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Analytical Methods

Volatile flavour profile of goat meat extracted by three widely used techniques

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ABSTRACT

Three procedures for the isolation of volatiles from grilled goat meat were compared: dynamic headspace entrainment on Tenax TA, simultaneous steam distillation-extraction, and solid-phase microextraction. Headspace entrainment on Tenax TA extracted the highest number of Maillard-derived volatile compounds. Two hundred and three volatile components were identified; 159 are reported for the first time in goat meat. Most of the volatiles detected (155) were lipid oxidation products, such as hydrocarbons, aldehydes, alcohols, ketones, carboxylic acids and esters. Forty-eight Maillard-derived compounds were identified, comprising pyrazines, pyrroles, thiophenes, furanthiol derivatives, alkyl and alicyclic sulphides, pyridines, and thiazoles. Some reported character impact compounds of cooked meat, e.g., 12-methyltridecanal, (*E,E*)-2,4-decadienal, methional, and dimethyl trisulphide were identified in the volatile profile of goat meat, together with a series of C_2 to C_5 alkylformylcyclopentenes, which have been reported in cooked chicken, pork, beef and lamb, as being important for the characteristic flavour impression of different animal species.

1. Introduction

Goat meat is a highly nutritious meat, with a distinctive and characteristic flavour note, that is well established in many cultures, mainly in Asia, Africa and South America. It is considered to be relatively lean, with a low percentage of fat, around 3 to 5 g/100 g, (Webb, Casey, & Simela, 2005), and has gained popularity amongst consumers from Europe and USA. World goat meat trade has risen significantly from 5000 to 34,000 t over the last two decades (FAOSTAT, 2003).

Although research has been conducted on goat carcass and meat quality (Webb et al., 2005), little attention has been paid to the flavour quality of cooked goat meat. Most of the scientific information on goat meat has been related to its sensorial and nutritional composition; there have also been studies on the effects of pre-slaughter treatments on chemical composition, fatty acid profile and acceptability. Goat meat flavour is generally regarded as being similar to that of lamb or mutton. Tshabalala, Strydom, Webb, and de Kock (2003) reported that the overall flavour intensity of meat from goat was weaker than that from sheep, and concluded that goat meat is a unique meat, which is not interchangeable with meat from lamb.

In contrast to beef, pork and lamb, very little is known about the volatiles of goat meat. In fact, only one paper has so far been published on the volatile compounds of cooked goat meat, emphasising the effect of castration and slaughter age (Madruga, Arruda, Narain, & Souza, 2000). On the other hand, a number of papers have investigated the volatile profile of lamb meat, studying volatile branched-chain fatty acids (Priolo et al., 2004), and the effects of diet (Elmore et al., 2005), breed (Elmore, Mottram, Enser, & Wood, 2000), castration and slaughter age (Sutherland & Ames, 1995).

Various techniques have been applied to evaluate volatile compounds in meat, and a review of them is available (Elmore, 2008). Simultaneous steam distillation-extraction (SDE), dynamic headspace entrainment on Tenax TA, and solid-phase microextraction (SPME) are three widely used techniques for the extraction of volatile compounds in cooked meat.

SDE is considered to be a simple technique which involves small volumes of solvent, efficient stripping of volatiles and quantitative recovery of many compounds; however, since sample distillation is a necessary step, thermal degradation and interaction of reactive compounds may occur leading to artefact formation, as well as losses of low-boiling volatile compounds during concentration steps (Reineccius, 2007). In this methodology, the sample is dispersed in water which is heated to boiling. The steam that is generated carries volatiles with it into a section of the apparatus where the steam condenses in the presence of extracting solvent vapour (Likens & Nickerson, 1964). The co-condensation of volatile-laden steam and extracting solvent results in an effective

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extraction of volatiles. This technique is used less today than in the past, but still has great value (Reineccius, 2007).

Dynamic headspace entrainment on Tenax has been applied to study cooked meat aroma since the 1980s; it probably has been used more than any other aroma extraction technique for the analysis of meat aroma and continues to be widely used. This technique involves purging the headspace of a sample with a purified inert gas, such as nitrogen or helium; followed by collection of the volatiles onto a trap containing a suitable adsorbent, which will retain the volatile analytes carried there by the purge gas. This adsorbent, e.g., Tenax (2,6-diphenylene oxide polymer), normally consists of a porous polymer with low affinity for water and methanol, and high affinity for volatile and semi-volatile compounds (Reineccius, 2007). The volatiles collected on this trap are then thermally desorbed or solvent eluted onto a GC or GC-MS column using a modified injection port. Headspace entrainment on Tenax is sensitive, extracts a wide boiling point range of volatile compounds with short preparation time and artefact formation is minimal; however, a dedicated injection system for traps may be expensive (Elmore, 2008).

SPME was developed and introduced as an alternative to the dynamic headspace technique as a sample pre-concentration method, prior to chromatographic analysis. It integrates sampling, extraction, concentration and sample introduction to the GC. In headspace SPME, an inert needle, coated with an absorbent or adsorbent material, is placed above the food product. Volatiles will migrate from the food matrix to the needle coating and be absorbed or adsorbed. Volatiles will be desorbed from the needle coating by placing the needle in a hot gas chromatograph injection port. SPME is a simple aroma extraction technique, which extracts a wide boiling point range of volatile compounds without artefact formation (Reineccius, 2007).

Several researchers have tried to find out the most appropriate technique to collect volatiles from cooked meat and meat products, drawing different conclusions. Elmore, Papantoniou, and Mottram (2001) concluded that SPME complements headspace adsorption on Tenax as a flavour extraction technique for cooked beef. Dirinck, van Opstaele, and Vandedriessche (1997) found out that SDE extraction should be preferred over dynamic headspace adsorption of volatiles from cured hams. Liu, Xu, and Zhoug (2007) reported that SPME was better than purge and trap using Tenax TA for extracting volatiles in marinated duck, and SPME could complement SDE.

In our laboratory, Headspace entrainment, SDE, and SPME methods have regularly been used to analyse volatiles in cooked meat, including headspace entrainment on Tenax for cooked beef and lamb (Elmore, Mottram, Enser, & Wood, 1999; Elmore et al., 2000, 2004a, 2005), SDE for lamb, beef and pork (Elmore, Mottram, & Dodson, 2004b), and SPME for pork and beef (Elmore et al., 2001). The objective of the present study was to examine the volatile profile of cooked goat meat, extracted by three widely used procedures, and to select the most appropriate extraction technique to be used for future studies on goat meat flavour. The Saanen breed was studied; this breed originated in West Switzerland, being probably the most widely-found dairy breed, and has been introduced into the United Kingdom for milk production (Gall, 1996).

2. Material and methods

2.1. Goat meat

Rump muscle from three castrated British Saanen goats, of approximately 6 months of age, reared indoors on a maize silage-based ration with free access to hay and straw, were provided by the Department of Agriculture of the University of Reading.

Rump muscles were vacuum packed, stored at −18 °C for no more than a period of 30 days, and defrosted overnight at 4 °C, prior to cooking. Chops were cut, weighed and then grilled using a two-plate grill (Cuisinart Griddle & Grill – GR4 U, Wigan, United Kingdom). The two-plate grill was set to "griddle" mode and allowed to reach 180 °C, before chops were grilled. The chops were grilled separately with the grill closed, the chop placed in the middle of the bottom plate and cooked until its centre internal temperature reached 80 °C, measured using a thermocouple type K, mode 3200 K (Digitron Instrumentation Ltd., Torquay, United Kingdom). Cooking to a fixed temperature compensated to an extent for any variations in the thickness and weight between and within the samples, although thicker chops took longer to cook and hence received more surface heat than thinner chops.

The grilled chops were placed on a China plate and left to cool to between 30 °C and 35 °C. After cooling, each chop was minced in an electric bowl chopper, and analysed immediately. For each animal three replicate analyses, for each one of the extraction techniques, were performed.

2.2. Dynamic headspace entrainment on Tenax TA

Samples (20 ± 0.1 g) of minced meat were placed in a 250 ml conical flask with a Dreschel head for the collection of headspace volatiles. The flask was held in a water bath at 60 °C for 1 h, while oxygen-free nitrogen was passed over the sample at a rate of 40 ml/min, sweeping the volatiles onto a preconditioned glass trap (4 mm i.d., ¼″ o.d. × 3.5 mm long), packed with Tenax TA (Supelco, Poole, United Kingdom). A standard of 1,2-dichlorobenzene in methanol (1 μl containing 130.6 ng) was added to the trap at the end of the collection and excess solvent and any water retained on the trap was removed by purging the trap with nitrogen at 40 ml/min for 10 min.

2.3. Solid-phase microextraction (SPME)

The grilled minced goat meat, 35 ± 0.1 g was placed in a 60 ml glass vial, with a screw cap containing one centre hole of 3 mm radius and a Teflon-lined septum. Extractions were carried out with an SPME device (Supelco) containing a fused-silica fibre coated with a 75 μm layer of Carboxen-PDMS. The stainless steel needle, housing the fibre, penetrated the septum, and after equilibration at 60 °C for 5 min, the fibre was exposed to the headspace above the meat for 60 min. After extraction, the SPME device was removed from the meat sample vial and inserted directly into the injection port of the GC-MS. Before the extraction the fibre was conditioned, by heating it in the gas chromatograph injection port at 250 °C for 30 min.

2.4. Simultaneous steam distillation-solvent extraction (SDE)

Minced grilled goat meat (200 g) plus distiled water (750 ml) were added to a 1 l round-buttoned flask. 1,2-Dichlorobenzene (100 μg in 1 μl methanol) was used as internal standard and added to the flask. The sample was extracted for 2 h with a mixture of redistilled 27 ml pentane + 3 ml diethyl ether in an SDE apparatus (Likens et al., 1964). During the extraction the sample was maintained at 100 °C. The SDE extract was stored at −18 °C to remove water as ice crystals, and was concentrated to 1 ml, before being analysed by GC-MS.

Blank analyses were carried out for the dynamic headspace entrainment on Tenax, SPME and SDE extraction procedures.

2.5. Gas chromatography-mass spectrometry conditions

Volatiles collected by dynamic headspace entrainment on Tenax were analysed using a Perkin-Elmer Clarus 500 GC-MS system (Perkin-Elmer, Beaconsfield, United Kingdom) equipped with an automated thermal desorber (Turbomatrix ATD). The Tenax tubes were desorbed at 300 °C (heating rate 40 °C/s) and cryofocused onto a packed cold trap at −30 °C.

A Hewlett-Packard (Palo Alto, CA, USA) 5972 mass spectrometer, coupled to a 5890 Series II gas chromatograph was used to separate and identify the volatiles collected by SDE and SPME.

The 1 μl SDE extract was injected in splitless mode at 250 °C, the spliter opening after 1 min. The volatile compounds on each SPME fibre were desorbed for 3 min in a split/splitless injection port, held at 250 °C, onto a non-polar deactivated fused-silica retention gap (5 × 0.25 mm I.D., Varian, Oxford, United Kingdom), which was attached to a non-polar analytical column. The retention gap contained 5 small loops in a coil, which were cooled in solid carbon dioxide, contained within a 250 ml beaker, which was removed after 3 min. The injection port was in splitless mode, the splitter opening after 3 min. Immediately before the desorption of the fibre, 0.1 μl of an internal standard (1000 ng μl^{-1} 1,2-dichlorobenzene in methanol) was injected into the gas chromatograph.

GC separation of volatiles collected by SPME and SDE methodology was carried out on a VF-5 ms low bleed/MS fused-silica capillary column (5% phenyl/95% PDMS, 60 m × 0.25 mm I.D., 0.25 μm film thickness, Varian). The Tenax TA extracts were analysed in a similar DB-5 non-polar column (60 m × 0.32 mm I.D., 1 μm film thickness, J & W Scientific). The temperature program employed was 2 min at 40 °C, a ramp of 4 °C/min to 280 °C, and held for 10 min. Helium was used as the carrier gas.

The Perkin-Elmer mass spectrometer was operated in electron impact mode with a source temperature of 200 °C, an ionising voltage of 70 eV, and a scan range from *m/z* 29 to *m/z* 350 at 3.33 scans/s. The Hewlett-Packard mass spectrometer also operated in electron impact mode with a lower source temperature (170 °C), and a scan range from *m/z* 29 to *m/z* 400 at 2.05 scans/s. The Tenax TA extract data were acquired and analysed using TurboMass software (Version 4.5, Perkin-Elmer); the SDE and SPME data were acquired and analysed using G1034C Chemstation software (Hewlett-Packard).

Identification of the compounds was based on the comparison of their mass spectra with spectra from authentic compounds previously analysed, spectra from the NIST/EPA/NIH Mass Spectral Database (Version 2.0a, 2002), or spectra published elsewhere. To confirm the identification, the linear retention index (LRI) was calculated for each volatile compound using the retention times of a homologous series of C_6–C_{25} *n*-alkanes, and by comparing the LRI with those of authentic compounds analysed under similar conditions. The approximate quantities of the volatiles were estimated by comparison of their peak areas with that of the 1,2-dichlorobenzene internal standard, obtained from the total ion chromatograms, using a response factor of 1.

3. Results and discussion

Application of dynamic headspace entrainment on Tenax TA, SDE, and SPME techniques for sampling volatiles in cooked goat meat, led to identification by GC-MS of 203 volatile compounds (Table 1). Almost all of these compounds (155) were formed from lipid oxidation (39 aldehydes, 42 hydrocarbons, 26 ketones, 21 alcohols, 10 carboxylic acids, 9 furans, 6 esters, and 2 phenols). Volatile compounds formed via the Maillard reaction (48), included heterocyclic oxygen, nitrogen and sulphur-containing compounds, such as pyrazines (13), thiophenes (5), thiazoles (4), pyrroles (3), pyridines (2), as well as non-heterocyclic compounds, such as Strecker aldehydes, alkanediones, hydroxyketones and aliphatic sulphides (10). Of these compounds, 159 were reported for the first time in cooked goat meat.

Table 1
Numbers of volatiles in cooked goat meat extracted by SDE, headspace entrainment on Tenax and SPME.

Volatiles	Total by chemical class	SDE	Tenax TA	SPME
Lipid oxidation	155	122	100	80
Hydrocarbons	42	37	27	30
Aldehydes	39	39	28	15
Ketones	26	15	19	13
Alcohols	21	13	15	8
Phenols	2	2	0	0
Carboxylic acids	10	9	5	1
Esters	6	0	0	6
Furans	9	7	6	7
Maillard reaction	48	31	37	26
Strecker aldehydes	4	2	4	3
Alkanes Diones	3	3	3	1
Hydroxyketones	1	0	1	1
Pyrrole	3	2	3	2
Pyridine	2	1	0	1
Pyrazine	13	11	11	11
Aliphatic sulphides	10	5	8	3
Thiophenes	5	2	4	3
Alicyclic sulphides	3	3	0	0
Thiazoles	4	2	3	1

The cooked goat meat volatile profile was consistent with the SDE goat extract results of Madruga et al. (2000) in relation to the lipid degradation products, since 81% of volatiles identified previously were present in our cooked goat meat. However, two Maillard-derived compounds reported in the SDE goat extract in that paper, e.g., dipropyl trisulphide and ethyl 1-methylethyl disulphide, were not identified amongst the volatiles extracted by any of the three techniques used in this paper.

The chemical profiles varied with the extraction technique. The number of compounds was higher in SDE and Tenax TA extracts, compared to SPME extracts. In fact, SDE, headspace entrainment on Tenax and SPME gave a wide spectrum of chemical compounds; 153 and 137 compounds were identified by, respectively, SDE and headspace entrainment on Tenax techniques, whereas 106 volatiles were identified using SPME. Furthermore, 68 compounds were detected by all three extraction methods; phenols and esters were, respectively, extracted only by SDE and SPME techniques.

Differences were also observed amongst the total amounts (ng/100 g) of volatiles in goat meat sampled by these procedures. Levels for SDE were 17 and 1.7 fold higher than for SPME and headspace entrainment on Tenax, respectively. The levels for headspace entrainment on Tenax compared to SPME were above those reported by Elmore et al. (2001) in the extracts of cooked beef; they found that the majority of aroma compounds were present at five times greater concentration in the Tenax headspace extract than in the SPME extract. The highest number and amount of volatiles extracted by SDE was due to the exhaustive extraction of volatile compounds, while headspace entrainment on Tenax and SPME methods extract only volatiles that are released into the headspace.

The reproducibility of SDE, Tenax and SPME was tested by performing triplicate analyses of cooked goat meat. The coefficients of variance for most of the peaks ranged from 21% to 33%, although coefficients of variation (CV) ranged from less than 10% for some compounds, to around 100% in others; highest variations were observed for low molecular weight and low-boiling point compounds; headspace entrainment on Tenax showed lower reproducibility comparing to the other two methods. Our results were comparable with those obtained by Elmore et al. (2001) comparing headspace entrainment on Tenax with SPME in cooked beef,

and Dirinck et al. (1997) who used SDE and dynamic headspace sampling techniques in cured hams.

Linear series of saturated hydrocarbons (C_7 to C_{21}), aldehydes (C_5 to C_{17}), alcohols (C_4 to C_9), ketones (C_5 to C_{13}), together with unsaturated series of aldehydes (C_5 to C_{11}) and 2,4-dienals (C_8 to C_{12}), were identified in cooked goat meat, and were mostly present in all three extracts. The volatile components present in the highest amounts in all chromatograms were the lipid-derived hexanal, 1-octen-3-ol, heptanal, octanal, pentylfuran, and 1-hexanol. This was not a surprising finding, since these compounds have been well reported in the literature as being major volatile compounds of cooked meat (Mottram, 1998) and arise from autoxidation of fatty acids present in the intramuscular triglycerides and structural phospholipids.

Aldehydes were the major class of volatiles that occurred in goat meat sampled by SDE, headspace entrainment on Tenax and SPME; the *n*-alkanals were present at a higher level than the unsaturated and aromatic aldehydes. This finding agrees with previous work on sheep meat flavour (Sutherland & Ames, 1995; Elmore et al., 2000, 2005). In addition, aldehydes with high boiling point (>200 °C) were detected in highest concentrations by SDE, but not detected by SPME; low-boiling aldehydes were detected by the three extraction procedures, with the highest concentrations in headspace entrainment on Tenax TA (Fig. 1).

Similar results were observed for other lipid-derived compound, e.g., ketones and alcohols. Those with high boiling points dominated in the SDE extracts, while those of low-boiling point could be extracted by SDE, headspace entrainment on Tenax and SPME, with predominance in Tenax TA. This result was consistent with the result of Liu et al. (2007), comparing volatiles in marinated duck. High boiling ketones identified in SDE extracts, e.g. 2-nonanone, 2-decanone, and 2-tridecanone have been suggested as contributors to mutton flavour by Caporaso, Sink, Dimick, Mussinan, and Sanderson (1977).

The ten straight-chain carboxylic acids identified, mostly by SDE extraction, in goat meat, were also generated from the thermal degradation of triglycerides and phospholipids of meat. Branched-chain representatives, in particular 4-methyloctanoic, 4-ethyloctanoic, and 4-methylnonanoic acids that have been associated with the "goaty/sweaty" odour of cooked lamb and goat meat (Wong, Nixon, & Johnson, 1975), were not detected in the lean tissue of goat. This is probably due to the fact that branched-chain fatty acids occur at high levels in subcutaneous fat, compared to lean meat (Miller, Field, & Agboola, 1986).

Alkylphenols were extracted only by SDE; they have been implicated in the characteristic aroma of sheep meat (Ha & Lindsay, 1991). Sutherland and Ames (1995) reported that phenols and branched-chain fatty acids were associated with the undesirable note of cooked sheepmeat.

Forty-eight Maillard-derived compounds were identified in cooked goat meat and many were present in each of the SDE, Tenax TA and SPME extracts. However, a clear effect of extraction technique on the number and amount of Maillard-derived compounds was observed. Tenax TA extracted a higher concentration of pyrazines, pyrroles, pyridines, and dimethyl sulphides compared to SDE and SPME (Fig. 2). Thiophenes, alicyclic sulphides and thiazoles were well extracted by SDE. These results were consistent with the result of Liu et al. (2007) who reported pyrrole, methylpyrazine, 2-acetylthiazole in SDE extract, but not in SPME extract of roasted and dried duck.

The importance of sulphur and nitrogen-containing compounds in meat flavour has been acknowledged by many workers (Mottram, 1998). Although, they occur in meat at very low concentrations, they are very potent contributors to meat flavour because of their low odour thresholds (Mottram & Mottram, 2002). A large production of pyrazines was detected in goat meat, all being reported for the first time; they were probably involved in the roasted aroma of goat meat, being formed from the condensation of two α-aminoketone molecules produced in the Strecker degradation of amino acids by dicarbonyl compounds (Mottram, 1998). Thiazoles were extracted mainly by headspace entrainment on Tenax technique, and could be associated with the roasted aroma of cooked goat meat. Their formation probably involves the reaction of α-dicarbonyls, or hydroxyketones with hydrogen sulphide and ammonia, formed via the hydrolysis or Strecker degradation of cysteine, and aldehydes (Mottram, 1998).

Even though SDE, headspace entrainment on Tenax and SPME were clearly valuable techniques for isolation of sulphur-containing compounds, 2-furanmethanethiol was only detected in SDE extracts, and other furfuryl sulphides, furans with thiol, sulphide and disulphide substitutions were not found in cooked goat meat. Furthermore, the present results do not show the presence of furanones, pyranones, which were reported by Elmore et al. (2001) and Cerny and Grosch (1992) in cooked beef extracted by SPME and SDE. All of these compounds have been reported as important contributors to meat flavour, acting as flavour contributors or as intermediates to other aroma compounds.

Out of the aroma profile of cooked goat meat, 61 target compounds are listed in Table 2. Aldehydes originating from proteolysis and amino acid degradation of isoleucine and leucine, i.e., 2-methylpropanal, 3-methylbutanal, 2-methylbutanal and 2-methyl-2-butenal, were extracted in highest concentrations by headspace entrainment on Tenax and SPME; these compounds

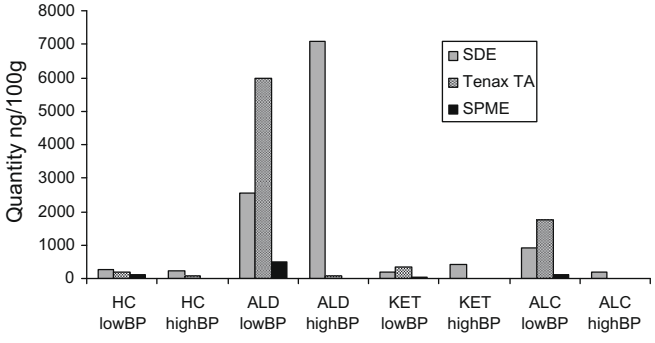

Fig. 1. Distribution of chemical classes of lipid-derived compounds in cooked goat meat prepared by SDE, Tenax TA and SPME (HC = hydrocarbons; ALD = aldehydes; KET = ketones; ALC = alcohols; Low BP = low-boiling point, <200 °C; HighBP = high boiling point, ⩾200 °C).

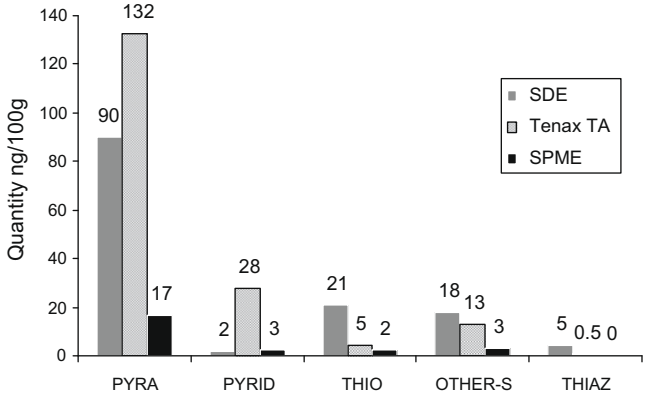

Fig. 2. Distribution of chemical classes of Maillard-derived compounds in cooked goat meat prepared by SDE, Tenax and SPME. (PYRA = pyrazines; PYRID = pyrroles and pyridines; OTHER-S = other sulphur compounds; THIAZ = thaizoles).

Table 2
Amounts (ng/100 g) of selected cooked goat meat compounds extracted by SDE, headspace entrainment on Tenax and SPME.

Identification [a,b]	LRIDB5[c]	ID[d]	SDE[e]	% CV	Tenax TA	%CV	SPME	% CV[f]
Aldehydes								
2-Methyl-propanal [a,b]	<600	A	nd		40	85	5	90
3-Methylbutanal [a,b]	654	A	1	31	89	82	14	15
2-Methylbutanal [a,b]	664	A	2	28	103	81	7	76
Hexanal [b]	804	A	917	78	3906	75	346	24
Heptanal [b]	903	A	432	64	584	68	42	56
Octanal [b]	1004	A	447	67	712	58	31	72
12-Methyltridecanal[a]	1577	B (1)	35	21	nd		nd	
Unsaturated aldehydes								
(*E*)-2-Methyl-2-butenal [a,b]	742	A	nd		0.3	92	nd	
(*E,E*)-2,4-Decadienal [b]	1325	A	180	23	8	22	nd	
5-Ethyl-1-formylcyclopentene [a,b]	1038	B (2)	49	72	11	84	nd	
5-Propyl-1-formylcyclopentene [a,b]	1130	B (2)	8	66	nd		nd	
5-Butyl-1-formylcyclopentene [a]	1228	C	15	41	nd		nd	
5-Pentyl-1-formylcyclopentene [a]	1331	C	5	63	nd		nd	
Aromatic aldehydes								
Benzeneacetaldehyde [a,b]	1051	A	57	28	3	54	0.3	14
Ketones								
2.3-Butanedione [a,b]	<600	A	nd		4	98	nd	
2,3-Pentanedione [a,b]	696	A	nd		1	82	nd	
3-Hydroxy-2-butanone [a,b]	709	A	nd		4	162	3	56
Alcohols								
1-Hexanol [a,b]	868	A	108	39	349	40	18	51
1-Octen-3-ol [b]	980	A	375	24	754	23	61	21
Phenols								
4-Methylphenol (p-cresol) [a,b]	1076	B (3)	0.1	38	nd		nd	
2,6-Dimethylphenol	1116	B (3)	1.2	42	nd		nd	
Furans								
2-Furfural [a,b]	834	A	1	30	0.3	67	0.2	35
2-Pentylfuran [b]	991	A	251	40	99	31	30	17
2-Furanmethanethiol [a]	914	A	1	60	nd		nd	
Nitrogen heterocycles								
N-Methylpyrrole [a,b]	738	A	nd		1	86	nd	
Pyrrole [a]	748	A	nd		2	83	nd	
2-Methyl-1-H-pyrrole [a]	836	A	2	35	24	66	0.1	12
Pyridine [a,b]	750	A	nd		nd		2	23
2-Methylpyridine [a]	819	A	0.1	50	nd		nd	
Pyrazines								
2-Methylpyrazine [a,b]	828	A	2	26	7	27	2	37
2,5(6)-Dimethylpyrazine [a,b]	916	A	31	16	34	12	7	14
Ethylpyrazine [a,b]	917	A	1	17	1	31	0.5	14
2,3-Dimethylpyrazine [a,b]	921	A	3	69	2	38	nd	
2-Ethyl-6-methylpyrazine [a,b]	1001	A	14	37	7	30	1	27
Trimethylpyrazine [a,b]	1006	A	nd		38	69	nd	
2-Ethyl-5-methylpyrazine [a,b]	1007	A	16	12	28	77	2	31
3,6-Dimethyl-2-ethylpyrazine [a]	1086	A	7	16	10	21	2	20
3,5-Dimethyl-2-ethylpyrazine [a]	1087	A	nd		2	86	1	29
2,3-Dimethyl-5-ethylpyrazine [a]	1089	A	6	25	1	14	0.5	52
2,3-Diethyl-5-methylpyrazine [a]	1154	A	2	39	nd		0.1	30
3,5-Diethyl-2-methylpyrazine [a]	1158	A	3	24	1	87	0.2	40
2,5-Diethyl-3-methylpyrazine [a]	1159	A	3	23	nd		0.2	47
Dimethyl sulphides								
Methanethiol [a]	<600	A	nd		1	67	nd	
1-(Methylthio)propane [a]	762	A	0.1	70	nd		nd	
3-Mercapto-2-butanone [a]	819	A	5	57	0.1	29	nd	
3-(methylthio)propanal [a,b]	910	A	2	50	0.4	42	0.2	9
Carbon disulphide [a,b]	<600	A	nd		0.3	39	nd	
Dimethyl disulphide [a,b]	748	A	nd		3	74	2	87
Dimetyl trisulphide [a,b]	981	A	1	58	6	54	1	17
Dimethyl tetrasulphide [a,b]	1242	C	nd		0.4	46	nd	
Thiophenes								
Thiophene [a,b]	669	A	17	21	1	87	nd	
2-Methylthiophene [a,b]	774	A	nd		1	58	1	48
2-Ethylthiophene [a,b]	871	A	nd		2	87	0.2	49

(*continued on next page*)

Table 2 (*continued*)

Identification [a,b]	LRIDB5[c]	ID[d]	SDE[e]	% CV	Tenax TA	%CV	SPME	% CV[f]
2-Pentylthiophene [a]	1167	A	2	37	nd		nd	
Alicyclic sulphides								
3,5-Dimethyl-1,2,4-trithiolane[a,b]	1153	A	4	115	nd		nd	
3,5-Dimethyl-1,2,4-trithiolane[a,b]	1160	A	4	105	nd		nd	
4,6-Dimethyl-1,2,3,5-tetrathiane [a]	1433	C	2	83	nd		nd	
Thiazoles								
5-Methylthiazole [a,b]	821	A	nd		0.2	17	nd	
4,5-Dimethylthiazole [a]	936	A	nd		nd		0	66
2-Acetylthiazole [a,b]	1025	A	3	3	0.1	35	nd	
Benzothiazole [a,b]	1250	A	2	26	0.2	24	nd	

[a] Compounds not previously identified in cooked goat meat.
[b] Compounds previously identified in cooked lamb meat or lamb fat aroma, analysed under similar conditions (SDE, Tenax).
[c] Linear retention indices.
[d] A, mass spectrum and LRI agree with those of an authentic compound ran on DB-5 column; B, mass spectrum agrees with reference spectrum in the NIST/EPA/NIH mass spectral database and LRI agree with those in the literature ((1) Grosch, Zeiler-Hilgart, Cerny, & Guth (1993), (2) Elmore et al. (2005), (3) Kondjoyan & Berdagué (1996)); C, tentative identification where mass spectrum agrees with reference spectrum in the NIST/EPA/NIH mass spectral database.
[e] nd, not detected.
[f] % CV, percentage of coefficient of variation.

probably contributed to goat meat flavour, since they have low threshold and distinctive odour characteristics (malty, pungent, sweet notes). The advantages of headspace entrainment on Tenax and SPME on extraction of minor components with low molecular weight probably resulted from the fact that these techniques involved a simultaneous concentration step, without risk of losses.

Some aldehydes were found only in SDE extracts, e.g., 12-methyltridecanal, and a series of C_2 to C_5 alkylformylcyclopentenes; 5-ethyl-1-formylcyclopentene was extracted also by Tenax. 12-Methyltridecanal was reported to be a character impact component of cooked beef extract analysed by SDE (Guth & Grosch, 1994), and may contribute to the "meaty" aroma of goat meat. (*E,E*)-2-4-Decadienal, which is a powerful roast aroma compound (Mottram, 1998), and contributes to the fatty aroma of cooked beef (Gasser & Grosch, 1988), was extracted by headspace entrainment on Tenax and SDE.

5-Ethyl, 5-propyl-, 5-butyl-, and 5-pentyl-1-formylcyclopentene, are cyclic compounds tentatively identified in cooked goat meat. 5-Ethyl-1-formylcyclopentene was reported in cooked lamb (Elmore et al.,2000, 2005), cooked chicken and pork, where its identity was confirmed by synthesis (Werkhoff, Bruning, Emberger, Guntert, & Hopp, 1993); the propyl homologue was found in cooked beef (Elmore et al., 1999). They are present in quite large concentrations in the headspace and extracts of goat meat.

It was observed that 2,3-butanedione and 2,3-pentanedione were extracted only by headspace entrainment on Tenax; these compounds have been suggested as important intermediates in the formation of pyrazines and thiazoles (Mottram & Mottram, 2002), and as contributors of buttery notes to cooked lamb by Sutherland and Ames (1995).

Eight dimethyl sulphides, five thiophenes, three alicyclic sulphides, one furanthiol derivative, and four thiazoles are reported for the first time as components of goat meat aroma. The sulphur-containing compounds identified in goat meat, are well-known constituents of lamb and beef volatiles (Mottram, 1998). Dimethyl disulphide, and trisulphide, which are odorous compounds found in high concentrations, probably will contribute to the meaty aroma of goat meat; 3-(methylthio)propanal has been reported as being an important contributor to roast and shallow-fried beef aroma (Mottram & Mottram, 2002).

Amongst the alkyl sulphides, only 3-(methylthio)propanal and dimethyl trisulphide were extracted by SDE, headspace entrainment on Tenax and SPME; low molecular weight sulphur-containing compounds, which are well-known to be essential intermediates in the formation of many heterocyclic compounds during the Maillard reaction (Mottram & Mottram, 2002), e.g., sulphur dioxide, methanethiol and carbon disulphide, were detected only in the Tenax TA extracts. The only mercaptoketone (3-mercapto-2-butanone) identified in cooked goat meat was extracted by both SDE and headspace entrainment on Tenax; it may be involved in the formation of 4,5-dimethyl-2-alkylthiazoles. Furthermore, alicyclic sulphides and 2-furanmethanethiol were extracted by SDE from cooked goat meat. It seemed that apart from thiophenes, SPME was not a suitable technique for extraction of sulphur-containing compounds in cooked goat meat.

The alicyclic sulphides were extracted only by SDE, while the thiophenes were found in Tenax TA and SPME extracts, and probably contribute to the overall flavour of goat meat, providing sulphurous notes to their flavour.

4. Conclusions

The application of three procedures for isolation of volatiles from cooked goat meat led to identification and quantification of 203 compounds, amongst them 159 were identified for the first time in goat meat. The extract profile varied with the extraction technique. Headspace entrainment on Tenax and SPME methods provided better extraction for volatiles with low molecular masses, while SDE extracted more high boiling volatiles. Phenol compounds, reported as contributing to mutton/goaty aromas, were extracted only by SDE. Higher numbers of Maillard-derived compounds, such as pyrazines, pyrroles, pyridines, and alkyl sulphides, were found with headspace entrainment on Tenax, compared to SDE and SPME. In this study, headspace entrainment on Tenax and SDE methods may well complement each other; SPME is an easy cheap, clean method to use, and would be recommended for use when comparing samples, or when a thermal desorption injector is not available. Some character impact compounds, 12-methyltridecanal, (*E,E*)-2,4-decadienal, 3-(methylthio)propanal, dimethyl trisulphide, and an interesting series of C_2 to C_5 alkylformylcyclopentenes, were identified for the first time in the goat meat volatile profile.

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