Headspace Oxygen in Sample Vials Affects Volatiles Production of Meat during the Automated Purge-and-Trap/GC Analyses

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Headspace oxygen in sample vial for the purge-and-trap dynamic headspace/gas chromatography method oxidizes meat if held hours before purging, influences volatile profiles, and misrepresents the true composition of volatiles. Helium flush and helium flush plus oxygen absorber were used to eliminate residual oxygen and minimize oxidative changes in meat during sample holding time. Both helium flush and helium flush plus oxygen absorber treatments were effective in preventing an increase in 2-thiobarbituric acid reactive substances (TBARSs) and volatiles production in raw meat for up to 640 min of sample holding. With helium flush plus oxygen absorber, only 1-octen-3-ol increased during the 1280-min sample holding time. However, the hexanal peak in raw meat was interfered by 2,6-dimethyl heptane when oxygen absorber was added. Therefore, use of oxygen absorber was not appropriate for raw meat. Helium flush reduced oxidative changes in cooked meat during sample holding time but was not able to stop oxidative changes in meat after 160 min sample holding. A combination of helium flush and oxygen absorber was effective in preventing volatiles production in cooked meat for over 20 h of sample holding at 4 °C.

Keywords: *Sample holding time; lipid oxidation; volatiles; helium flush; oxygen absorber*

INTRODUCTION

It has been shown that purge temperature and sample holding time before purge influenced the profile of volatiles in raw and cooked meat (Ahn et al., 1999). Many of the changes in volatiles were related to oxidation of lipids, and the changes were more pronounced in cooked meat than in raw meat. Ahn et al. (1992) showed that oxygen contact with meat was the most important factor in the development of lipid oxidation. The purge-and-trap dynamic headspace/gas chromatography (GC) method uses sample vials that can have up to 10 mL of oxygen in the headspace. Oxygen, therefore, in a sample vial could oxidize the sample if held hours before purging and would produce more volatiles than the one purged immediately after sampling. Waiting time in an autosampler tray, therefore, could considerably influence volatile profiles and misrepresent the true composition of volatiles. Therefore, residual oxygen in sample vials should be eliminated to prevent oxidative changes during sample holding time.

Flushing with inert gas (e.g., helium or nitrogen) or adding an oxygen absorber packet in the vial can reduce residual oxygen in the sample vial. Mistry and Min (1992) used a glucose-oxidase enzyme system to remove oxygen dissolved in salad dressing. Min et al. (1989) reported that a hydrogen−palladium system could remove residual oxygen in the headspace of gas-packaged pouch. However, mixing meat samples with a glucose-oxidase enzyme system could generate products that can influence volatile profiles. The hydrogen−palladium system is not practical to be used in this study because it is designed to remove oxygen in the headspace of the packaging pouch by impregnating palladium in the packaging film (Min et al., 1989). Oxygen absorber packets are readily available and are currently used to remove oxygen in food products such as coffee and other dry foods during storage.

The objective of this work was to determine the effect of helium flush and helium flush plus oxygen absorber on the production of volatiles and lipid oxidation in raw and cooked meat during sample holding time. A refrigerated sample tray designed for autosampling of the purge-and-trap dynamic headspace/GC method was used to hold the samples. The ultimate goal of this study was to develop optimal conditions for automated volatile analysis in raw and cooked meat using a purge-and-trap dynamic headspace/GC method.

MATERIALS AND METHODS

Sample Preparation. Boneless and skinless breast meats (5 kg) were separated from eight turkeys raised in the Poultry Research Farm at Iowa State University. Breast meats from two turkeys were pooled and treated as a replication. Pooled meats were ground twice through a 3-mm plate, and a total of 48 patties (12 from each pooled meat, 100 g/patty) were prepared. Twenty-four of the patties were used for raw meat study, and the other 24 patties were cooked in an electric oven (300 °C) to an internal temperature of 78 °C. Raw meat patties were vacuum packaged and stored at 4 °C and opened before use. The cooked patties were individually vacuum packaged in oxygen-impermeable nylon/polyethylene bags (O_2 permebility, 9.3 mL O_2/m^2/24 h at 0 °C, Koch, Kansas City, MO) 30 min after cooking and stored at 4 °C.

To determine the effect of residual oxygen on the lipid oxidation and volatiles production in meat samples, three sets of raw and cooked meat samples were prepared: one set with no treatment (control), one set flushed with helium (99.999%) for 5 s at 40 psi, and one set with added oxygen absorbers (Ageless type Z-100, Mitsubishi Gas Chemical America, Inc., New York, NY) plus helium flush. The oxygen absorber was added in a sample vial before helium flush. Vials were capped tightly with a Teflon-lined, open-mouth cap and then placed in a refrigerated (4 °C) sample tray 40 min before the

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Table 1. Effect of Helium Flush and Helium Flush plus Oxygen Absorber (Ageless) on the TBARSs of Raw and Cooked Turkey Breast Meat during Sample Holding Time in an Autosampler at 4 °C[a]

	sample holding time (min)							
treatment	baseline	0	80	160	320	640	1280	SEM
raw turkey breast meat								
control	0.34^{c}	0.35^{cx}	0.36^{cx}	0.53^{bx}	0.44^{bcx}	0.56^{bx}	0.84^{ax}	0.034
He flush	0.34	0.31^{y}	0.30^{y}	0.33^{y}	0.33^{y}	0.31^{y}	0.32^{y}	0.010
He flush + oxygen absorber	0.34^{a}	0.27^{cz}	0.23^{dz}	0.33^{ay}	0.29^{bcy}	0.28^{cy}	0.31^{aby}	0.010
SEM		0.010	0.010	0.010	0.040	0.015	0.032	
cooked turkey breast meat								
control	0.67^{g}	1.35^{fx}	2.66^{ex}	3.24^{dx}	3.64^{cx}	3.89^{bx}	4.88^{ax}	0.063
He flush	0.67^{d}	1.11^{dy}	1.81^{cy}	2.00^{cy}	2.71^{by}	3.01^{aby}	3.47^{ay}	0.010
He flush + oxygen absorber	0.67^{c}	0.94^{bcz}	1.33^{aby}	1.48^{aby}	1.24^{abz}	1.38^{az}	1.65^{az}	0.010
SEM		0.042	0.085	0.163	0.133	0.128	0.200	

[a] mg MDA/kg meat. Samples (5 g) were put in 50-mL test tubes, capped tightly, and held at 4 °C for designated times before analysis. $n = 4$. $^{a-g}$Different letters within a row are significantly different ($P < 0.05$). $^{x-z}$Different letters within a column are significantly different ($P < 0.05$). SEM, standard error of the mean.

designated sample holding time. The samples were analyzed at 0, 80, 160, 320, 640, and 1280 min after being placed in the refrigerated sample tray. Samples were heated to 40 °C before purging with helium gas. Lipid oxidation of the meat samples was also determined at 0, 80, 160, 320, 640, and 1280 min after a stay in a refrigerated sample tray. Analyses were replicated four times.

Volatiles Analysis. A purge-and-trap apparatus connected to a gas chromatograph (GC) was used to analyze the volatiles potentially responsible for the off-odor in meat. Precept II and Purge-and-Trap Concentrator 3000 (Tekmar-Dorham, Cincinnati, OH) were used to purge and trap volatiles from the samples. A GC (Model 6890, Hewlett Packard Co., Wilmington, DE) equipped with a mass selective detector (MSD, HP 5973, Hewlett-Packard Co.) was used to characterize and quantify the volatile compounds influenced by headspace oxygen during sample holding periods. A 5-g sample was used for raw meat, and a 3-g sample was used for cooked meat analyses. The meat sample was placed in a sample vial (40 mL) and purged with helium gas (40 mL/min) for 15 min. Volatiles were trapped at 30 °C using a Tenax/Silica gel/Charcoal column (Tekmar-Dorham) and desorbed for 1 min at 220 °C. A split inlet (split ratio, 49:1) was used to inject volatiles into a GC column (HP-5MS capillary column, 0.25 mm i.d., 30 m, and 0.25 μm film thickness, Hewlett-Packard Co.) and ramped oven temperature conditions (30 °C for 2 min, increased to 40 °C at 2 °C/min, increased to 50 °C at 5 °C/min, increased to 100 °C at 10 °C/min, increased to 140 °C at 20 °C/min, increased to 200 °C at 30 °C/min, and held for 4.5 min) were used. Inlet temperature was 180 °C. Helium was used as a carrier gas, and column flow was 1.1 mL/min. The ionization potential of MS was 70 eV; scan range was m/z 45−450. Identification of volatiles was achieved by comparing mass spectral data with those of the Wiley library (Hewlett-Packard Co.). The area of each peak was integrated by using ChemStation software (Hewlett-Packard Co.), and the total ion counts $\times 10^3$ was reported as an indicator of volatiles generated from the meat samples.

Lipid Peroxidation. Lipid peroxidation of raw and cooked turkey meat was determined by the modified (Ahn et al., 1998) method of Buege and Aust (1978). A 5-g meat sample was placed in a 50-mL test tube and homogenized with 15 mL of deionized distilled water by using a homogenizer (Type PT 10/35, Brinkman Instruments Inc., Westbury, NY) for 15 s at speed 7−8. Meat homogenate (1 mL) was transferred to a disposable test tube (13 × 100 mm), and butylated hydroxyanisole (50 μL, 7.2%) and thiobarbituric acid/trichloroacetic acid (TBA/TCA) solution (2 mL) were added. The mixture was vortexed and then incubated in a boiling water bath for 15 min to develop color. After color development, the samples were cooled in cold water for 10 min and then centrifuged for 15 min at 2000g. The absorbance of the resulting supernatant solution was determined at 531 nm against a blank containing 1 mL of DDW and 2 mL of TBA/TCA solution. The 2-thiobarbituric acid reactive substance (TBARS) numbers were expressed as milligrams of malondialdehyde (MDA) per kilogram of meat. The TBARS values were determined after subjecting them to exactly the same time, oxygen absorber, and temperature conditions as in the samples for volatile analyses.

Statistical Analysis. The experiment was designed primarily to determine the effect of sample holding time before volatiles analysis on the lipid peroxidation and volatiles production in raw and cooked meat. The TBARS and selected volatile components of raw and cooked meat at different residual oxygen conditions were analyzed independently by SAS software (SAS Institute, 1989). Analyses of variance were conducted to test the effects of oxygen and sample holding time, and the Student-Newman-Keuls multiple range test was used to compare differences among mean values. Mean values and standard errors of the mean (SEM) were reported when necessary.

RESULTS AND DISCUSSION

Lipid Oxidation of Meat. In raw turkey breast meat with no treatment to remove oxygen in sample vial (control), TBARS of meat samples held longer than 160 min in an autosampler at 4 °C were significantly higher than those of 0 and 80 min (Table 1). TBARS changes in raw meat samples held between 160 and 640 min were not significantly different, but the samples held for 1280 min had higher TBARS than others. With helium flush, however, TBARS of raw turkey breast meat did not change during the 1280-min sample holding time. With helium flush plus oxygen absorber, significant differences in TBARS of raw meat samples held different lengths of time were found, but the differences were small and inconsistent. The TBARS of control raw turkey breast meat was higher than those of the helium flush and helium flush plus oxygen absorber at all sample holding times (Table 1).

In cooked turkey breast meat, TBARS increased with the increase of sample holding time in all oxygen removal treatments. Control and helium flush treatments had 3-fold and helium flush plus oxygen absorber treatment had <2-fold increase in TBARS during 1280 min holding time. However, the changes were greater in magnitude in controls than in helium flush or helium flush plus oxygen absorber treatments. After 320 min or longer of sample holding time, the TBARS of cooked meat treated with helium flush plus oxygen absorber were less than half of the helium flush and were not changed after 80 min of sample holding. As in raw turkey breast meat, helium flush plus oxygen absorber had the lowest, control had the highest, and helium flush samples had intermediate TBARS at all sample holding times (Table 1).

Table 2. Production of Volatiles in Raw Turkey Breast Meat with No Helium Flush or Oxygen Absorber (Control) during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	30[b]	31[b]	71[b]	52[b]	76[b]	200[a]	12.1
hexane	20[c]	23[bc]	27[bc]	25[bc]	37[ab]	44[a]	3.9
heptane	tr[b]	tr[b]	tr[b]	tr[b]	tr[b]	45[a]	3.6
propanal	tr[b]	tr[b]	34[bc]	42[bc]	78[b]	258[a]	15.6
hexanal	423[c]	501[c]	653[bc]	666[bc]	1242[b]	2934[a]	164.8
1-pentanol	84[c]	92[c]	131[c]	151[c]	314[b]	610[a]	34.4
nonanal	144[b]	106[b]	113[b]	123[b]	166[ab]	236[a]	23.8
1-octen-3-ol	189[c]	159[c]	206[c]	242[c]	435[b]	854[a]	47.8
total volatiles	935[c]	955[c]	1254[c]	1330[c]	2367[b]	5181[a]	272.3

[a] Area (ion count × 1000). Samples (5 g) were purged at 32 °C. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a–c]Different letters within a row of the same storage time are different ($P < 0.05$). SEM, standard error of the mean. tr: trace amount.

Table 3. Production of Volatiles in Raw Turkey Breast Meat with Helium Flush during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	64[c]	123[abc]	98[bc]	94[bc]	165[ab]	193[a]	20.8
hexane	39[c]	50[bc]	69[bc]	50[bc]	98[b]	164[a]	13.4
heptane	tr[b]	tr[b]	14[b]	12[b]	33a	37[a]	3.6
propanal	tr[b]	tr[b]	tr[b]	tr[b]	tr[b]	66[a]	7.5
hexanal	174[b]	202[b]	180[b]	154[b]	291[b]	621[a]	55.9
1-pentanol	82[c]	81[c]	76[c]	104[c]	238[b]	315[a]	24.7
nonanal	252	211	156	147	193	154	24.2
1-octen-3-ol	402[b]	490[ab]	441[ab]	443[ab]	650[a]	640[a]	52.3
total volatiles	1032[c]	1178[c]	1042[c]	1009[c]	1668[b]	2189[a]	128.8

[a] Area (ion count × 1000). Samples (5 g) were flushed immediately after sampling. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a–c]Different letters within a row of the same storage time are different ($P < 0.05$). SEM, standard error of the mean. tr: trace amount.

Table 4. Production of Volatiles in Raw Turkey Breast Meat with Helium Flush plus Oxygen Absorber during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	29	33	37	32	43	46	7.1
hexane	19	19	21	20	22	20	0.9
2,6-dimethyl-heptane + hexanal	1084	1871	1871	1415	1162	1048	220.2
1-pentanol	49	71	86	94	94	85	10.6
nonanal	95	109	176	107	158	95	26.0
1-octen-3-ol	158[b]	231[ab]	263[ab]	302[a]	322[a]	312[a]	32.3
total volatiles	1453	2355	2473	1992	1819	1625	237.8

[a] Area (ion count × 1000). Samples (5 g) were added with oxygen absorber and flushed immediately after sampling. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a,b]Different letters within a row are significantly different ($P < 0.05$). SEM, standard error of the mean.

In raw meat, the oxidative changes during holding period were relatively small, but oxygen removal from sample vial had significant impact on the TBARS of raw meat samples after 160 min of holding. Both helium flush and helium flush plus oxygen absorber treatments were effective in preventing an increase in TBARS in raw meat during the 1280 min holding time. In cooked meat, however, the use of helium flush alone was not sufficient to prevent TBARS from increasing during sample holding time. Ahn et al. (1993) reported that lipid oxidation in cooked meat developed rapidly during the first 2 h after exposure to air. This study indicated that cooked meat developed lipid oxidation rapidly even after helium flush and helium flush plus oxygen absorber treatments. The amount of residual oxygen in the sample vial after helium flush or helium flush plus oxygen absorber treatment is <2% of the control (data not shown), but it still could trigger oxidative changes in cooked meat. Samples with control and helium flush treatments showed a continuous increase in TBARS over the 1280-min sample holding time. However, no further oxidative changes were observed in cooked meat samples with helium flush plus oxygen absorber treatment after 2 h (40 min after sampling plus 80 min sample holding time, see Materials and Methods for details) because oxygen absorber removed all the residual oxygen in the vial. This illustrates the importance of oxygen removal from sample vials to prevent further oxidative changes in raw and cooked meat during sample holding time. For raw meat, either helium flush or helium flush plus oxygen absorber can minimize oxidative changes. For cooked meat, however, we suggest to use helium flush plus oxygen absorber treatment when volatiles are analyzed using an automated Precept II.

Lipid Oxidation-Related Volatiles of Raw Meat. In raw turkey breast meat with control treatment (no helium flush or oxygen absorber added), the production of volatile compounds related to lipid oxidation gradually increased during sample holding time (Table 2). However, significant increases in volatiles content in raw meat samples were observed after 640 min or longer of sample holding time. Among the volatiles, hexanal was the major volatile compound influenced by oxidative changes in raw meat during holding time. The amount of total volatiles also was significantly increased after 640 min of sample holding. This result agrees well with the TBARS values of raw meat with control treatment (Table 1). Ahn et al. (1998) reported that lipid oxidation and production of volatile compounds correlated well, and hexanal and total volatiles represented the lipid oxidation status better than any other individual volatile components in irradiated cooked pork patties. Pentanal and hexanal also have been used to determine lipid oxidation in meat (Ang and Young, 1989; Liu et al., 1992; Shahidi and Pegg, 1994).

In raw turkey breast meat with helium flush (Table 3), significant differences in the contents of most of the volatiles were observed after 640 min or longer of sample holding time. Unlike the control treatment, the increases of volatiles over sample holding time were neither clear nor significant. The proportion of each volatile in helium flushed raw meat was significantly different from that of the control: hexanal was not the major volatile, the increase of hexanal over holding time was less, and the amount of total volatiles was less than half of the control treatment after 1280 min of sample holding (Table 2). This indicates that less oxidative changes occurred in raw meat samples with helium flush than the control during holding time. Although the TBARS of raw turkey breast meat was not changed during the 1280-min holding time (Table 1), holding

Table 5. Production of Volatiles in Cooked Turkey Breast Meat with No Helium Flush or Oxygen Remover (Control) during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	560[d]	810[d]	1113[c]	1532[b]	1687[ab]	1922[a]	85.3
hexane	75[c]	89[c]	115[b]	140[b]	141[b]	175[a]	8.2
heptane	125[c]	136[c]	227[bc]	259[b]	368a	397[a]	31.9
propanal	650[f]	1320[e]	1941[d]	2689[c]	3923[b]	5456[a]	139.2
octane	163[d]	205[cd]	258[bc]	303[ab]	323[ab]	370[a]	22.2
2-propanone	954[d]	1001[cd]	1077[bcd]	1147[bc]	1205[b]	1354[a]	40.6
2-butanone + butanal	36[d]	89[c]	104[bc]	124[ab]	136[a]	126[ab]	7.2
2-methylbutanal	53[c]	51[c]	74[bc]	93[b]	105[b]	137[a]	10.3
3-methylbutanal	27[d]	34[cd]	50[bcd]	61[bc]	78[b]	106[a]	8.0
2-methyldecane + pentanal	1866[e]	2478[de]	2950[d]	4060[c]	4995[b]	6797[a]	237.2
hexanal	9983[f]	18977[e]	26611[d]	35532[c]	49883[b]	65094[a]	1707.7
heptanal	279[e]	387[d]	470[d]	626[c]	737[b]	963[a]	34.6
1-pentanol	191[e]	307[de]	422[d]	628[c]	915[b]	1458[a]	42.3
nonanal	324[f]	499[e]	724[d]	879[c]	1094[b]	1359[a]	49.7
1-octen-3-ol	314[e]	491[de]	655[d]	934[c]	1302[b]	1826[a]	67.3
total volatiles	15577[f]	26873[e]	36789[d]	48573[c]	66891[b]	87515[a]	2169.0

[a] Area (ion count × 1000). Samples (3 g) were purged immediately after sampling. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a–f]Different letters within a row are significantly different ($P < 0.05$). SEM, standard error of the mean.

Table 6. Production of Volatiles in Cooked Turkey Breast Meat with Helium Flush during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	715[b]	1231[b]	1540[b]	2637[a]	2431[a]	2996[a]	261.6
hexane	107[b]	169[ab]	175[ab]	315[a]	260[ab]	307[a]	43.1
heptane	155[c]	223[c]	227[c]	394[b]	434[ab]	538[a]	35.3
propanal	220[d]	388[cd]	677[cd]	1051[c]	2268[b]	2972[a]	190.8
octane	190[b]	231[ab]	291[ab]	392[ab]	396[ab]	450[a]	53.3
2-propanone	1306	1301	1224	1250	1250	1250	70.9
2-butanone + butanal	20[ab]	10[b]	28[ab]	36[b]	58[ab]	83[a]	15.5
2-methylbutanal	14[c]	10[c]	20[c]	41[bc]	67[ab]	84[a]	10.9
3-methylbutanal	10[b]	10[b]	18[b]	28[b]	48[ab]	76[a]	9.5
2-methyldecane + pentanal	949[d]	1311[cd]	1667[cd]	2348[c]	3486[b]	4787[a]	321.0
hexanal	3334[e]	7490[d]	9281[d]	15403[c]	21142[b]	25112[a]	996.7
heptanal	66[c]	78[c]	82[c]	255[b]	288[b]	452[a]	37.5
1-pentanol	209[e]	342[de]	401[d]	745[c]	988[b]	1238[a]	50.1
nonanal	344[c]	432[c]	456[c]	703[b]	770[ab]	876[a]	45.1
1-octen-3-ol	614[b]	952[b]	1152[b]	2082[a]	2225[a]	2595[a]	150.0
total volatiles	82528[e]	14174[d]	17237[d]	27678[c]	36108[b]	43817[a]	1697.6

[a] Area (ion count × 1000). Samples (3 g) were flushed immediately after sampling. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a–e]Different letters within a row are significantly different ($P < 0.05$). SEM, standard error of the mean.

helium flushed raw meat sample longer than 640 min produced higher level of volatiles than at 0 min. Therefore, sample-holding time for raw turkey breast meat should be shorter than 640 min with helium flush.

In raw turkey breast meat with helium flush plus oxygen absorber (Table 4), none of the individual volatile and total volatiles except for 1-octen-3-ol, which increased after 320 min of sample holding, changed during the 1280 min sample holding time. This suggests that the use of helium flush plus oxygen absorber is the best treatment to stop oxidative changes in raw meat during sample holding. Overall, the use of helium flush or helium flush plus oxygen absorber was effective in reducing oxidative changes in raw turkey meat during holding time. However, the hexanal peak was masked by 2,6-dimethylheptane when oxygen absorber was added. Therefore, the maximal suggested sample holding time for helium flush would be 10 h, and for helium flush plus oxygen absorber would be 20 h if hexanal alone is not to be used as an indicator for oxidative changes in the raw samples.

Lipid Oxidation-Related Volatiles of Cooked Meat. In cooked turkey breast meat with control (Table 5), significant increases in volatiles were observed after 80 min of sample holding time. In samples held for 80 min, the production of propanal, 2-butanone plus butanal, hexanal, heptanal, nonanal, and total volatiles were significantly higher than those at 0 min. The increased levels of propanal and hexanal in cooked meat during the first 80 min of sample holding time were 2-fold and that of total volatiles 1.7-fold those of 0 min. Significant increases in hexane, pentane, octane, decane plus pentanal, 1-pentanol, and 1-octen-3-ol were observed after 160 min and in heptane, 2-protanone, 2-methylbutanal, and 3-methylbutanal after 320 min of sample holding. This indicates that oxygen removal from the sample vial is more critical in cooked meat than in raw meat. The rapid increase in volatiles agrees well with the TBARS changes in cooked meat under oxygen conditions (Table 1). Mei et al. (1994) suggested that the higher susceptibility of cooked meat, compared with raw meat, could partially be caused by heat inactivation of endogenous antioxidant enzymes such as catalase, glutathione peroxidase, and superoxide dismutase in addition to the structural damage by cooking. Ahn et al. (1998) reported that cooking itself

Table 7. Production of Volatiles in Cooked Turkey Breast Meat with Helium Flush Plus Oxygen Aabsorber during Sample Holding Time in an Autosampler at 4 °C before Purge[a]

	sample holding time (min)						
compound	0	80	160	320	640	1280	SEM
pentane	482	681	606	610	599	481	127.6
hexane	62	65	62	55	51	56	10.1
heptane	111	129	136	118	131	151	20.6
propanal	183	249	312	303	347	284	64.2
octane	333	276	272	252	251	319	43.4
2-propanone	1107	1072	1037	884	718	834	128.9
2-butanone + butanal	72	101	42	14	23	10	21.1
2-methylbutanal	12	39	50	22	20	10	11.2
3-methylbutanal	10	10	17	16	16	22	5.2
2-methyldecane + pentanal	3866	4495	3536	2742	3269	3198	562.4
hexanal	3153	3906	3966	3885	4308	3764	610.4
heptanal	80	44	47	51	65	32	13.0
1-pentanol	151	150	178	176	199	271	36.4
nonanal	201	234	255	196	265	191	25.2
1-octen-3-ol	292	446	516	517	626	626	101.7
total volatiles	10112	11895	11029	9838	10103	10257	1482.5

[a] Area (ion count × 1000). Samples (3 g) were flushed immediately after sampling. Sample vials were held in a sample holder (4 °C) and purged after the designated time. Only the volatiles related to the oxidative changes of meat are listed. $n = 4$. [a,b]Different letters within a row are significantly different ($P < 0.05$). SEM, standard error of the mean.

did not increase lipid oxidation, but structural damages by cooking process enhanced oxygen contact with membrane lipids and accelerated lipid oxidation. Therefore, oxygen contact with cooked meat is more critical for oxidative changes than with raw meat.

In cooked turkey breast meat with helium flush (Table 6), hexanal and total volatiles increased after 80 min, 1-pentanol increased after 160 min, and the rest of the volatiles increased after 320 min or longer of sample holding. The amounts of aldehydes and total volatiles in helium-flushed cooked turkey breast meat were approximately half of those found in controls at each of the holding times. This indicates that helium flush reduced oxidative changes in cooked meat during sample holding time but could not prevent oxidative reactions completely, due to incomplete removal of oxygen in the vial.

In cooked turkey breast meat with helium flush plus oxygen absorber (Table 7), no differences in volatiles related to oxidative changes were observed during the 1280-min sample holding periods. Also, the amounts of individual and total volatiles were smaller than those of control and helium flush treatments at each of the holding time. The oxygen absorber should have removed all of the residual oxygen in the sample vial after helium flush, quickly and effectively. The manufacturer of the oxygen absorber suggested that the complete removal of oxygen in sample vial would take 2−4 h, but we speculate it should have taken much less time than that because of the low residual oxygen in the sample vial after helium flush. Therefore, if meat samples are purged at 40 or 50 °C (temperature at sensory analysis) as suggested previously (Ahn et al., 1999) and residual oxygen in sample vials is eliminated using the combination of helium flush and oxygen absorber, volatiles production by oxidative changes in cooked meat can be prevented almost completely for over 20 h.

CONCLUSION

Residual oxygen in sample vials accelerated oxidative changes and increased volatile production in both raw and cooked meats during sample holding time. However, oxygen removal from sample vials by helium flush and oxygen absorber made the automated Precept II and purge-and-trap dynamic headspace/GC−MS method possible without significant oxidative changes in meat. For automated analysis of volatiles, raw turkey breast meat could be held at 4 °C for up to 10 h if helium flush is used and for over 20 h if helium plus oxygen absorber is used. However, the use of helium flush is preferred over helium flush plus oxygen absorber for raw meat because hexanal, the major indicator for oxidative changes in meat, is masked by a volatile (2,6-dimethylheptane) when an oxygen absorber is used. For cooked turkey breast meat, however, only helium flush plus oxygen absorber is recommended because of the high susceptibility of cooked meat to oxidative change during sample holding time. With helium flush plus oxygen absorber, cooked meat also could be held for about 10 h without changes in volatiles and TBARS.

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