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Effect of temperature on the analysis of beef flavor volatiles: Focus on carbonyl and sulfur-containing compounds

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This paper demonstrates that the volatile flavor profiles from cooked and cooked/stored ground beef are directly affected by and related to the purge temperature for volatile isolation. Minimal analytical efficacy and recovery are seen when the samples are purged at 50°C. This is thought to arise from inefficient extraction of flavor volatiles. Different and potentially misleading chromatographic profiles are obtained when the samples are purged at 100°C. This response is thought to be due to conversion of one volatile species to another. Optimal extraction and limited conversion of the volatiles are seen at a temperature of 75°C. The data clearly suggest that a more accurate picture of food volatile composition and, therefore, potential flavor can best be appreciated by a thorough examination and understanding of the effect of temperature on the development and content of these volatile mixtures.

INTRODUCTION

The mild, serum-like flavor of raw/uncooked meat does not resemble that of its cooked counterpart. Although the chemical compositions, and thereby flavor, of raw and cooked meat differ (Spanier *et al.*, 1988), they initially contain the same flavor-producing precursors. On heating/cooking, these precursors react, producing the series of complex volatile and non-volatile mixtures which are characteristic of meat aroma (Wasserman, 1979) and taste (Spanier *et al.*, 1990; Spanier & Miller, 1993). The relative levels and types of precursors present in the meat are dependent upon several variables: age, breed, and sex of the animal, as well as the feeding regimen, the manner of slaughter, the method of storage after slaughter, and the muscle primal cut (Spanier & Miller, 1993).

Meat is a complex structural matrix (Spanier & Miller, 1993) composed of proteins, carbohydrates, fats, water, and, to a lesser extent, some vitamins and other organic and inorganic components. A thorough knowledge of the flavor of meat, therefore, requires a thorough understanding of the precursors available for reaction, their compartmentation within the muscle structure, and the manner in which the meat is cooked/heated. Several methods have been employed (Risch & Reineccius, 1989) in the extraction of flavor volatiles from their chemical and physical bonds in the complex muscle matrix. These methods include steam distillation, solvent extraction, headspace analysis and supercritical fluid extraction. Each method has specific advantages and shortcomings when used in the isolation of numerous aromatic compounds, such as carbonyls, thiols, thiazoles, pyrazines, furans, and sulfur-containing compounds (Teranishi *et al.*, 1981; Drumm & Spanier, 1991). The mechanisms responsible for the formation of these flavor volatiles have been proposed and developed based on such experimentation (Manley, 1989; Parliment *et al.*, 1989).

All methods for extraction/isolation of flavor volatiles involve some form of heating for the volatilization of the compounds from the sample into a headspace collection region or trapping matrix or for elution and movement of the compounds from the adsorbent column. The temperatures used for sparging, purging, and thermal desorbing may result in either thermal degradation of some of the compounds present or in generation of new compounds from existing and

cooking-derived precursors. Since a detailed gas chromatographic analysis may result in 200–1000 peaks (MacLeod & Ames, 1986; Gasser & Grosch, 1988), the investigator must use caution in judging the validity of the results of these volatiles and interpreting the data. For example, Drumm and Spanier (1991) recently demonstrated that several sulfur compounds in cooked ground beef [4-methylthiazole, 2-acetylthiazole, benzothiazole, and 2-furylmethanethiol] were fairly stable during periods of refrigerated storage. These compounds were identified in ground beef patties that had undergone a four-hour steam distillation—extraction protocol. These data contradict the results of Vercellotti and colleagues (1989), whose data indicated that benzothiazole levels increased steadily with storage, those of methional increased and then decreased, and methylsulfone levels decreased and then leveled off over time. This study involved extraction but no distillation. Finally, a milder extraction method [extraction at 50°C for 24 h in a Soxhlet thimble; Spanier, 1992] followed by gas chromatographic/mass spectrophotometric (GC/MS) detection indicated that thiazoles were not detectable in precooked ground beef. Thus, deciphering the true volatile profile of the food being analyzed is a complex problem compounded by the choice of extraction method. The experiments described here were designed to analyze the effect of temperature on the development and/or degradation of the volatile flavor precursors and products of cooked and cooked/stored ground beef.

MATERIALS AND METHODS

Preparation and handling of ground beef patties

Beef was purchased from a local supermarket that received its supplies from Monfort Beef of Colorado. Beef was USDA-choice, top round (*semimembranosus* muscle) obtained from Angus-cross steers. The beef was trimmed of all visible tallow and ground by two passes through a General Slicer (Model MC-100): once through a disk with 1·0 cm holes and once through a disk with 0·75 cm holes. The fat content of the final ground round patties averaged 4·25%, based on the perchloric—acetic acid method of Koniecko (1985).

Patties (85 g) were cooked on an open-top grill (Farberware, 180°C) for 7·5 min on each side. The final end-point internal temperature was 66·2°C and the average final cooking weight was 59·8 g (approximately 30% weight loss). The cooked patties were cooled and reground (0·75 cm grinding disk) to obtain more homogeneously distributed samples. The cooked/reground beef patties (52 g) were tightly packed into glass Petri dishes, covered, and stored in a refrigerator at 4°C for 0, 1, 2, and 4 days.

Volatile analysis via dynamic purge-and-trap gas chromatography

Ground beef patties were analyzed using dynamic purge-and-trap gas chromatography and volatile detection by a Tracor Model 100AT detector (Fig. 1; Tracor, Austin, Texas). The detector, described by Brown *et al.* (1986) for egg volatiles combines flame photometric detection (FPD; used in sulfur mode) with flame ionization detection (FID). A Tekmar 25 ml needle impinger-assembly was substituted for the generally accepted Tekmar semiautomatic purge-and-trap concentrator (Model LSC-3; Vercellotti *et al.*, 1992). Freshly cooked/reground beef patties (1·0 g) and those stored at 4°C for 1, 2, and 4 days were sparged for 30 min at either 50, 75, or 100°C within the needle impinger assembly. Nitrogen was used as a carrier gas at a flow rate of 20 ml/min. The beef volatiles, after passing through a transfer line and a six port, 1/16 in Valco™ valve (Valco Instruments, Houston, TX), were trapped/concentrated directly onto a packed Tenax GC–8% polymetaphenyl ether column (10 ft × 1/8 in) held at ambient temperature (20–21°C). Samples were concentrated on the column for 30 min Volatiles were eluted from the column by purging with nitrogen for a total of 60 min (nitrogen flow: 20 ml/min; column heated from 25 to 250°C at 3°C/min). The sample tube was removed from the injection port after completion of the volatile stripping/sparging process and replaced with a tube containing a few milliliters of water to effect steam distillation. The six-port valve permitted switching to a valve purge position that allowed the valve and transfer lines to be cleaned between runs by steam distillation from an impinger tube containing water alone.

Identification of volatile components was accomplished with the identical system [packed Tenax GC–8% polymetaphenyl ether column (10 ft × 1/8 in)] coupled to a Finnigan-MAT 4500 gas chromatograph/mass spectrometer/data system (Sunnyvale. California) as described by St. Angelo *et al.* (1987).

RESULTS & DISCUSSION

Rudimentary knowledge of the chemical and physical composition of the food being examined should be available prior to the design of experiments for study of the food's flavor-volatile composition. The experiments described here used ground beef, a food with a complex structural matrix and a relatively high water content (Lawrie, 1991; Spanier *et al.*, 1992; Spanier & Miller, 1993). Volatile components of ground beef can be analyzed in several ways, including steam distillation, solvent extraction, headspace analysis, and/or supercritical fluid extraction (Weurman, 1969; Jennings & Filsoof, 1977; Cole, 1980; Reineccius & Anandaraman, 1984). Use of steam distillation was ruled out in these temperature experiments since the extraction could be performed at only one temperature (100°C). Furthermore, volatiles not originally present in the sample might form as a result of heating. According to Reineccius and Anandaraman (1984) and Risch and Reineccius (1989), steam distillation–based recoveries of model compounds from aqueous solution at 10 ppm

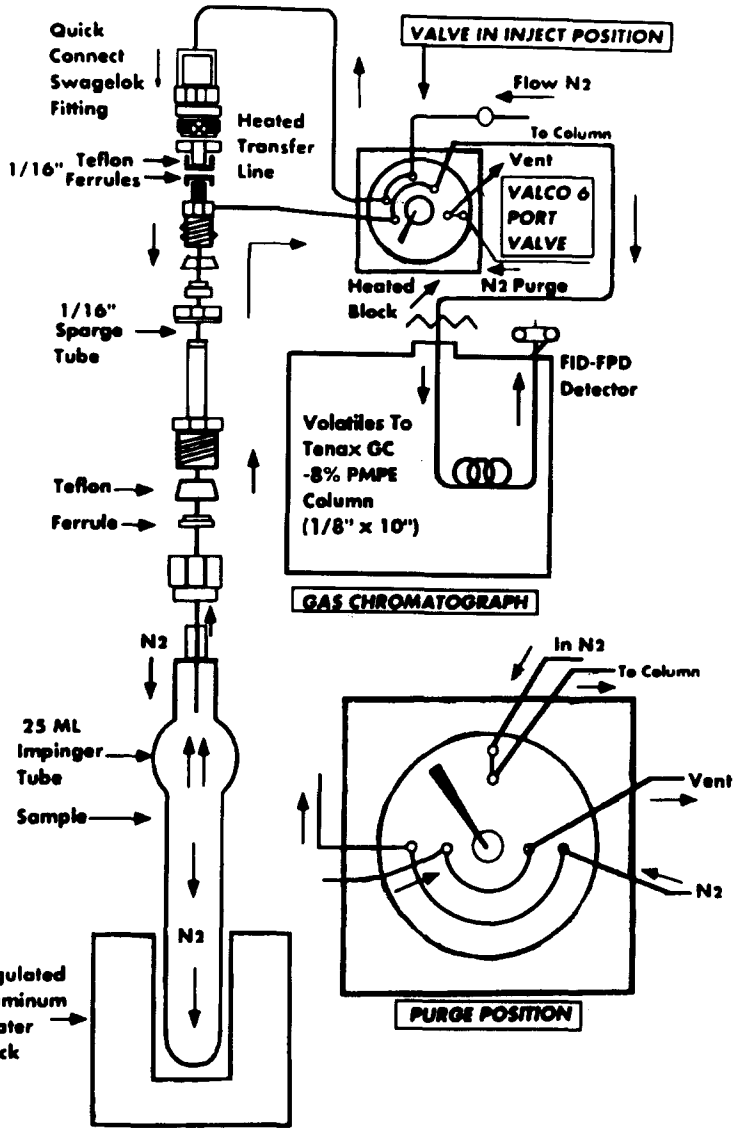

Fig. 1. Diagram of the dynamic purge-and-trap gas chromatographic setup.

ranged from 23–100%, suggesting that steam distillation is a rather indiscriminate technique. Organic extraction methods were eliminated from this study because of the variability in the relative proportions of solute that would partition between the aqueous and organic phases, and because more volatile components could be lost during the evaporation/concentration step (Cole, 1980). Use of supercritical fluid chromatography (SFC) was ruled out, even though the extraction (solvent) and temperature conditions were milder, because the products extracted by SFC show great variability, depending upon the supercritical fluid used. Therefore, headspace analysis was the method of choice for analysis of meat flavor volatiles.

Headspace analysis determines the composition of the volatiles by passing an inert gas through the sample and collecting the compounds in equilibrium with the air above the food. These constituents can be separated from the water that co-elutes with them (particularly muscle foods) by extracting any trapped water with an organic solvent. Extraction methods using organic solvents introduces problems inherent to most extraction protocols, even though the amount of solvent used is significantly less than that used in other extraction techniques. Alternatively, volatiles from meat can be trapped, typically at room temperature, on an adsorbent matrix such as Tenax, charcoal, Porapak or Chromosorb materials (Zlatkis *et al.*, 1973). Unfortunately, the compounds must be desorbed either by heating at relatively high temperatures or eluting with solvents. The former method results in selective desorption from the trap, depending upon the matrix composing the trap, and will require high temperatures that might alter the composition of the components trapped. Thus, solvent extraction is not appropriate for the reasons cited above.

An alternative mode of headspace analysis involves the direct purging of the volatiles onto the head of a column via cryofocusing of the volatile materials onto the column without first passing through a solid trap. Unfortunately, the high water content of muscle foods (Lawrie, 1991) makes this method infeasible with capillary columns known for their inability to cope with the presence of water. Even though packed columns have

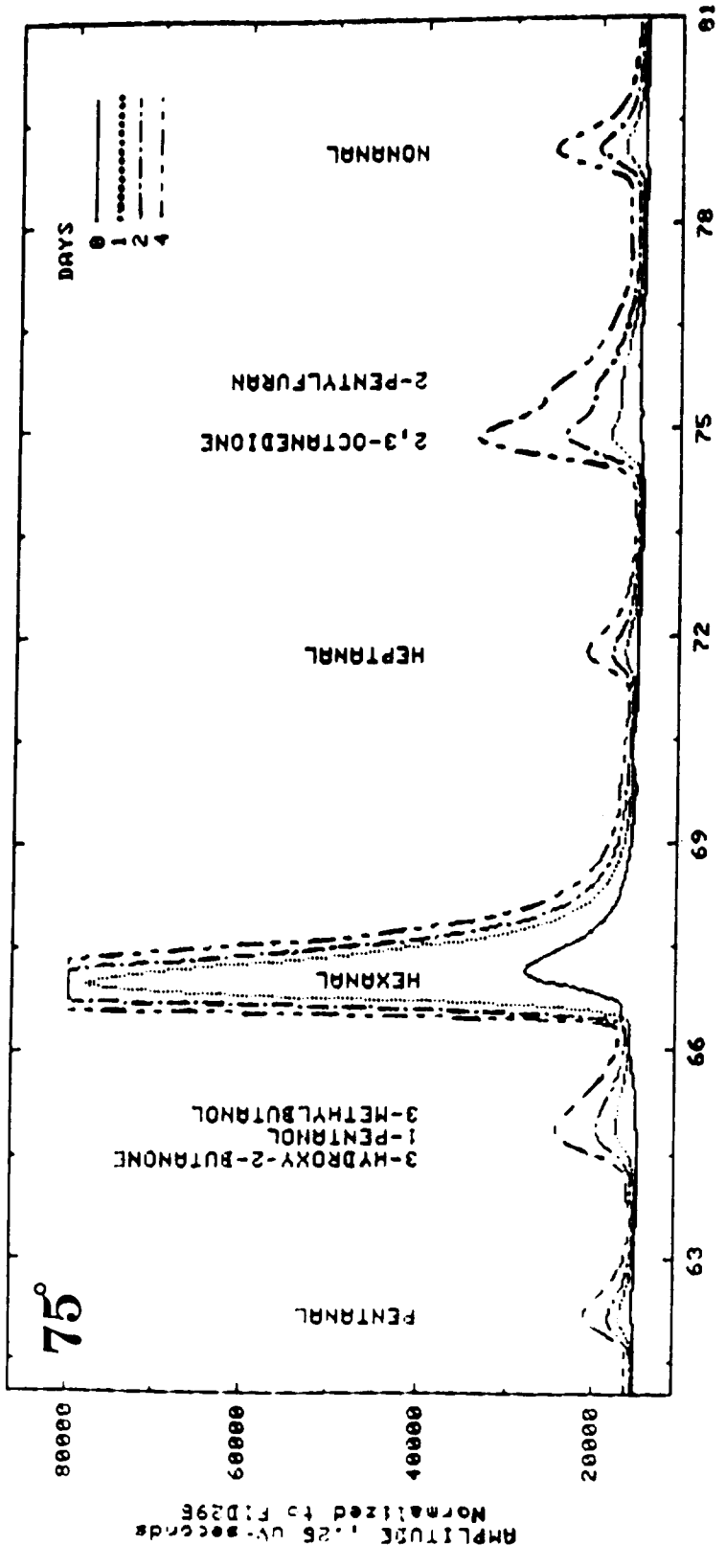

Fig. 2. Packed column (Tenax GC–8% polymetaphenyl ether; 10 ft × 1/8 in) gas chromatographic (GC) profile of volatiles from either freshly cooked and unstored ground beef (top round 0 days), or precooked and stored (4°C) for 1, 2, and 4 days. Detection by flame ionization detection. Volatiles were identified by GC/MS and are noted directly on the figure.

Table 1. Effect of sparge temperature on the determination of the alcohols, and carbonyl compounds of cooked/stored beef

Compound	Sparging temperature (°C)	Peak Area ($\times 10^4$) after cold storage (4°C) [mean/sem[a]]			
		0 day	1 day	2 days	4 days
Methanol	50	N.D.	14·1/0·6	N.D	56·3/7·4
	75	66·0/0·78	17·8/0·7	11·6/1·8	N.D.
	100	24·5/2·1	35·9/5·7	23·5/2·5	20·1/2·0
Ethanol	50	4·6/1·7	N.D.	N.D.	N.D.
	75	11·0/2·7	N.D.	9·8/1·7	N.D.
	100	14·0/1·2	12·3/0·8	N.D.	N.D.
	50	9·8/2·7	22·2/4·5	19·4/2·6	24·5/5·9
Pentane (pentanal on front shoulder)	75	33·1/2·7	27·9/3·8	34·4/1·8	45·4/2·2
	100	9·8/2·3	21·5/1·2	31·5/1·7	44·4/2·1
2-Methylpropanal	50	12·9/1·3	12·1/1·7	5·8/1·9	7·8/1·2
	75	16·4/0·3	13·7/0·2	7·3/0·2	5·0/0·2
	100	13·5/0·4	8·3/1·4	9·5/0·3	8·8/0·4
2,3-Butanedione	50	0·1/0·0	N.D.	N.D.	N.D.
	75	N.D.	N.D.	5·1/0·3	7·8/0·1
	100	11·3/0·2	10·0/0·5	11·5/0·1	12·2/0·4
3-Methylbutanal	50	1·4/0·1	2·9/0·2	2·0/0·1	4·5/0·2
	75	3·8/0·4	4·0/0·4	1·5/0·5	2·3/0·4
	100	9·8/2·9	22·8/1·0	21·6/1·4	33·3/4·1
Pentanal	50	2·0/0·1	7·4/0·1	8·2/0·2	10·2/0·3
	75	4·9/0·7	9·4/0·2	12·1/0·1	19·1/0·8
	100	22·0/3·7	11·3/0·5	16·3/0·7	24·6/0·8
3-Hydroxy-2-butanone	50	0·2/0·1	6·6/0·1	8·0/0·2	12·7/0·6
(1-pentanol and/or 3-methyl	75	6·4/0·2	13·7/0·6	22·5/0·9	37·8/1·5
butanol on back shoulder)	100	5·1/0·6	16·7/1·1	26·4/0·4	38·8/0·6
Hexanal	50	22·9/1·2	128·9/3·3	179·9/3·3	246·5/5·7
	75	43·8/1·6	163·7/2·4	248·5/3·9	371·6/7·8
	100	33·5/2·7	142·9/5·6	212·8/1·6	320·4/4·5
Heptanal	50	2·3/40·4	8·4/0·3	9·3/0·8	13·5/1·3
	75	5·6/0·4	10·5/0·2	14·9/0·2	22·9/1·0
	100	3·9/0·2	8·3/0·3	15·8/0·6	26·1/0·9
2,3-Octanedione	50	2·3/0·3	5·0/0·2	17·2/1·3	13·7/0·8
	75	2·7/0·4	10·8/0·8	27·9/0·5	42·6/0·8
	100	3·3/0·3	14·7/0·2	31·4/1·2	47·8/1·8
2-Pentylfuran	50	0·8/0·2	10·0/0·6	7·6/1·5	18·5/1·5
	75	6·7/0·2	14·1/0·3	21·0/0·2	36·2/1·0
	100	7·6/0·7	12·8/0·4	22·7/1·0	36·0/0·4
Nonanal	50	2·7/0·6	5·6/0·2	N.D.	10·6/0·5
	75	3·7/0·3	9·8/0·2	17·5/0·5	32·6/0·6
	100	5·2/0·2	14·0/0·5	26·4/0·5	45·1/0·3

Ground beef patties were cooked on an open-top Farberware grill (180°C) for 7·5 min on each side. The final end-point internal temperatures was 66·2°C with an average final cooking weight of 59·8 g. The cooked beef patties were tightly packed into labeled glass Petri dishes, covered, and stored in a refrigerator at 4°C for 0, 1, 2, and 4 days.

[a] mean/sem = means and standard errors of the mean were determined on multiple samples ranging from 3 to 6 determinations. N.D. = Not determined. Data is not included for one or more reasons including (1) sample value too low to measure, (2) less than 3 samples available for analysis, (3) sample lost or otherwise mishandled, e.g. smudged or unidentifiable label.

lower resolution than capillary or megabore columns, they are able to function in the presence of significant amounts of water. Therefore, muscle food volatiles were trapped/concentrated (at ambient temperature: 20–21°C) directly onto a packed Tenax GC–8% polymetaphenyl ether column which has been shown to be useful for analysis of carbonyl compounds from beef (Dupuy *et al.*, 1987).

Figure 2 shows a typical volatile profile for stored cooked ground beef patties (St. Angelo *et al.*, 1987, 1988; Drumm & Spanier, 1991; Spanier, 1992; Spanier *et al.* 1992; Spanier & Miller, 1993). These data clearly show that off-flavor volatiles progressively develop in cooked meat during storage. The samples in this figure were sparged with nitrogen and maintained at a temperature of 75C°, approximating the final cooking temperature of many meat products. Most reports in the literature examined volatiles isolated from meats at much higher (greater than 100°C) purging temperatures (Bailey & Einig, 1989; Larick & Turner, 1992). These high temperatures produce an environment in which new volatiles might be produced pyrolytically. These experiments were designed to examine the effect of purge temperature on analysis of carbonyl and sulfur-containing compounds in beef.

The carbonyl components found in precooked/stored meat sparged at three different temperatures (50°, 75°, 100°C) were examined. Volatile profiles were quantitatively similar to those seen in Fig. 2, but the intensities and areas (Table 1) of various peaks differed signifi-

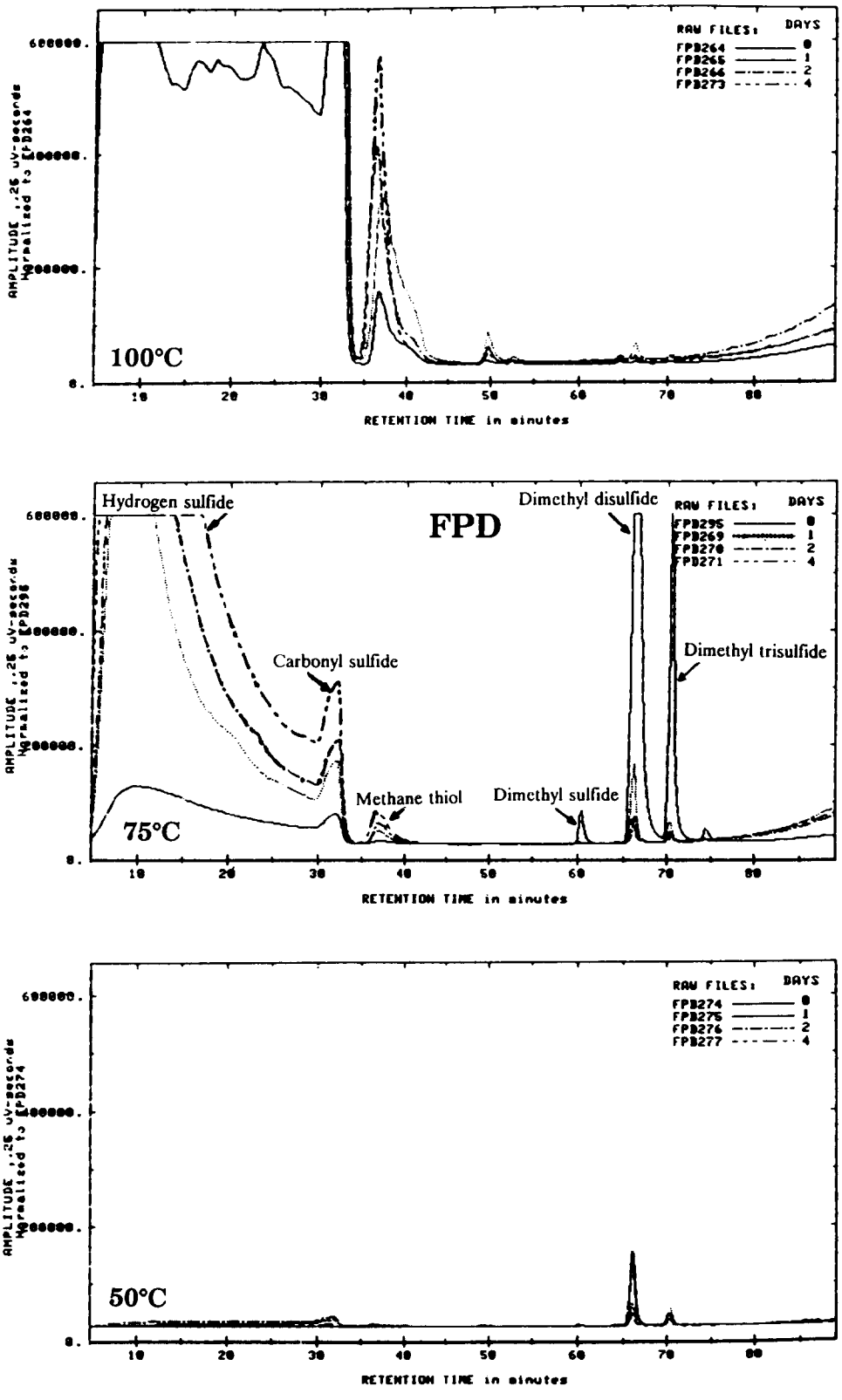

Fig. 3. Packed column gas chromatographic profile with flame photometric detection (FPD) of cooked ground beef stored for 0, 1, 2, and 4 days at 4°C (as described in Fig. 2). Samples were sparged and heated with nitrogen at 50°C 74°C, and 100°C.

cantly as described below. The temperature-dependent quantitation of low carbon number materials, i.e. from methanol to 2,3-butanedione, was significantly variable and difficult to reproduce. On several occasions, the expected peaks did not appear or were superimposed on peaks of other materials. A typical increase with storage was seen for volatiles ranging in mass from 3-methyl butanal to nonanal. These latter carbonyl compounds were at the lowest levels and were most difficult to quantitate at sparging temperatures of 50°C. Reproducibility, peak heights, and peak patterns appeared to reach their optimum level at sparging temperatures of 75°C; their patterns at 100°C are similar to that at 75°C (Table 1). Previous studies have shown similar results at different cooking temperatures (Drumm & Spanier, 1991).

Table 2. Effect of sparge temperature on the determination of the sulfur compounds of cooked/stored beef

Compound	Sparging temperature (°C)	Peak Area ($\times 10^6$) after cold storage (4°C) [mean/sem]			
		0 day	1 day	2 days	4 days
Hydrogen sulfide	50	U.D.	1·01/0·1	0·12/0·0	0·13/0·0
	75	45·80/2·1	208·14/4·0	280·07/2·0	332·11/4·5
	100	406·00/2·1	412·00/2·9	433·00/54·2	458·00/5·6
Carbonyl sulfide	50	0·02/0·0	0·73/0·1	0·55/0·0	0·25/0·0
	75	3·15/0·5	8·53/0·3	12·88/0·3	23·59/0·3
	100	72·46/1·3	92·84/1·3	108·00/1·1	111·00/1·1
Methane thiol	50	0·02/0·00	0·10/0·00	0·19/0·05	0·21/0·01
	75	0·425/0·02	0·67/0·05	2·48/0·07	4·31/0·04
	100	37·96/1·62	32·76/1·09	31·36/1·23	34·98/1·22
Dimethyl sulfide	50	0·041/0·005	0·035/0·007	0·029/0·005	0·002/0·001
	75	0·076/0·004	0·061/0·001	0·038/0·002	0·028/0·001
	100	1·963/0·04	1·940/0·01	0·691/0·03	0·253/0·02
Dimethyl disulfide	50	2·77/0·06	2·88/0·03	0·83/0·05	0·38/0·02
	75	28·39/1·00	2·88/0·03	1·21/0·03	0·93/0·03
	100	0·34/0·02	0·73/0·04	0·43/0·04	0·15/0·05
Dimethyl trisulfide	50	0·485/0·01	0·67/0·03	0·24/0·02	0·15/0·02
	75	13·28/1·33	0·76/0·04	0·39/0·02	0·24/0·02
	100	0·40/0·01	0·21/0·01	0·17/0·01	0·13/0·01

U.D. = undetectable peak value.

Unlike the carbonyl compounds, the analyses of sulfur-containing components show a very high degree of temperature dependence (Table 2). Precise assessment of the content and composition of sulfur compounds is essential in the study of meat flavor since the sulfur-containing compounds, long known to be involved in the generation of Maillard reaction products (MRPs; Danehy, 1986), play a vital role in the production of flavor in meat (Dwivedi, 1975). A significant proportion of the organic compounds which are known to produce meat-like flavor contain sulfur, and a significant number of these compounds are aldehydes. The nature of the aldehydes produced during the Maillard reaction is dependent upon the characteristics of the amino acids and sugars involved (Danehy, 1986). Reasonably, the production of MRPs can be controlled through control of reactants and reaction conditions such as temperature. There is an ever-growing list of MRPs found in synthetic mixtures and in meat, but we were unable to find published quantitative correlations between the levels of MRPs and the sensory responses to these compounds in meat. Although much is known about the formation of MRPs *in vitro* little is know of their formation and influence on the overall sensory perception and flavor of meat *in vivo*.

The relative impact of sulfur-containing compounds to beef flavor cannot be understated (Chang & Peterson, 1977). For that reason, it is important to have methods available that will yield accurate assessments of the true composition of the flavor components of foods. Analyses of the sulfur compounds found in cooked ground beef have indicated that several of the heterocyclic compounds, such as 4-methylthiazole, 2-acetylthiazole, benzothiazole, and 2-furylmethanethiol, are fully stable (Drumm & Spanier, 1991). These latter compounds were identified in ground beef patties that had undergone a four-hour steam distillation–extraction protocol. On the other hand, use of milder methods (methylene chloride extraction for 24 h in a Soxhlet thimble at 50°C followed by concentration in a Kadurna–Dannish apparatus), having efficiency of recovery greater than 95%, indicated that precooked ground beef did not contain any thiazoles (Spanier, 1992).

Other sulfur-containing compounds in meat are thought to be formed by Strecker degradation of cysteine and methionine and from hydrogen sulfide. Hydrogen sulfide is produced via several mechanisms, including free-radical reactions (Schutte, 1974). Hydrogen sulfide, which has been shown to be a product of the degradation of dimethyl trisulfide (Schutte, 1974), can also react with several components of meat to give ethanedithiol, (methylthio)ethanethiol, and dimethyltrithiolane. The three compounds have been shown to increase in meat during storage (Drumm & Spanier, 1991). Table 2 demonstrates the increase in the tissue level of hydrogen sulfide during prolonged storage of precooked meat, particularly at sparge temperatures above 75°C. This correlates well with the decline in dimethyl trisulfide. The generation of free radicals during storage, through lipid oxidation reactions and the reaction of these radicals with sulfur amino acids and sulfur-containing compounds (Gardner, 1988) would contribute to an increase in hydrogen sulfide content.

The content of dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide decreases with increased storage, regardless of the sparging temperature (Table 2; Fig. 3). Similar results were observed for dimethyl trisulfide using distillation-extraction techniques (Drumm & Spanier, 1991). Oxidation of methanethiol results in the formation of dimethyl disulfide with further reactions to form dimethyl trisulfide and dimethyl sulfide. Dimethyl trisulfide may undergo further degra-

dation to hydrogen sulfide, carbonyl sulfide, and methanethiol (Schutte, 1974; Baines & Mlotkiewicz, 1984).

While the general trend of the data at most sparging temperatures shows a decrease in some sulfur-containing compounds (dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide) and an increase in other sulfur-containing compounds (hydrogen sulfide, carbonyl sulfide, and methane thiol), it is important to note that temperatures that are either too low (50°C) or too high (100°C) can lead to misinterpretation of the data. This effect arises from inefficient extraction of the volatile components from the products (low temperatures) or the conversion of one form of a sulfur-containing compound to another (high temperatures, Fig. 3). The data suggest that a sparging temperature of 75°C, which is the final temperature of many beef products cooked medium to well done (Cross *et al.*, 1978), is the optimum temperature at which to analyze meat for levels of sulfur-containing (Table 2) and carbonyl-containing (Table 1) products. Independent studies of enzyme activity (hydrolases) and volatile production (Spanier *et al.*, 1990) also point to 75°C as an optimum temperature for investigation of flavor volatiles.

SUMMARY AND CONCLUSIONS

The study of volatile flavor compounds, specifically the carbonyl and sulfur-containing compounds, is a key to understanding the changes that occur in the flavor quality of meat products during cooking and storage. Due to the sensitivity of these compounds to heat, the sparge temperature must be carefully controlled to maximize recovery of volatile flavor compounds and to minimize artifact formation when obtaining volatile flavor profiles characteristic of the meat product.

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