	23.38	octanal	1.67 (0.40)[a]	4.60 (0.04)[c]	1.52 (0.68)[a]	4.19 (0.72)[b]
31	1027	24.00	2-ethyl-2-hexenal	0.24 (0.05)[a]	0.08 (0.02)[b]	0.08 (0.08)[b]	0.09 (0.00)[b]
34	1063	26.18	2-octenal	0.09 (0.03)[a]	0.92 (0.05)[b]	0.13 (0.11)[a]	0.78 (0.29)[b]
40	1106	28.24	nonanal	3.29 (0.83)	3.50 (0.24)	3.39 (1.11)	4.18 (0.65)
43	1155	30.42	2-nonenal	0.39 (0.27)[b]	0.25 (0.09)[ab]	0.11 (0.04)[a]	0.16 (0.05)[ab]
48	1208	32.44	decanal	0.56 (0.11)	0.41 (0.16)	0.57 (0.19)	0.43 (0.05)
54	1267	34.24	2-decenal	0.08 (0.08)[a]	1.72 (0.29)[b]	0.33 (0.19)[a]	3.12 (0.35)[c]
57	1311	37.28	undecanal	0.09 (0.02)[a]	0.08 (0.03)[a]	0.21 (0.09)[b]	0.21 (0.04)[b]
60	1367	39.12	2-undecenal	0.43 (0.04)[a]	1.57 (0.43)[b]	0.36 (0.14)[a]	2.17 (0.15)[b]
69	1513	44.32	tridecanal	0.10 (0.10)	0.17 (0.05)	0.15 (0.27)	0.09 (0.02)
77	1619	47.50	tetradecanal	0.93 (1.01)	0.21 (0.06)	0.46 (0.07)	0.19 (0.19)
82	1733	51.06	pentadecanal	1.23 (0.47)	0.98 (0.01)	0.90 (0.44)	1.07 (0.07)
93	1822	53.52	hexadecanal	6.33 (2.57)[a]	6.32 (1.14)[a]	13.10 (1.78)[b]	7.15 (0.66)[a]
105	2035	60.08	9-octadecenal	0.00 (0.00)[a]	1.11 (0.51)[b]	1.47 (0.40)[b]	1.31 (0.13)[b]
106	2366	60.40	octadecanal	3.50 (1.37)[a]	3.70 (0.61)[a]	7.02 (0.90)[b]	4.34 (0.55)[a]
			total	**32.39 (1.96)**	**35.79 (4.99)**	**36.07 (4.34)**	**35.47 (2.38)**
			Alcohols				
5	652	7.28	1-butanol	0.67 (0.10)[c]	0.37 (0.02)[b]	0.29 (0.10)[b]	0.15 (0.01)[a]
6	663	8.34	2-methyl-1-propanol	0.20 (0.06)[a]	0.71 (0.24)[b]	0.25 (0.15)[a]	0.38 (0.05)[ab]
7	687	8.48	2-(propenyloxy)ethanol	0.56 (0.09)[b]	0.13 (0.02)[a]	0.22 (0.07)[a]	0.19 (0.12)[a]
11	763	11.50	1-pentanol	1.44 (0.44)[b]	0.09 (0.05)[a]	0.68 (0.10)[a]	0.00 (0.00)[a]
13	788	12.44	1,3-butanediol	0.61 (0.48)	0.14 (0.07)	0.54 (0.43)	0.02 (0.02)
16	871	16.46	1-hexanol	0.09 (0.03)[a]	0.21 (0.02)[b]	0.06 (0.04)[a]	0.15 (0.05)[ab]
22	947	20.20	2-(2-methoxyethoxy)ethanol	0.21 (0.09)	0.51 (0.10)	0.31 (0.34)	0.03 (0.03)
24	976	22.00	heptanol	0.08 (0.05)	0.72 (0.02)	0.46 (0.80)	0.94 (0.24)
25	984	22.00	1-octen-3-ol	0.28 (0.16)	0.16 (0.13)	0.28 (0.44)	0.28 (0.26)
32	1034	24.50	2-ethyl-1-hexanol	0.38 (0.09)[ab]	0.38 (0.00)[ab]	0.41 (0.16)[b]	0.10 (0.02)[a]
33	1039	25.10	2,4-heptadien-1-ol	0.15 (0.03)	0.04 (0.04)	0.06 (0.08)	0.05 (0.00)
35	1074	26.50	1-octanol	0.25 (0.07)[a]	0.36 (0.36)[a]	0.17 (0.07)[a]	1.26 (0.16)[b]
37	1095	27.16	α-dimethylbenzenemethanol	0.00 (0.00)[a]	0.19 (0.19)[ab]	0.27 (0.15)[b]	0.46 (0.03)[b]
42	1133	29.12	nonanol	0.02 (0.03)[a]	0.37 (0.13)[b]	0.06 (0.04)[a]	0.03 (0.03)[a]
46	1195	31.56	2-(2-butoxyethoxy)ethanol	0.00 (0.00)	0.52 (0.52)	0.60 (0.71)	0.92 (0.23)
50	1224	33.16	1-decanol	0.11 (0.11)	0.14 (0.02)	0.19 (0.10)	0.06 (0.01)
64	1465	43.02	1-dodecanol	0.20 (0.13)	0.17 (0.11)	0.21 (0.11)	0.17 (0.03)
75	1577	46.30	1-tridecanol	0.14 (0.08)[a]	0.19 (0.03)[ab]	0.35 (0.16)[b]	0.23 (0.03)[ab]
89	1792	53.24	1-pentadecanol	0.37 (0.12)	0.15 (0.03)	0.20 (0.12)	0.33 (0.06)
102	1949	1743	1-heptadecanol	0.14 (0.10)[a]	0.32 (0.06)[b]	0.32 (0.04)[b]	0.38 (0.05)[b]
104	2005	59.56	1-octadecanol	0.67 (0.21)	0.82 (0.25)	2.42 (1.52)	1.00 (0.05)
			total	**6.57 (0.58)**	**6.71 (0.99)**	**8.33 (2.74)**	**7.13 (0.86)**

Effect of Packaging Method on Beef Volatile Compounds

J. Agric. Food Chem., Vol. 43, No. 3, 1995 **775**

Table 1. (Continued)

peak no.	Kovats index[b]	retention time (min)	compound	oven roasted		microwave cooked	
				dry aged, mean (SD)	vacuum pack, mean (SD)	dry aged, mean (SD)	vacuum pack, mean (SD)
			Phenols				
65	1475	43.44	BHA	0.87 (0.07)	0.78 (0.13)	1.13 (0.28)	0.63 (0.08)
71	1538	44.52	BHT	0.15 (0.06)	0.28 (0.07)	0.17 (0.22)	0.28 (0.05)
84	1745	51.22	4-nonylphenol	0.11 (0.11)	0.03 (0.03)	0.24 (0.15)	0.17 (0.02)
			total	**1.12 (0.23)**	**1.09 (0.23)**	**1.53 (0.38)**	**1.08 (0.06)**
			Ketones				
10	744	10.30	acetoin	0.56 (0.25)[b]	0.15 (0.12)[ab]	0.23 (0.20)[ab]	0.01 (0.01)[a]
26	986	22.36	2,3-octanedione	0.56 (0.23)	0.49 (0.32)	0.29 (0.27)	0.84 (0.30)
38	1097	27.58	7-octen-2-one	0.03 (0.05)[a]	0.37 (0.01)[b]	0.05 (0.08)[a]	0.19 (0.19)[ab]
53	1256	34.3	cyclononanone	0.00 (0.00)[a]	0.06 (0.06)[b]	0.00 (0.00)[a]	0.13 (0.04)[c]
81	1717	50.24	2-pentadecanone	0.74 (0.07)[ab]	0.36 (0.15)[a]	0.85 (0.27)[ab]	1.15 (0.59)[b]
94	1850	55.02	6,10,14-trimethyl-2-pentadecanone	2.72 (0.68)	2.76 (0.55)	2.97 (1.40)	3.72 (0.68)
98	1906	56.30	2-heptadecanone	2.71 (0.78)	2.86 (0.25)	2.84 (1.29)	4.08 (0.71)
			total	**7.31 (1.17)[a]**	**7.05 (1.21)[a]**	**7.22 (2.53)[a]**	**10.11 (0.26)[b]**
			Lactones				
61	1370	39.24	γ-nonalactone	0.22 (0.03)[a]	0.23 (0.23)[a]	0.32 (0.15)[a]	0.86 (0.24)[b]
70	1519	44.46	γ-decalactone	1.64 (1.37)	0.19 (0.06)	0.48 (0.29)	0.11 (0.03)
78	1679	49.04	δ-decalactone	0.06 (0.06)	0.25 (0.06)	0.16 (0.13)	0.32 (0.04)
			total	**1.91 (1.43)**	**0.67 (0.11)**	**0.95 (0.34)**	**1.28 (0.31)**
			Furans				
20	908	19.06	dihydro-4,5-dimethyl-2(3H)-furanone	0.23 (0.10)[b]	0.27 (0.06)[b]	0.07 (0.08)[ab]	0.00 (0.00)[a]
49	1215	32.58	4-(1-methylethyl)-4-methylfuran	0.12 (0.14)	0.04 (0.04)	0.13 (0.13)	0.10 (0.02)
51	1241	34.04	2-propyl-4-methylfuran	0.14 (0.06)	0.06 (0.03)	0.11 (0.03)	0.08 (0.02)
			total	**0.49 (0.21)**	**0.37 (0.00)**	**0.31 (0.21)**	**0.19 (0.03)**
			Carboxylic Acids				
3	602	5.26	acetic acid	3.41 (1.12)[b]	0.87 (0.49)[a]	0.39 (0.07)[a]	0.15 (0.05)[a]
4	641	6.22	acetate anhydride	0.00 (0.00)[a]	0.13 (0.08)[ab]	0.18 (0.12)[b]	0.12 (0.03)[ab]
12	783	12.40	butanoic acid	0.26 (0.07)[a]	0.82 (0.19)[b]	0.20 (0.04)[a]	0.27 (0.13)[a]
17	892	17.34	pentanoic acid	0.02 (0.04)[a]	0.18 (0.12)[b]	0.05 (0.05)[a]	0.19 (0.06)[b]
30	1007	23.44	hexanoic acid	1.45 (0.70)	1.28 (0.17)	1.16 (0.76)	0.36 (0.36)
36	1091	27.00	heptanoic acid	0.23 (0.03)[a]	1.28 (1.27)[b]	0.23 (0.14)[a]	1.49 (0.21)[b]
45	1189	31.04	octanoic acid	0.09 (0.08)[a]	2.31 (1.91)[b]	0.19 (0.12)[a]	3.93 (0.01)[b]
55	1288	35.24	nonanoic acid	0.25 (0.36)[a]	1.43 (1.43)[a]	0.55 (0.62)[a]	4.74 (0.30)[b]
59	1357	38.58	decanoic acid	0.10 (0.01)[a]	0.36 (0.07)[d]	0.16 (0.06)[b]	0.23 (0.01)[c]
72	1546	45.18	dodecanoic acid	0.19 (0.05)[a]	0.26 (0.06)[ab]	0.32 (0.07)[b]	0.21 (0.05)[ab]
88	1786	53.00	tetradecanoic acid	0.43 (0.23)[a]	0.31 (0.11)[a]	0.66 (0.03)[b]	0.67 (0.05)[b]
			total	**6.44 (1.24)[a]**	**9.22 (4.64)[ab]**	**4.10 (1.35)[a]**	**12.35 (0.04)[b]**
			Esters				
2	588	5.14	ethyl acetate	1.58 (0.22)[c]	1.17 (0.10)[bc]	0.84 (0.41)[ab]	0.57 (0.07)[a]
41	1126	28.50	2-ethylhexanoic acid	0.04 (0.06)	0.16 (0.16)	0.10 (0.07)	0.00 (0.00)
62	1385	39.48	2-(methylheptyl)propanoic acid ester	0.43 (0.07)	0.51 (0.02)	0.44 (0.21)	0.56 (0.05)
66	1493	43.44	benzoic acid 2-propenyl ester	0.51 (0.08)[a]	0.00 (0.00)[a]	0.87 (0.25)[b]	0.00 (0.00)[a]
83	1744	51.22	tetradecanoic acid methyl ester	1.22 (0.49)	0.14 (0.02)	1.04 (0.68)	0.22 (0.05)
100	1924	57.18	hexadecanoic acid methyl ester	1.28 (0.65)	1.38 (0.40)	2.26 (0.54)	2.18 (0.32)
			total	**5.05 (0.24)[b]**	**3.36 (0.46)[a]**	**5.54 (1.16)[b]**	**3.53 (0.16)[a]**
			Terpenoids				
86	1755	52.08	pristane	0.24 (0.07)[a]	0.44 (0.08)[b]	0.90 (0.09)[c]	0.48 (0.11)[b]
87	1777	52.42	phyt-1-ene	0.36 (0.28)	0.25 (0.01)	0.57 (0.18)	0.63 (0.20)
91	1804	53.50	phytane	0.15 (0.15)[a]	0.23 (0.05)[ab]	0.33 (0.05)[c]	0.30 (0.02)[bc]
92	1811	53.28	phyt-2-ene	0.23 (0.05)[a]	0.20 (0.02)[a]	0.38 (0.09)[b]	0.26 (0.04)[ab]
95	1881	55.54	farnesol	0.80 (0.28)[b]	0.44 (0.06)[a]	0.64 (0.17)[ab]	0.57 (0.08)[ab]
96	1895	56.24	phytol	0.51 (0.12)[a]	0.66 (0.09)[ab]	0.88 (0.08)[bc]	1.12 (0.41)[c]
			total	**2.29 (0.90)[a]**	**2.22 (0.31)[a]**	**3.70 (0.21)[b]**	**3.36 (0.86)[b]**
			Sulfur Compounds				
21	924	18.22	dimethyl sulfone	0.00 (0.00)[a]	0.28 (0.25)[b]	0.02 (0.04)[a]	0.02 (0.02)[a]
52	1245	34.24	benzothiazole	0.17 (0.06)[ab]	0.06 (0.03)[b]	0.30 (0.22)[a]	0.06 (0.03)[a]
			total	**0.17 (0.06)**	**0.34 (0.22)**	**0.33 (0.24)**	**0.08 (0.05)**
			Miscellaneous				
1	520	4.18	dichloromethane	1.99 (0.40)[b]	0.94 (0.16)[a]	1.35 (0.35)[ab]	0.49 (0.00)[a]
99	1917	57.04	1,2-benzenedicarboxylic acid	0.22 (0.22)	0.24 (0.15)	0.26 (0.24)	0.26 (0.20)
			total	**2.21 (0.25)[b]**	**1.18 (0.00)[ab]**	**1.62 (0.57)[ab]**	**0.75 (0.20)[a]**

[a] Data are means of duplicate experiments. Means bearing different superscripts within a row differ significantly ($P < 0.05$). [b] Kovats indices were determined as previously described (King et al., 1993).

SC-CO$_2$ Equipment and Extraction. The extraction methods have been described (King et al., 1993). Some modifications in equipment were made before extraction and analysis of the cooked and vacuum-packaged products. The supercritical CO$_2$ extraction apparatus is shown in Figure 1. For the present experiments, SFE grade CO$_2$ was pressurized to the desired pressure using an Isco Micro-LC 500 high-pressure syringe pump. This pump holds 50 cm^3 of liquefied CO$_2$ and pumps the entire volume with one long, smooth piston stroke. The flow rate can be controlled accurately between 0.6 and 0.06 cm^3/min.

Extraction temperature was 35 °C, which was maintained with a forced-air circulation oven which housed CO$_2$ preheat tubing and the extraction vessel. The extraction vessel had a volume of 10 cm^3 and held 1–2 g of sample. Approximately 0.5 g of sample was mixed with the same amount of calcium sulfate and placed in the supercritical extraction vessel. Calcium sulfate was added to trap moisture and prevent it from depositing with the extracted materials. Extraction was performed in duplicate at 35 °C and 6000 psia. Extraction continued until approximately 50 cm^3 of CO$_2$ had been passed over the beef. This gave a "treatment ratio" of 30 g of CO$_2$/g of beef.

The extract was cooled at point e (Figure 1), which caused most of the lipid material to condense. The remaining extract contained mostly the volatile compounds in the supercritical phase at high pressure and subsequently flowed through the restrictor where the pressure was reduced to 1 atm. To prevent the extract from freezing upon expansion, the restrictor was placed in an aluminum block that was independently heated to 50 °C. Downstream of the restrictor, at point f (Figure 1), the noncondensable volatile fraction was adsorbed onto Tenax TA (polymer adsorbent, 60–80 mesh, Alltech Associates, Inc.) in a series of three parallel traps that were cooled in an ice bath.

Three other methods of capturing the volatile compounds were investigated: (1) Collect gas phase samples of the extract after the pressure restrictor. (2) Capture the volatile compounds by dry deposition in a cold finger chilled in dry ice. (3) Collect the volatile compounds by bubbling the low-pressure extract through vials of organic solvent. None of these three techniques captured sufficient volatile compounds to give an adequate signal by GC analysis. Therefore, use of Tenax or other suitable adsorbent material to capture extracted volatile compounds is strongly recommended.

Before extraction, Tenax TA was conditioned at 275 °C for 4 h as described previously (King et al., 1993). To collect volatile compounds, 100 mg of loose-packed Tenax TA was placed in parallel in three glass liners (40 cm long × 6 mm wide) at point f (Figure 1). CO$_2$ and volatile compounds flowed through the adsorption tube where volatile compounds were retained. Three parallel glass liners were used so that replicate GC/MS analyses could be made. After each run, two Tenax samples were analyzed by GC/FID immediately and the other Tenax was capped and stored in a freezer (−20 °C) until analyzed by GC/MS. Mass spectrometry was used to identify the compounds, and FID analysis was used to quantify peak areas. This was followed by statistical analysis (analysis of variance) to compare samples, compound by compound, as well as chemical class by chemical class. Instruments, GC conditions, and other details have been reported (King et al., 1993).

RESULTS AND DISCUSSION

Routine analyses for moisture and lipids were performed. Oven-roasted beef that had been vacuum-packaged had a higher moisture content than dry-aged or microwave-cooked roasts. No packaging method differences in moisture content were found, and no differences in lipid content were observed.

Table 1 lists the compounds extracted from the oven-roasted and microwave-cooked samples that had been dry-aged or vacuum-packaged. Analogous results from raw, ground beef were reported by King et al. (1993). One hundred seven compounds were identified in the cooked beef samples, including 24 hydrocarbons, 6 terpenoids, 19 aldehydes, 21 alcohols, 3 phenols, 7 ketones, 3 lactones, 3 furans, 11 acids, 6 esters, 2 S-containing compounds, and 2 miscellaneous compounds.

Of the hydrocarbons identified, heptane was present in the greatest proportion regardless of cooking or packaging method (Table 1). This hydrocarbon was greatest ($P < 0.05$) for oven-cooked beef but decreased ($P < 0.05$) when the beef had been vacuum-packaged instead of dry-aged. Heptane can arise from autoxidation of oleate (Grosch, 1987); oleate is the major fatty acid in bovine muscle (Christie, 1981). Generally, n-hydrocarbons develop from reaction of hydrogen free radicals with alkyl free radicals (hydroperoxide decomposition products) (Selke et al., 1975). Dry-aged beef cooked by oven roasting had the greatest exposure to air, whereas microwave-cooked, vacuum-packaged beef had the least exposure to air. Several of the other hydrocarbons observed were affected by either cooking method, aging method, or both. For example, the appearance of octane was transient, and values were highly variable. 1-Dodecene was found in low to nondetectable quantities in dry-aged beef samples and was significantly greater ($P < 0.05$) in vacuum-packaged samples. 1-Tetradecene was highest ($P < 0.05$) for dry-aged, oven-roasted beef and lowest for dry-aged, microwave-cooked beef. Tetradecane, 1-hexadecene, and hexadecene did not vary consistently with any treatment main effect, but when beef samples were cooked by oven roasting, these compounds were highest ($P < 0.05$) from vacuum-packaged beef. The reverse was true for microwave cooking, after which dry-aged beef had higher proportions than vacuum-packaged. Pentadecane and heptadecane were higher ($P < 0.05$) in vacuum-packaged beef, regardless of cooking method, which was the opposite of that observed for 2,4-dimethyl-1-decene, 2,6,10-trimethyldodecane, and heneicosane, all of which were higher ($P < 0.05$) for dry-aged beef. Octadecane was lower ($P < 0.05$) for dry-aged and vacuum-packaged oven-roasted beef when compared to microwave-cooked beef.

Total aldehydes were not influenced by packaging or cooking methods. Different proportions of the aldehydes identified, however, were observed. Pentenal was highest for oven-roasted beef and, regardless of cooking method, vacuum packaging resulted in lower pentenal weight percentages. Hexanal and 2-ethyl-2-hexanal were highest ($P < 0.05$) for dry-aged, oven-cooked beef; hexanal would develop from thermal oxidation of linoleic acid (Frankel et al., 1961). Several aldehydes were lower ($P < 0.05$) for dry-aged than vacuum-packaged beef without much influence of cooking method: heptanal, 2-heptenal, octanal, 2-octenal, 2-decenal, and 2-undecenal. On the other hand, undecanal, hexadecanal, 9-octadecenal, and octadecanal were higher ($P < 0.05$) in microwave-cooked beef. The latter three aldehydes were higher ($P < 0.05$) in dry-aged than in vacuum-packaged beef, but only for microwave-cooked samples.

Development of several alcohols was affected by treatment (Table 1). 1-Butanol was lower ($P < 0.05$) for vacuum-packaged than for dry-aged samples. Similar changes were observed for 1-pentanol and 1,3-butanediol. The opposite effect was observed for 1-hexanol, for which vacuum-packaged beef had the highest ($P < 0.05$) proportion regardless of cooking method. For 2-ethyl-1-hexanol, proportions were reduced ($P < 0.05$)

Effect of Packaging Method on Beef Volatile Compounds

J. Agric. Food Chem., Vol. 43, No. 3, 1995 **777**

only by microwave cooking of vacuum-packaged samples. Conversely, 1-heptadecanol was lowest ($P < 0.05$) in oven-roasted, dry-aged samples. Proportions of 1-tridecanol increased ($P < 0.05$) during microwave cooking of dry-aged samples.

Oxidative degradation of low molecular weight fatty acids, aldehydes, and alcohols is thought to contribute to lactone formation (Watanabe and Sato, 1971); however, little treatment variation was observed for proportions of lactones or for phenols (Table 1). Several individual ketones were influenced by treatment, notably 7-octen-2-one which was highest ($P < 0.05$) in oven-roasted samples that had been vacuum-packaged prior to cooking. Cyclononanone was barely traceable unless samples were vacuum-packaged prior to microwave cooking. Autoxidation of 18-carbon unsaturated fatty acids contributes to formation of compounds such as methyl ketones (Thomas et al., 1971). Of the furans, only dihydro-4,5-dimethyl-2(3H)-furanone was developed by oven-roasting of beef.

For dry-aged, oven-roasted beef, acetic acid was higher ($P < 0.05$) than for the other treatments and, within this treatment, was severalfold higher than the other acids. Pentanoic, heptanoic, octanoic, and nonanoic acid weight percentages were lowest ($P < 0.05$) for dry-aged beef regardless of cooking method, suggesting that exposure to air before exposure to heat was responsible for their development. Except for acetic acid, these acids have been observed as autoxidation products of oleate (Grosch, 1987). The reason for the higher proportion of acetic acid in dry-aged, oven-roasted beef is not clear but may be due in part to development of short-chain aldehydes (Selke et al., 1975).

Total esters were highest ($P < 0.05$) when beef had been dry-aged before cooking. Ethyl acetate was highest in beef that was oven-roasted. 2-Propenyl ester of benzoic acid was detected only in samples dry-aged before cooking and was not affected by cooking method.

Total terpenoids were influenced by cooking method but not by packaging method. This also was observed for phytane, phytol, and phyt-2-ene. Effects on terpenoids were induced by treatment since these compounds would have come from the diet fed the cattle (Urbach and Stark, 1975).

Of the sulfur compounds identified, dimethyl sulfone had relative abundance only in oven-roasted beef that had been vacuum-packaged. Dichloromethane was identified but was greatest ($P < 0.05$) in oven-roasted beef that was dry-aged.

Results of analysis of data pooled to compare cooking method effects on development of volatile compounds are shown in Table 2. Microwave-cooked beef had lower percentages of total hydrocarbons and a higher percentage of terpenoids than beef cooked by using dry heat; terpenoids contribute a "grassy" flavor to meat. Packaging method effects on development of major volatile compounds pooled for cooking method are shown in Table 3. Vacuum-packaged beef had a higher proportion of total acids but lower proportions of esters, hydrocarbons, and miscellaneous compounds than dry-aged beef. In general, vacuum-packaging prevented oxidation of the beef and reduced acid losses.

Thirty-three compounds not previously identified in raw, ground beef (King et al., 1993) were found in extracts of cooked beef in the present study. Moreover, relative proportions of hydrocarbons, aldehydes, ketones, and esters were higher in extracts of roasted beef

Table 2. Effect of Cooking Methods on Weight Percentages of Volatile Components of Beef[a]

	cooking method	
volatile component	conventional	microwave
hydrocarbons (%)	33.36 (3.23)	28.43 (5.61)
terpenoids (%)	2.27 (0.83)[a]	3.58 (0.60)[b]
aldehydes (%)	33.53 (4.01)	35.87 (4.17)
alcohols (%)	6.56 (0.72)	7.93 (2.58)
phenols (%)	1.11 (0.25)	1.39 (0.42)
ketones (%)	7.22 (1.30)	8.18 (2.72)
lactones (%)	1.50 (1.43)	1.06 (0.40)
furans (%)	0.45 (0.20)	0.27 (0.20)
acids (%)	7.43 (3.39)	6.85 (4.42)
esters (%)	4.49 (0.95)	4.87 (1.48)
sulfur compounds (%)	0.23 (0.17)	0.24 (0.25)
miscellaneous compounds (%)	1.87 (0.57)	1.33 (0.69)

[a] Data are means of duplicate experiments (standard deviation) pooled for packaging method. Means bearing different superscripts within a row differ significantly ($P < 0.05$).

Table 3. Effect of Packaging Methods on Weight Percentages of Volatile Components of Beef[a]

	packaging method	
volatile component	dry aged	vacuum pack
hydrocarbons (%)	32.17 (4.79)	28.34 (5.26)
terpenoids (%)	2.99 (1.02)	2.79 (0.99)
aldehydes (%)	34.23 (4.10)	35.63 (4.51)
alcohols (%)	7.45 (2.31)	6.82 (1.02)
phenols (%)	1.33 (0.40)	1.09 (0.20)
ketones (%)	7.26 (2.11)	8.58 (2.04)
lactones (%)	1.43 (1.23)	0.98 (0.44)
furans (%)	0.40 (0.25)	0.28 (0.11)
acids (%)	5.27 (1.86)[a]	10.88 (4.01)[b]
esters (%)	5.30 (0.94)[b]	3.44 (0.40)[a]
sulfur compounds (%)	0.25 (0.21)	0.21 (0.24)
miscellaneous compounds (%)	1.91 (0.57)[b]	0.97 (0.30)[a]

[a] Data are means of duplicate experiments (standard deviation) pooled for cooking method. Means bearing different letters within row differ significantly ($P < 0.05$).

(oven-roasted and microwave-cooked pooled) than in raw ground beef as reported by King et al. (1993). On the other hand, in raw ground beef, terpenoids, phenols, and sulfur compounds were substantially more prevalent.

Development of meat flavor by cooking, and flavor differences in meat caused by various preparation procedures, could occur from changes in proportions of many of the compounds observed in this study, in particular aldehydes, ketones, and terpenoids. Flavor characteristics of numerous aldehydes and ketones such as those observed in the present study were discussed previously (Grosch, 1987). Data from the present study indicate that differences due to cooking and packaging methods exist for many aldehydes and ketones, and very likely for several acids, esters, and terpenoids.

Several studies have reported GC/MS data from extracts of meat. Ramarathnam et al. (1991) compared spectral data obtained from cured and uncured pork and found differences in several compounds, most notably in hexanal, but in other carbonyl compounds as well. Larick et al. (1987) evaluated beef flavor in volatile compounds of head space samples from fat obtained from cattle fed either forage or grain. These researchers reported data for several types of compounds that were observed in the present study, namely hydrocarbons, aldehydes, acids, ketones, lactones, and terpenoids. Several of the same compounds reported by Larick et al. (1987) were observed in the present study, but variation in several other compounds between the two studies also occurred. Volatiles from samples of beef that were prepared similarly to those used in the

778   *J. Agric. Food Chem.,* Vol. 43, No. 3, 1995

King et al.

present study were reported by Liebich et al. (1972). In comparison with their study we identified numerous compounds that were observed in either roasted or boiled beef by Leibich et al. (1972). These included 9 hydrocarbons, 11 aldehydes, 8 alcohols, 2 ketones, and 2 lactones; benzothiazol and dimethyl sulfone were only observed in boiled beef, and no acids, esters, or terpenoids were reported by Leibich et al. (1972). The major difference in the present study and those by Liebich et al. (1972) and Ramarathnam et al. (1991) was the use of supercritical CO_2 in the present study.

CONCLUSION

The various combinations of volatile compounds present in cooked meat impart the flavor attributes of beef. Differences in relative proportions of numerous volatile compounds were observed in beef cooked by conventional or microwave methods as well as for dry-aged or vacuum-packaged beef. Differences in relative proportions of certain hydrocarbons, terpenoids, aldehydes, and ketones could be responsible for differences in meat flavor that occur when oven-roasting compared to microwave-cooking. Future flavor studies should emphasize changes in beef flavor when individual and combinations of these compounds are applied to beef samples and evaluated by means that include sensory tests. The present study provides a data base that, along with results of others, can be used to delve further into elucidation of the chemistry of beef flavor or the elucidation of changes in beef flavor that occur with variations in product preparation.

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