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Trace-level determination of polar flavour compounds in butter by solid-phase extraction and gas chromatography–mass spectrometry

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Abstract

Volatile compounds are responsible for the aromas of butter. A simple technique for the determination of these components is described which is based on solid-phase extraction (SPE) after melting of the butter and separation of the aqueous phase from the fat. Volatile flavours present in the water fraction are collected by off-line SPE on cartridges packed with a copolymer sorbent. After desorption with 500 μl of methyl acetate, 1-μl aliquots are quantified and/or identified by gas chromatography–mass spectrometry. The procedure was tested with respect to recovery, linearity and limit of detection in real-life samples using five polar model analytes. It allows the characterisation of polar flavour compounds in butter prior to and after heat treatment at 170°C. From the five model compounds, vanillin, traces of diacetyl and maltol were found to be present in the butter samples. After heat treatment 500–1000-fold increased concentration of maltol, and substantial amounts of furaneol were detected.

Keywords: Food analysis; Butter; Aroma compounds

1. Introduction

Volatile flavour compounds in samples such as butter are determined by many different methods. Gas chromatography (GC) is usually the separation technique of choice because of the volatile nature of the analytes of interest. The high separation efficiency, and the availability of several sensitive detectors, which allow the detection of trace components at a level of 1 ng or less, also favour the use of GC [1–3]. When GC is coupled to mass spectrometry (GC–MS), confirmation and/or provisional identification can be achieved [4,5].

Polar flavours are, generally speaking, present in the aqueous phase of the samples. The complex composition of butter and related types of sample makes the isolation of the analytes, and, especially, the enrichment of the analytes of interest rather difficult. Separation from the fat with its rather high-boiling triglycerides is mandatory because the presence of even traces of fat can damage the GC column. In addition, sample pre-treatment should not cause losses of the most volatile analytes. Various methods have been used to achieve these goals, e.g. headspace analysis [6], steam distillation and supercritical fluid extraction [7], trapping on a porous polymer [8], solid-phase extraction (SPE) over resins [9,10], purge-and-trap techniques [11] and distil-

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lation-plus-extraction [12]. Although extraction and isolation in the gas phase using techniques such as purge-and-trap and (static or dynamic) headspace is attractive for the determination of volatile compounds in food and beverages, the main disadvantage is the low sensitivity with respect to analytes of somewhat lower volatility. Distillation-plus-extraction has the advantage of being rapid, but artefact formation due to thermally induced changes can occur quite easily. Distillation and liquid–liquid extraction are well suited to isolate and monitor aroma components, but they are rather laborious. Furthermore, quite stable emulsions are often formed and phase separation then becomes time-consuming.

Although SPE has, (to our knowledge), never been used for a complex sample such as butter, it has various attractive features. The solid supports used in SPE are bonded silicas and various polymeric resins of varying hydrophobicity and with different selectivities. Proper selection of the SPE sorbent should allow sufficient enrichment of the flavour compounds present in butter at the trace level and, next, the desorption with a relatively small volume of organic solvent to achieve a considerable preconcentration of the analytes of interest.

The aim of the present work is to study and evaluate the characteristics of five types of sorbents; octyl-, octadecyl-, cyano- and amino-modified silicas and a styrene–divinylbenzene (PS–DVB) copolymer, for the determination of polar aromas in butter. Five test analytes were selected which represent various classes of compounds, some of which may be found in butter. Two sorbents, SDB-1 and C_{18}-bonded silica, were selected for the final study on the off-line combination of SPE and GC–MS. The most important flavours in fresh and heat-treated butter were identified. This helps to provide insight into the influence of heating on the flavour profile of butter.

2. Experimental

2.1. Reagents

Methyl acetate (J.T. Baker, Deventer, The Netherlands) was freshly distilled and HPLC-grade water was prepared by purifying demineralised water in a Milli-Q (Millipore, Bedford, MA, USA) filtration system. Helium carrier gas (99.999% purity) was supplied by Hoekloos (Schiedam, The Netherlands). Vanillin and diacetyl were from Fluka (Zwijndrecht, The Netherlands), maltol and furaneol from Acros (Geel, Belgium), and sotolon from Aldrich (Brussels, Belgium). All flavours were of 95–99% purity. Stock solutions of standards were made at a concentration of 0.5–2 mg/ml by weighing and dissolution in methyl acetate or HPLC-grade water. They were kept in the dark at 4°C for a period of maximally 4 months.

Two different types of fresh butters, ‘gezouten roomboter’ and ‘grasboter’ (Melkunie, Breda, The Netherlands) were purchased from a local supermarket.

2.2. Preparation of butter extracts

Amounts of butter ranging from 25 to 500 g were melted at 40°C in a water bath, in the dark. The liquid butter was centrifuged (4000 rpm) for 20 min; then, the fat was removed. The water phase was first filtered over a 0.45-μm filter, and next over several 0.2-μm filters. Typically 2 ml of butter could be handled by one filter. The filtered aqueous sample was stored in the dark at 4°C prior to analysis. Per 100 g of butter an average of 7 ml of water phase was obtained after centrifugation and filtration. This corresponds with ca. 35% of the original amount of water present in the butter (which is ca. 20%, v/v).

Several butter samples were treated at high temperature to study the formation of polar flavours. To this end, 25 g of butter were heated at 100°C to evaporate all water and, next, for 5 min at 170°C. After cooling to ambient temperature, an aliquot of the sample was taken, and 20% (v/v) of water were added to re-establish the initial proportion. After mixing, the treated sample was prepared following the above procedure.

2.3. Solid-phase extraction procedure

Prior to use, disposable cartridges with a volume of 1 ml containing 100 mg C_{18}-, C_8-, NH_2- or CN-bonded silica, or 50 mg of SDB-1 (PS–OVB copolymer) (all cartridges were from J.T. Baker, The Netherlands) were conditioned by rinsing with 2 ml of methanol and, next, 2 ml of HPLC-grade water.

An aqueous solution (2 ml) containing the analytes was loaded on the SPE cartridge (2 ml/min) via vacuum suction. Clean-up was obtained by flushing the cartridge with 1 ml of HPLC-grade water (2 ml/min). The cartridge was dried for 15 min at room temperature under vacuum. Finally, the flavours trapped on the column, were desorbed with 0.5–1 ml of methyl acetate (0.5 ml/min) and the extract dried by adding 1 g of anhydrous sodium sulphate. A 0.5–1 μl volume was injected with an autosampler in the GC–MS system, using the cold on-column technique. The total procedure required 4–5 h, but up to eight samples could be handled simultaneously.

Breakthrough curves and elution profiles were recorded at the 5-μg/ml level, while recovery was studied at a level of 400 ng/ml. Detection limits (signal-to-noise ratio, 3:1) were determined using spiked aqueous phases of butter.

2.4. GC–MS

Separation and detection/identification were achieved on a Hewlett-Packard (Palo Alto, CA, USA) 5890 Series II gas chromatograph equipped with a 50 m×0.32 mm I.D. fused-silica capillary column coated with DB-1 (crosslinked methylsilicone gum; d_f, 1 μm) and a HP-5971A mass-selective detector. Conditions were as follows: injector temperature, 53°C in the 'oven-track on' mode; GC column temperature, 50°C (2 min), at 10°C/min to a final temperature of 280°C (3 min); transfer line temperature, 280°C; ion source temperature, 200°C; carrier gas, He at 48 kPa in the 'constant flow on' mode (1.5 ml/min). Analyses were performed in the electron ionisation (EI) mode; the ionisation voltage was 70 eV. Both full-scan acquisition (m/z 35–350) and selected ion-monitoring (SIM) were used for detection.

3. Results and discussion

3.1. GC–MS of model flavour compounds

Next to diacetyl, furaneol, maltol and vanillin, which always are present in butter samples, sotolon was added in the test mixture because (i) it is an isomer of furaneol, (ii) it is not expected in butter samples and (iii) it is occasionally present in soy sauce. Some analytical characteristics and relevant GC–MS information is reported in Table 1. As regards the EI mass spectra, furaneol and maltol exhibited $[M]^{+\cdot}$ as the base peak. Diacetyl, sotolon and vanillin generated $[M\text{-}MeCO]^{+\cdot}$, $[M\text{-}CO_2H$ or $C_2H_5O]^{+\cdot}$ and $[M\text{-}H]^{+\cdot}$ as the base peak, respectively. As regards the GC separation, all peaks (also those of sotolon and maltol) were well resolved. Because numerous peaks can be expected in the real samples, no attempt was made to reduce the present run time of ca. 16 min. One should add that diacetyl elutes very close to the solvent peak and that its mass spectrum is characterised by two peaks only. Problems with respect to detection/identification can therefore be expected.

Table 2 summarises the limits of detection (LODs; signal-to-noise ratio, 3:1) for all flavours obtained in GC–EI-MS under full-scan (m/z 35–350) and SIM conditions. They were calculated from 50 pg–5 ng injections. Under full-scan conditions, the LODs varied from 100 pg to 1 ng when using the total ion current (TIC) trace. This values could be improved 2–15-fold by using ion traces, which is generally preferred in target analysis. The best result for furaneol is somewhat disappointing, being about an order of magnitude higher than that for the other model analytes. Finally, when using SIM (for m/z values, see Table 2), LODs of 2–10 pg could be obtained for all flavour compounds. This can be

Table 1
Trivial name, molecular weight, log K_{ow}, GC retention time and mass spectral data of volatile flavours used as model compounds

Flavour	M_r	log K_{ow}[a]	t_R (min)	Base peak	Other major ions (rel. abund.%)		
Diacetyl	86	−1.37	2.8	43	86 (15)		
Furaneol	128	1.07	10.6	128	43 (65)	57 (65)	85 (17)
Sotolon	128	0.33	11.3	83	55 (90)	43 (65)	128 (60)
Maltol	126	0.02	11.6	126	71 (30)	97 (27)	43 (25)
Vanillin	152	1.28	15.9	151	152 (92)	81 (40)	109 (35)

[a] Calculated via Clog P (version 1.0, Biobyte; CA, USA).

Table 2
Detection limits (pg; S/N 3:1) of the five model flavours under full-scan (m/z 35–350) and SIM conditions (numbers in brackets represent m/z values)

Flavour	Full-scan LODs (pg)					SIM LODs (pg)	
	TIC	Ion extraction					
Diacetyl	150	10 (43)	20 (86)			2 (43)	5 (86)
Furaneol	1000	500 (128)	1000 (43)	1250 (57)		10 (128)	15 (43)
Sotolon	500	85 (83)	120 (55)	100 (43)	300 (128)	6 (83)	10 (128)
Maltol	300	35 (126)	150 (71)	370 (97)	1000 (43)	6 (126)	24 (71)
Vanillin	100	20 (151)	25 (152)	170 (81)	120 (109)	3 (151)	3 (152)

called satisfactory for the trace-level study we have in mind. As regards identification, the ion extraction data of Table 2 show that, with the diagnostic ions available, 0.5–1 ng will in principle be sufficient for analyte recognition.

3.2. Selection of SPE sorbent

Since the volatile flavours of interest differ widely in polarity as well as other physicochemical characteristics, five mutually rather different sorbents were tested with regard to breakthrough and desorption characteristics. Enrichment studies were done with spiked real-life samples, i.e. spiked aqueous phases of butter. The sample volume was limited to 2 ml (spiking level, 5 mg/kg) to prevent losses due to early breakthrough as much as possible. After loading and washing of the SPE cartridges, a 15-min drying step was used to avoid the risk of introducing some remaining water into the GC. As a further safety measure, the methyl acetate extract was dried with anhydrous sodium sulphate. Recoveries, which have been summarised in Table 3 for four of the test analytes, were calculated after subtraction of the signal obtained for a control sample (blank butter).

Table 3
Average percent recoveries (n=3) of flavours on five sorbent types using preconcentration of 2 ml of aqueous phase of butter spiked at 5-mg/kg level[a]

Flavour	NH_2	CN	C_8	C_{18}	SDB-1
Furaneol	–	20	15	47	81
Sotolon	–	14	5	55	77
Maltol	–	13	10	46	85
Vanillin	19	35	12	56	88

[a] Condition: loading of sample at 2 ml/min; desorption with 1 ml methyl acetate at 0.5 ml/min; GC–MS, see text.

Diacetyl is not included because interferences caused by co-eluting compounds were observed in essentially all experiments. Even with a mere 2 ml of sample, the recoveries of all flavours on C8- and CN-bonded silicas were less than 35%. Whit NH_2-bonded silica, only vanillin, the analyte with the highest log K_{ow} in the test set (log K_{ow} 1.28), was partly recovered. Furaneol, sotolon and maltol were not recovered at all, which is no doubt due to breakthrough during preconcentration and the additional washing. Much more promising results were obtained with C_{18}-bonded silica for which recoveries ranged from 46 to 64%, and with SDB-1, for which sorbent fully satisfactory recoveries of 77–88% were obtained. Repeatability was satisfactory, i.e. below 5% even when recoveries were low. Not unexpectedly, they were best with the copolymer sorbent (0.5–3.5%). Further optimisation of the SPE procedure was carried out with SDB-1 and C_{18}-bonded silica. This part of the study included the recording of the complete breakthrough curves and desorption profiles. As was to be expected on the basis of the data of Table 3, SDB-1 consistently gave the higher breakthrough volumes, viz. (7 ± 1) ml for all five test analytes (Fig. 1(A)). Actually, the findings for diacetyl should be interpreted with some care: the 40% relative response at 10 ml – when all other flavours are reduced to some 10% – indicate that the interference referred to above, influenced the results. For the rest, it is interesting that analytes with log K_{ow} as far apart as vanillin (1.28) and maltol (0.02) behave in a closely similar way.

Typical desorption curves, recorded with methyl acetate as the desorption solvent, are shown in Fig.1(B). Elution of all analytes was achieved with 0.4 ml (diacetyl) or 0.5 ml (other flavours) of organic solvent. Obviously, analyte concentration factors of

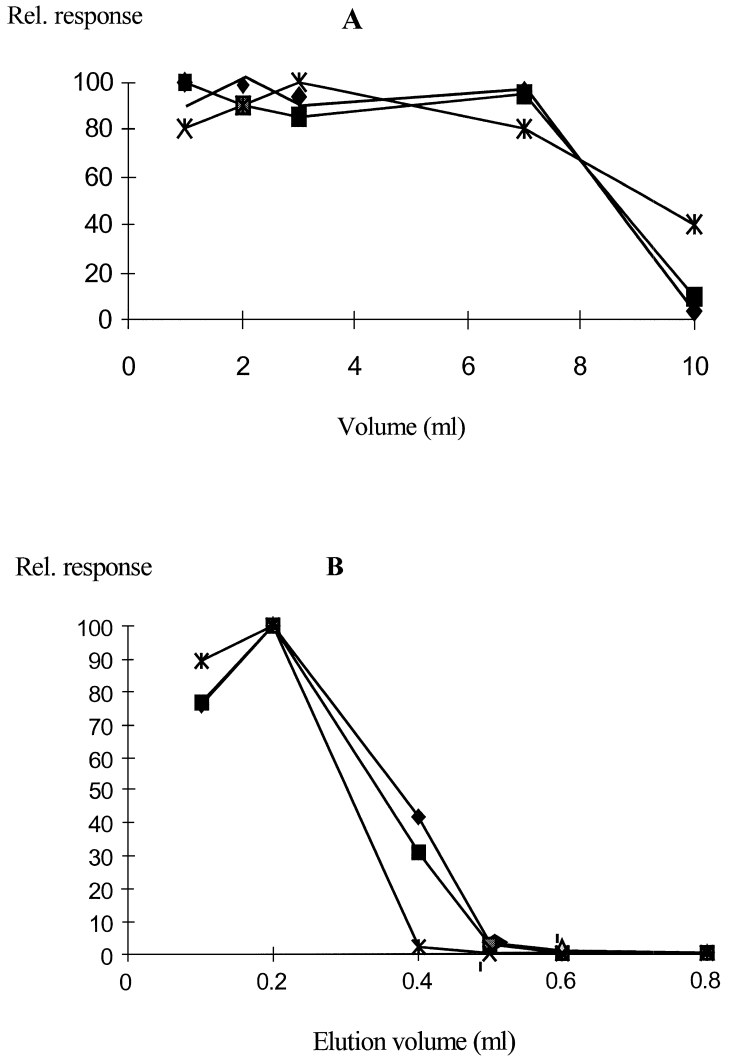

Fig. 1. (A) Breakthrough curves of diacetyl (*), maltol (■) and vanillin (♦) on SDB-1; aqueous sample spiked at 5-μg/ml level. (B) Desorption curves of the same analytes from SDB-1; desorption with methyl acetate.

about 7/0.5=14 can be achieved by means of the present procedure.

3.3. Analytical data

The linearity of the total procedure was tested by trace enrichment of the five selected flavours in spiked 25-g amounts of butter in the concentration range 0.1–4 mg/kg (five data points). The correlation coefficients of the calibration curves (response of quantification ion vs. concentration of analyte)

Table 4
Calibration data for polar flavours (0.1–4 mg/kg) after treatment of 25 g of butter at 40°C

Flavour	Calibration equation	R^2
Diacetyl	NA[a]	NA
Furaneol	$y=(13.483\pm13)x-2.9577$	0.995
Sotolon	$y=(23.133\pm23)x+2.8091$	0.997
Maltol	$y=(34.943\pm35)x-1.9468$	0.999
Vanillin	$y=(28.843\pm29)x+5.3806$	0.998

[a] NA: not applicable to quantification.

were fully satisfactory (R^2>0.995) for all analytes with the exception of diacetyl (Table 4). As will also be discussed below, diacetyl eluted very close to the solvent peak in a region which was fully occupied by other volatile sample constituents. Moreover, neither the base peak (m/z 43) nor the molecular weight peak (m/z 86) were selective enough for unambiguous peak identification.

The repeatability of the total procedure, that is SPE (on SDB-1) with subsequent GC–full-scan-MS, was tested at the 0.4-mg/kg level. When using 25 g butter, the relative standard deviations (RSDs) were 9, 8, 4 and 2% (n=5) for furaneol, sotolon, maltol and vanillin, respectively. The average recovery of the flavours was found to be about 80% (Table 3). The detection limits (signal-to-noise ratio, 3:1) of all test flavours (except diacetyl) were in the range of 20–100 μg/kg. If SIM (for m/z values, see Table 2) was used instead of full-scan acquisition, detection limits improved to 3–12 μg/kg.

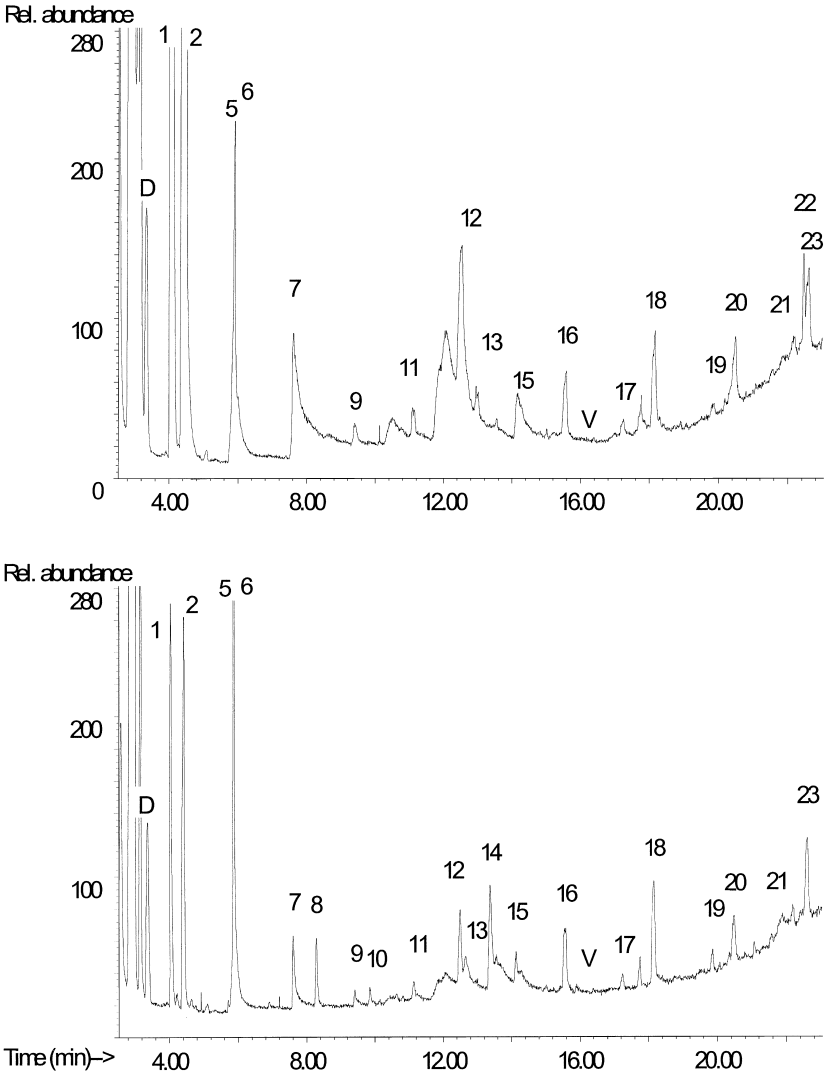

Fig. 2. TIC traces of flavours present in aqueous phase of (top) 'grasboter' and (bottom) 'gezouten roomboter' (each 2 ml water phase which is equivalent to 25 g butter). For peak assignment, see Table 5. Conditions for SPE and GC–MS, see Section 2.

3.4. Applications

3.4.1. Aqueous phase of butter

As an illustration of the practicality of the procedure, the aqueous phases of two 'grasboter' and one 'gezouten roomboter' butter were analysed. Two typical results are shown in Fig. 2. As regards the test analytes, vanillin was identified in all butter samples by using background-subtracted spectra of the peak found via reconstructed ion traces. Diacetyl could, again, not be quantified because of strongly interference signals. However, after careful baseline subtraction, the peak labelled D could be identified as diacetyl. The presence of diacetyl and vanillin was confirmed by re-analysis under SIM conditions, the selected m/z values being 43 and 86, and 151 and 152, respectively (see Table 2). A typical result is shown in Fig. 3, which clearly illustrates the general problem of using m/z values below 50–60. Finally, maltol was identified in one sample. It was quantified via the reconstructed ion trace of m/z 126, and found to be present at a level of 15 μg/kg. As regards vanillin, quantification via the m/z 152 ion trace

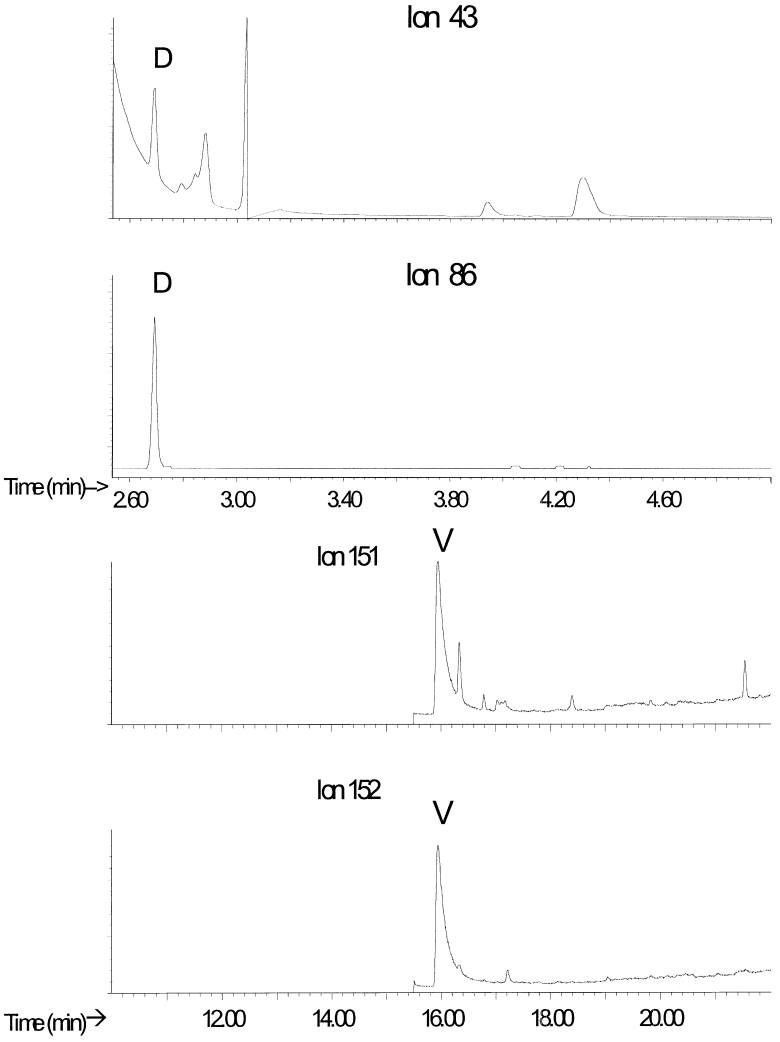

Fig. 3. EI extracted ion chromatogram of diacetyl (m/z 34 and 86) and vanillin (m/z 151 and 152) in SPE extract from 'grasboter' under SIM conditions. For details, see text.

gave concentrations of ca. 50 μg/kg in all three samples.

Some 20–25 compounds could be identified in the three samples on the basis of their mass spectra. Most of the major peaks in the TIC chromatograms were members of the homologous series of fatty acids, methyl ketones and alcohols. The peaks of the fatty acids were rather broad and, probably, obscure other sample constituents. Volatile analytes (retention indices below 1000) were difficult to separate from the solvent peak in the present system. Selection of a thick film column would have reduced this problem, but the determination of one of the target analytes, vanillin, would then have been impossible. No further attempts were made to resolve these peaks. The qualitative and quantitative data for 23 main peaks are summarised in Table 5. Quantification was done using response factor with respect to the five model compounds used in this study. The GC–MS patterns of the two 'grasboter' samples, which were bought 4 months apart and, thus, were expected to show notable differences in composition, were found to be essentially the same (see Table 5).

3.4.2. Heat treatment of butter

Heat treatment has an important effect on food aroma: flavours initially present in the sample are decomposed or converted into other compounds, and many new compounds are formed [13–17]. To illustrate that the present method can be used to study the influence of heat processing on the generation of flavours from precursors, a 'grasboter' and a 'gezouten roomboter' butter sample were heat-treated for 5 min at 170°C (for details, see Section 2.2). As an example, the result for 'grasboter' is shown in Fig. 4. Comparison with the earlier analysis of the same sample, which is depicted in Fig. 2 (also see Table 5), reveals a completely different pattern

Table 5
Analytes identified in aqueous phase of butter samples by SPE plus GC–MS (numbers correspond with peak numbers of Fig. 2)

No.	Compound	t_R (min)	Major ions (m/z)	Concentration (mg/kg)	
				'Grasboter'	'Roomboter'
1	1-Methoxy-2-propanol	4.0	45[a]	7.4	2.7
2	3-Hydroxy-2-butanone	4.4	43, 45[a]	6.3	3.6
3	1-Ethoxy-2-propanol	5.2	45[a], 79	6.6[b]	–
4	2-Ethoxy propane	5.5	45[a], 73	1.2[b]	–
5	Butanoic acid	5.8	60[a], 73	2.5	4.5[c]
6	2,3-Butanediol	5.8	45[a]	4.7[b]	4.5[c]
7	Dimethylsulfone	7.6	79[a], 94	1.3	0.7
8	2-Methyl-2,4-pentandiol	8.3	43, 59[a]	0.1	0.6
9	Hexanoic acid	9.4	60[a], 73	0.8	0.1
10	2-(2-Ethoxyethoxy)ethanol	9.8	45[a], 59, 72	0.6	0.1
11	6-Methyl-2H-pyran-tetrahydro-2-one	11.1	42[a], 70	0.6	0.1
12	Benzoic acid	12.5	77, 105[a], 122	2.2	0.8
13	Octanoic acid	12.7	60[a], 73	0.1	0.1
14	Triglycol	13.4	45[a]	0.4	1.4
15	Nonanoic acid	14.1	60[a], 73	0.3	0.2
16	Decanoic acid	15.6	60[a], 73, 129	1.2	0.5
17	Unknown	17.2	99[a], 71	0.4	0.3
18	Dodecanoic acid	18.1	60[a], 73[a], 129	1.8	1.3
19	Unknown	19.8	99[a], 71	0.3	0.1
20	Tetradecanoic acid	20.5	60[a], 73[a], 129	0.9	0.1
21	Unknown	22.2	99[a], 71	0.6	0.1
22	1,2-Benzenedicarboxylic acid	22.4	149[a]	0.6	–
23	Hexadecanoic acid	22.6	60[a], 73, 129	0.8	0.5

[a] Base peak in the EI spectrum.
[b] Found in one of two samples tested only.
[c] Sum of areas of peaks 5 and 6; compounds not separated on DB-1 column (see Fig. 2).

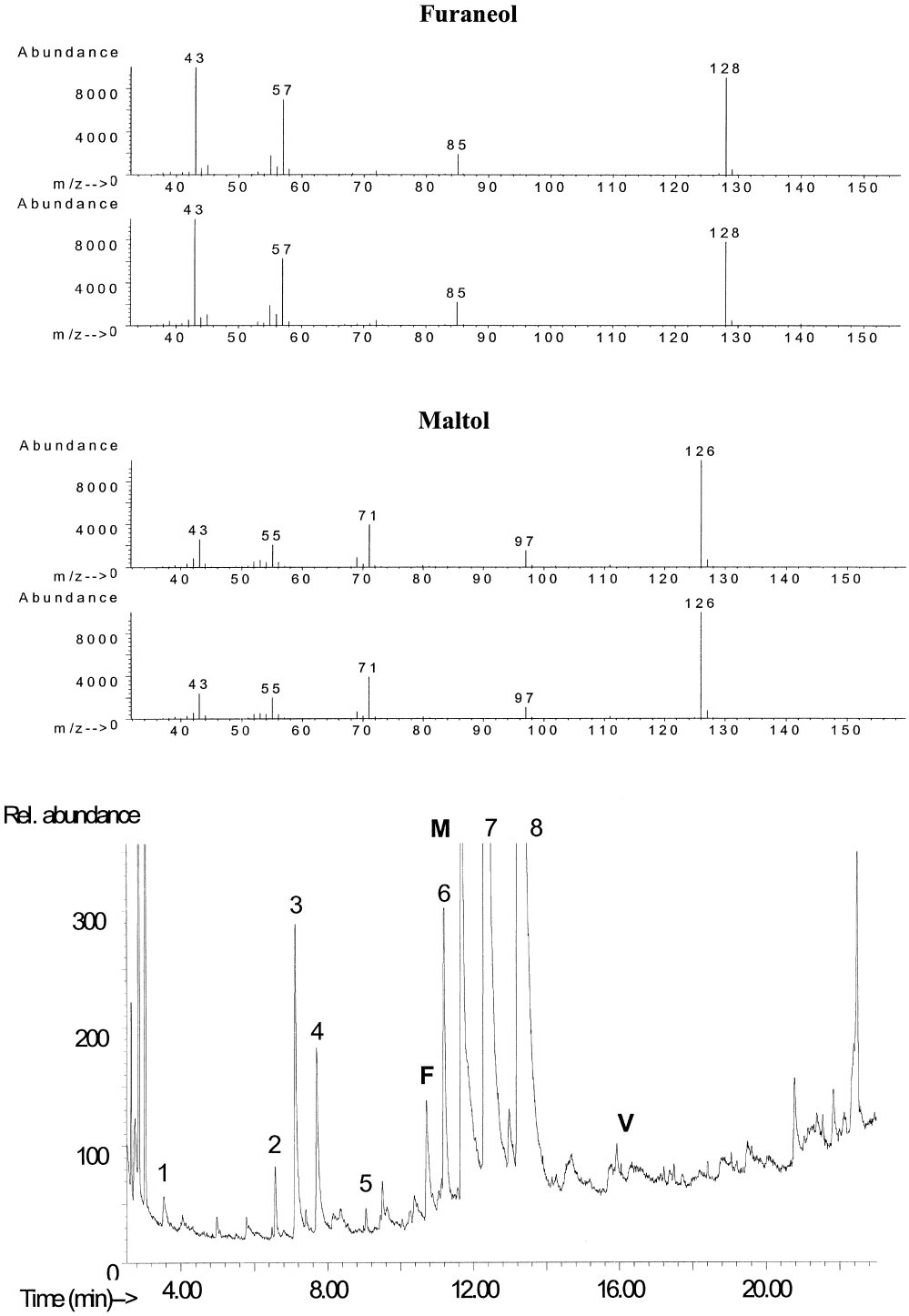

Fig. 4. GC–EI-MS chromatogram of flavours in SPE extract in methyl acetate of 'grasboter' treated at 170°C. Conditions: see Section 2. **1**=2-propanone-1-hydroxy; **2**=furfural; **3**=furfuralcohol; **4**=cyclopentanone; **5**=5-methylfurfuraldehyde; **6**=1-(2-furanyl)-2-hydroxy-ethanone; **7**=2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one; **8**=5-hydroxymethylfurfuraldehyde; **F**=furaneol; **M**=maltol; **V**=vanillin. Mass spectra: (top) sample spectra, (bottom) library spectra.

of the flavours. Some ten compounds could be identified in the heat-treated butter. The result of mass spectral identification is summarised in the legend to Fig. 4. Closely similar results were obtained for the 'gezouten roomboter'. It is interesting to note that two of the model compounds used in this study, furaneol and maltol, were produced in rather large quantities, especially maltol. The sample and Wiley library spectra are included in the figure. Quantitative analysis showed that the concentration of maltol increased 600–1050-fold, viz. to about 15 mg/kg, in both butter samples by the 5-min heating at 170°C, while furaneol was exclusively present in the heat-treated sample, viz. at levels of 4 mg/kg. The concentration of vanillin, on the other hand, remained essentially the same, and the small amount of diacetyl initially present, completely disappeared. No sotolon was detected in any sample, either with or without heat treatment.

Most of the other flavours produced are furanic and pyranic derivatives such as furfural, furfuralcohol, 5-methylfurfualdehyde, 1-(2-furanyl)-2-hydroxyethanone, 5-hydroxymethylfurfuraldehyde and 2,3-dihydro-3,5dihydroxy-6-methyl-4H-pyran-4-one. None of these were present in the original butter samples. On the other hand, flavours present in the aqueous phase of these original samples, such as members of the homologous series of fatty acids, methyl ketones and alcohols (see Fig. 2), were completely lost, either due to evaporation [18] or consumption by means of a chemical conversion reaction.

3.4.3. Sauce samples

In order to illustrate briefly that the present method can also be used to study flavour compounds in related sample types, a soy sauce sample was analysed. In this case sample handling could be limited to 1:1 dilution with HPLC-grade water. Fig. 5 shows the GC–MS chromatogram obtained after extraction of 1 ml of the diluted sauce under the same conditions as described for butter samples. Some 19 compounds could be identified on the basis of their mass spectra, following the procedures discussed above. One of the compounds was maltol, which was present at a level of ca. 40 μg/kg. As is evident from the legend to Fig. 5, most of the major peaks in the TIC trace of the soy sauce were methyl carboxylic acids, methyl ketones and alcohols, ben-

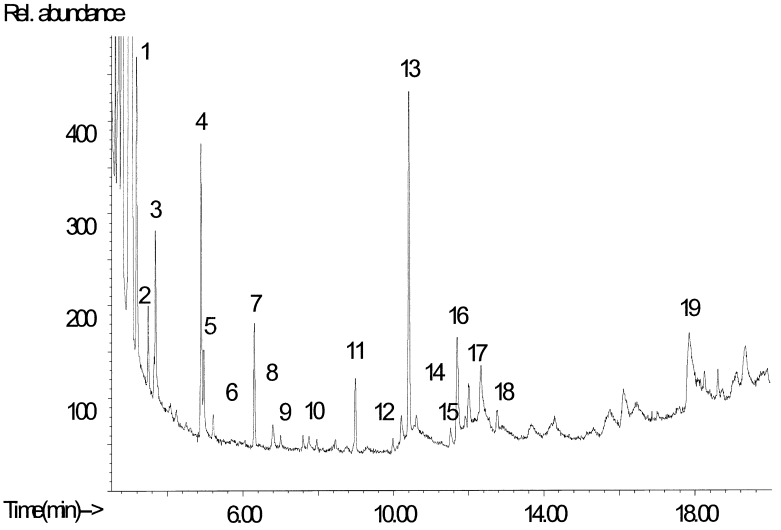

Fig. 5. GC–EI-MS chromatogram of flavours in SPE extract in methyl acetate of 1 ml of soy sauce. Conditions: see Section 2. **1**=1-propanol-2-methyl; **2**=butanal-3-methyl; **3**=1-butanol; **4**=1-butanol-3-methyl; **5**=1-butanol-2-methyl; **6**=propanoic acid-2-methyl; **7**=propanoic acid-2-hydroxy-,ethyl ester; **8**=butanoic acid-3-methyl; **9**=butanoic acid-2-methyl; **10**=tetrahydro-2-furanone; **11**=benzaldehyde; **12**=2,5-hexanedione; **13**=benzeneacetaldehyde; **14**=2-pyrolyl methyl ketone; **15**=maltol; **16**=benzene-ethanol; **17**=3,4-dimethoxy-2-methylfuran; **18**=4H-pyran-4-one,3-hydroxy-2,6-dimethyl (methyl maltol); **19**=vanillic acid.

zaldehyde derivatives and furanic and pyranic derivatives.

4. Conclusions

The present approach of an off-line combination of SPE and GC–EI-MS has been shown to be a powerful tool for the trace-level identification and quantification of volatile flavours in the aqueous phase of butter. In order to obtain sufficient trace enrichment of the polar flavour compounds of primary interest, SPE should be performed with SDB-1 copolymer as the sorbent. Under the present conditions, concentration factors of 10 to 15 could be obtained. As regards the analysis of fresh and heat-treated butter or sauce samples, no technical or analytical problems were encountered, and no injector fouling or deterioration of the separation column did occur.

It is an aspect of much practical relevance that the significant changes of the flavour compound pattern caused by thermal treatment, can easily be charted with the present analytical procedure. The rapid formation of appreciable amounts of furaneol and maltol can be cited as a relevant example.

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