



HAL
open science

SPME analytical method for the determination of Hexanal in hazelnuts as indicator of the interaction of active packaging materials with food aroma compounds

Sarah Pastorelli, Sandro Valzacchi, Ana Rodriguez, Catherine Simoneau

► To cite this version:

Sarah Pastorelli, Sandro Valzacchi, Ana Rodriguez, Catherine Simoneau. SPME analytical method for the determination of Hexanal in hazelnuts as indicator of the interaction of active packaging materials with food aroma compounds. *Food Additives and Contaminants*, 2006, 23 (11), pp.1236-1241. 10.1080/02652030600778744 . hal-00577484

HAL Id: hal-00577484

<https://hal.science/hal-00577484>

Submitted on 17 Mar 2011

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



SPME analytical method for the determination of Hexanal in hazelnuts as indicator of the interaction of active packaging materials with food aroma compounds

Journal:	<i>Food Additives and Contaminants</i>
Manuscript ID:	TFAC-2006-052.R1
Manuscript Type:	Original Research Paper
Date Submitted by the Author:	06-Apr-2006
Complete List of Authors:	Pastorelli, Sarah; Joint Research Centre, IHCP, PCE Valzacchi, Sandro; Joint Research Centre, IHCP, PCE Rodriguez, Ana; Joint Research Centre, IHCP, PCE Simoneau, Catherine; Joint Research Centre, IHCP, PCE
Methods/Techniques:	Chromatography - GC, Chromatography - GC/MS, SPME
Additives/Contaminants:	Packaging active packaging, Volatiles
Food Types:	Dried fruit

SCHOLARONE™
Manuscripts

1
2
3
4 **Solid-phase Microextraction (SPME) analytical method for the**
5 **determination of hexanal in hazelnuts as an indicator of the interaction**
6 **of active packaging materials with food aroma compounds**
7
8
9

10
11
12
13 S. Pastorelli*, S. Valzacchi, A. Rodriguez and C.Simoneau
14

15
16 European Commission DG-Joint Research Centre, Institute for Health and Consumer
17 Protection, Physical and Chemical Exposure Unit, TP 260, 21020 Ispra (VA) – Italy
18
19

20
21
22
23
24
25 *Corresponding author: Sarah Pastorelli
26

27 E-mail: sarah.pastorelli@jrc.it
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

Abstract

Fatty foods are susceptible to lipid oxidation, which results in a deterioration of the quality of the product, since this process is responsible of generation of off-flavours. Hexanal has been reported to be a good indicator of the rancidity. A method based on solid-phase microextraction (SPME) coupled with gas chromatograph with flame ionization detection to determine hexanal formed in hazelnuts during storage was developed. The optimum conditions were the follow: carboxen-polydimethylsiloxane 75µm fibre, extraction time 10 min, equilibrium time 10 min and equilibrium temperature 60°C. A study to evaluate the effect of the oxygen scavengers on the oxidation process was also conducted by measuring the hexanal formed in hazelnuts stored with and without the oxygen absorbers sachets. Oxygen scavengers shown to reduce the oxidation, however the analysis of the sachet revealed that also other volatile compounds from the headspace were absorbed.

Keywords: SPME, hexanal, lipid oxidation, oxygen scavengers

Introduction

Food products with high fat content, such as hazelnuts and nuts in general, are susceptible to rancidity because of lipid oxidation, which involves the development of off-flavours that make the product undesirable for the consumers. As it is well known, the oxidation process is accelerated by different factors such as light and temperature. In order to delay the oxidation and to extend the product shelf-life, several approaches have been developed by the food industry. Special packaging techniques, such as modified atmosphere, oxygen-barrier packaging material and active packaging (oxygen scavengers) (Lee and Krochta 2002) as well as the addition of natural or synthetic antioxidants have been recently investigated (Stashenko et al. 2002).

Hexanal was found to be a good marker of the oxidative rancidity since it is one of the most abundant products of the oxidation of fats and its content increases during storage (Stashenko et al. 2002; Lee and Krochta 2002; Goodridge et al. 2003; Brunton et al. 2000; Shahidi 2001; Jensen et al. 2005). It is the main aldehyde formed during the oxidation of linoleic, gamma-linolenic and arachidonic acids (Shahidi 2001).

Different techniques for volatile isolation and pre-concentration, such as dynamic headspace, purge and trap (Cadwallader and Xu 1994), and static headspace (Romeu-Nadal et al. 2004) have been described in the literature. Although they are simple and fast, these methods present certain drawbacks. The first two of these, are not easy to automate and are time-consuming and on static headspace techniques can lack of sensitivity (Goodridge et al. 2003; Snow 2002). To overcome these disadvantages

1
2
3
4 during the last years solid phase micro extraction (SPME) has been proven to be an
5
6 alternative, promising and innovative technique that combines the simultaneous
7
8 extraction and pre-concentration steps. It is simple, fast, uses little solvent, does not
9
10 require sample preparation and is inexpensive.
11
12

13
14
15
16 SPME has been successfully applied in food analysis to evaluate the volatile profiles in
17
18 different foodstuffs: fruits (Augusto et al. 2000; De Lourdes-Cardéal et al. 2005), meat
19
20 (Ramirez et al. 2004), vinegar (Natera-Marin et al. 2002), olive oil (Garcia-Gonzalez et
21
22 al. 2004), soybean (Boué et al. 2003), truffle (Diaz et al. 2002) and also to analyse
23
24 hexanal in several matrices: turkey (Brunton et al. 2000), powder infant milk formula
25
26 (Fenaille et al. 2003) and freeze-dried chicken (Goodridge et al. 2003).
27
28
29
30
31
32

33 The purposes of the present paper were to develop a simple and fast SPME-GC-FID
34
35 method to determine hexanal and to evaluate the effect of the oxygen scavengers on the
36
37 oxidation of hazelnuts samples stored under controlled conditions.
38
39
40
41

42 **Materials and methods**

43 *Sampling*

44
45 Powdered hazelnuts packed with a transparent film were purchased from a local
46
47 supermarket. A gas chromatography-flame ionization detector (GC-FID) analysis was
48
49 performed immediately after being acquired. Then they were stored under controlled
50
51 conditions of light and temperature in 20 mL hermetically closed flasks with and
52
53 without oxygen absorbers sachets added to the packaging until analysis.
54
55
56
57
58
59
60

Oxygen-absorbing sachets based on iron powder and with capacities of 20 mL were used in the experiments.

Reagents and Analytical standards

Hexanal was purchased from Sigma-Aldrich (Steinheim, Germany) with purity of 98%. Two stock standard solutions of 500 mg/L of hexanal were prepared by using Milli-Q water and triacetin as solvents. Working solutions employed were prepared by diluting different amounts of the stock standard solution in Milli-Q water and triacetin respectively. Both solutions were stored at 4°C. Ultra pure water was obtained using a Milli-Q filter system (Millipore, Bedford, MA, USA) and triacetin was supplied from Sigma-Aldrich (Steinheim, Germany).

SPME procedure

The SPME holder for manual sampling and the commercial fibres tested in this study were purchased from SUPELCO (Bellefonte, PA, USA). Five fibres with different coating materials were examined: PDMS with 100 µm thickness; CAR/PDMS with 75 µm thickness, PDMS/DVB with 65 µm thickness, CAR/DVB with 65 µm thickness and DVB/PDMS/CAR with 50-30 µm thickness. Prior to their use they were conditioned by inserting them into the GC injector according the supplier's instructions summarized in Table I.

One mL of the standard solution or 0.1 g in the case of the hazelnuts samples were placed in a 20 mL vial that was hermetically sealed with a PTFE-coated silicone septum and analysed by SPME-GC. A small magnetic stirring bar was added to the vial before it was capped. Then the vials were placed in an aluminium thermostated block at 60°C

1
2
3
4 for 10 min to reach the equilibrium with the headspace. After equilibrium time the
5
6 SPME fibre was exposed to the headspace of the sample at 60°C for 10 min (extraction
7
8 time). Then the fibres were directly inserted into the injector port of the GC for thermal
9
10 desorption at the temperature reported in table I, during 15 min.
11
12
13

14
15
16 To ensure that all impurities were removed blank runs were conducted between sample
17
18 analysis. Each sample extraction was carried out in triplicate.
19
20
21

22 23 *Gas chromatography*

24
25 Analyses were performed on a 5890 Hewlett-Packard gas chromatograph equipped with
26
27 a flame ionization detector (FID). The GC system was operated using splitless injection
28
29 mode. A 0.75 mm i.d. narrow-bore glass liner (Supelco, Bellefonte, USA) was used to
30
31 prevent peak broadening (Vas and Vékey 2004) and consequently to improve the
32
33 resolution of the analysis. A DB-wax fused capillary column (30 m 0.25 mm ID, 0.5
34
35 µm) was used for hexanal analysis. Operating conditions were as follows: injector
36
37 temperature was set between 240-300°C depending on the fibre used and detector
38
39 temperature was kept at 350°C. The ramp temperature was initially set at 50°C, then
40
41 raised at the rate of 10°C/min to 100°C, then increased at 20°C/min until 200°C and held
42
43 for 7min. The carrier gas was helium with a flow rate of 1.2 mL/min.
44
45
46
47
48
49
50

51
52 A Hewlett Packard 6890 gas chromatograph equipped with a Hewlett Packard 5973
53
54 mass detector working in electronic impact ionization mode in full scan acquisition
55
56 mode was used for hexanal identification. The chromatographic conditions were the
57
58 same as those specified for the GC-FID system. Hexanal was identified by the library
59
60

1
2
3
4 search software of the instrument and confirmed by the correspondence of the retention
5
6 time obtained with the analysis of pure standard.
7
8
9

10
11 The software HP-Chem Station from Hewlett-Packard was used for data processing.
12

13
14 “[Insert Table I about here]”
15
16
17

18 **Results and Discussion**

19 *SPME-GC method Optimisation*

20
21 In developing gas chromatographic method several temperature ramps were studied, the
22
23 selected ramp was that provided the best resolution in a shorter time. Triacetin and
24
25 Milli-Q water were evaluated as solvents for standard solutions. Hexanal showed
26
27 considerable instability in water, however with triacetin good results in terms of
28
29 reproducibility were obtained, and for this reason triacetin was chosen as solvent.
30
31 Furthermore, as triacetin has a higher boiling point than hexanal, a good
32
33 chromatographic separation without interferences was achieved.
34
35
36
37
38
39
40
41

42 The SPME sampling procedure depends on several variables such as equilibrium and
43
44 extraction time and temperature, the amount of the sample, and the kind of the fibre
45
46 coating. In order to optimise all these parameters, several experiments were conducted.
47
48 Initially, a hexanal standard solution was used to fix time and temperature conditions,
49
50 then a series of assays with hazelnuts samples were conducted taking into account the
51
52 effect of the matrix. A PDMS 100 µm fibre was used to carry out the preliminary trials
53
54 since it is the most often used due to its physicochemical properties, it is also very
55
56 rugged and recommended for the extraction of volatile flavour compounds (Vas and
57
58
59
60

1
2
3
4 Vékey 2004; Kataoka et al. 2000). In a first series of assays the parameter studied was
5 the equilibrium temperature. Different values were tested (50, 60, 70 and 80°C) keeping
6 equilibrium time (10 min) and extraction time (10 min) fixed. Under these conditions
7 the best results were found at 60 and 70°C, so that we selected the lower temperature.
8
9 Once the temperature was optimised the equilibrium time was studied ranging from 5 to
10 60 min; it was found that 10 min was enough to reach the equilibrium, therefore an
11 equilibration time of 10 min was selected for successive analyses. Three different
12 extraction times, were also evaluated: 5, 10 and 20 min. Best results were achieved at 10
13 min. These results are in accordance with those reported by Ezquerro et al. 2003, where
14 it is indicated that the extraction time depends on the compound, in general, for small
15 compounds such as the hexanal after 10-20 min of exposure of the fibre to the
16 headspace the signal (peak area) decreased.
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34

35 In order to establish the optimal sample amount 0.1 g, 1 g, 10 g and 15 g of ground
36 hazelnuts were assayed, and 0.1 g was considered a suitable sample amount to produce
37 quantitative results. Finally, the effect of the dilution of the sample with Milli-Q water
38 (1mL) and the addition of a saturated salt solution of NaCl were also evaluated. As it
39 has been reported in the literature (Pino et al. 2002) the addition of the salt to the sample
40 could modify the nature of the matrix and the extraction efficiency since this addition
41 can affect the partition coefficient of the analytes, however in terms of improvement of
42 analyte extraction from the sample no significant differences were observed so
43 subsequent analyses were carried out with dry sample.
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

1
2
3
4 A small magnet bar was also added into the vial in order to decrease the time necessary
5 for the equilibrium and to facilitate the transfer of the analyte from the matrix to the
6 fibre coating material (Vas and Vékey 2004; Pillonel et al. 2002). Five commercial
7 available fibres (PDMS, CAR/PDMS, PDMS/DVB, CAR/DVB and DVD/CAR/PDMS)
8 were compared. The CAR/PDMS fibre had the highest sensitivity. This result is in
9 agreement with those reported by Augusto et al. 2000 and Brunton et al. 2000. Natera-
10 Marin et al. 2002 pointed out that the high efficiency of this coating could be attributed
11 to the high porosity that provides a strong adsorption. Furthermore, Ezquerro et al.,
12 (2002) reported that for low molecular mass compounds the CAR/PDMS provides good
13 results and Diaz-Maroto et al., (2004) obtained high recoveries when using a fibre
14 containing a porous coating. According to the method proposed by Augusto et al. 2000,
15 the inter fibres comparison (figure 1) was made calculating the normalized extraction
16 efficiency ($N_{i,x}$) defined as $N = 100 * A_{i,x} / A_{i,CAR/PDMS}$ where $A_{i,x}$ is the hexanal area
17 analyzed with the fibre x and $A_{i,CAR/PDMS}$ is the corresponding area with the most
18 efficient fibre (CAR/PDMS).
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39

40 The experiments were performed in triplicate.

41
42 “[Insert Figure 1 about here]”
43
44
45
46

47 *Performance characteristics*

48 A calibration curve was carried out using a series of six hexanal standards of known
49 concentration. Parameters of linearity were the follow: -388.05 (intercept); 974216
50 (slope); 0.999(r^2); and 0,25-2,5 ng/mL (range). Another calibration line was constructed
51 by spiking hazelnuts samples. 10 μ l of a triacetin solution with different hexanal
52 quantities were added to the samples. The equation was $Y = 506322x - 120.94$, the
53
54
55
56
57
58
59
60

1
2
3
4 determination coefficient and the linearity were 0.997 and 0,25-2,5 ng/mL respectively.
5
6
7 Detection limit calculate in accordance with the Subcommittee on environmental
8
9 analytical chemistry (ACS) guidelines (defined as a signal three times the height of the
10
11 noise level) was 0.08 ng/mL. The value obtained is lower than the LOD obtained by
12
13 Romeu-Nadal et al. 2004 in infant formulas.
14

15
16
17
18 Repeatability was estimated as relative standard deviation (RSD) for the determination
19
20 of six extracts, from the same oxidized sample with an hexanal content of 0.25 ppb,
21
22 each prepared separately, was 5.32%.
23
24

25
26
27
28 *Evaluation of oxygen scavengers in the oxidation process.*
29

30
31 In the first tests to evaluate the effect of the oxygen scavenger on the oxidation process
32
33 the headspace of the vial containing the hazelnuts with and without the oxygen absorber
34
35 sachets was analysed. The O₂ content was measured using a Chek Mate 9900 Dansensor
36
37 (Milano, Italy) and it was found that in the flasks stored with oxygen scavenger
38
39 practically all oxygen was removed while in the flasks without oxygen scavenger the
40
41 content of oxygen was 18%.
42
43

44
45
46
47 A lower content in hexanal in the samples containing the absorber sachet were
48
49 observed. The samples containing oxygen scavenger presented, after three days, a
50
51 12.25% less of hexanal than the samples stored without the oxygen absorber. The test
52
53 results are summarized in Table II.
54

55
56
57 “[Insert Table II about here]”
58
59
60

1
2
3
4 In a second series of trials the content of the sachets were also analysed by SPME/GC-
5
6 FID. The content of the sachets were place into 20 mL headspace vials and they were
7
8 hermetically capped with a silicone septum. The SPME and chromatographic conditions
9
10 were the same as those specified for the hazelnuts samples analysis. Different
11
12 components were found and among these hexanal was also detected. For that reason
13
14 other assays were conducted to investigate the possible absorption of volatile
15
16 compounds by the oxygen scavenger.
17
18
19

20
21
22
23 Two vials (120mL) with 10mL of a hexanal solution in water (500 mg kg⁻¹) were
24
25 prepared. In one of the vials, oxygen scavenger was suspended on the top of the
26
27 container (not to be in contact with the liquid) and both vials were hermetically closed
28
29 and stored at room temperature. After one week an SPME/GC-FID analysis of the
30
31 headspace of the two vials were made.
32
33
34

35
36
37 As shown the figure 2, the hexanal content in the headspace of vials with the oxygen
38
39 scavenger was considerably reduced comparing with the one without oxygen scavenger.
40
41 The oxygen scavenger sachet was opened and the powder contained in the sachet was
42
43 analysed. The quantity of hexanal found in the powder cannot explain the differences
44
45 found in the 120mL headspace vials and this could be due to the fact that probably it's
46
47 difficult to extract again hexanal from the powder after the adsorption.
48
49
50

51
52
53 Briefly, although the oxygen scavengers shown to reduce the oxidation process, other
54
55 volatile components among these hexanal were also absorbed. Since the hexanal is an
56
57 indicator of the rancidity, this fact could mask the real state of the food product and
58
59
60

1
2
3
4 could mislead consumers. Further research is required in order to evaluate the
5
6 interactions between food and oxygen scavenger.
7
8
9

10
11
12 “[Insert Figure 2 about here]”
13

14 **References**

- 15
16 Augusto F, Pires-Valente AL, dos Santos-Tada E, Rivellino SR. 2000. Screening of
17
18 Brazilian fruit aromas using solid-phase microextraction gas chromatography-mass
19
20 spectrometry. *Journal of Chromatography A* 873:117-127.
21
22
23
24 Boué SM, Shih BY, Carter-Wientjes CH, Cleveland TE. 2003. Identification of volatile
25
26 compounds in soybean at various developmental stages using solid phase
27
28 microextraction. *Journal of Agriculture and Food Chemistry* 51:4873-4876.
29
30
31 Brunton NP, Cronin DA, Monahan FJ, Durcan R. 2000. A comparison of solid-phase
32
33 microextraction (SPME) fibres for measurement of hexanal and pentanal in cooked
34
35 turkey. *Food Chemistry* 68:339-345.
36
37
38 Cadwallader KR, Xu Y. 1994. Analysis of volatile components in fresh grapefruit juice
39
40 by purge and trap/gas chromatography. *Journal of Agriculture and Food Chemistry*
41
42 42:782-784.
43
44
45 De Lourdes-Cardel Z, Moreira-Guimaraes E, Vilela-Parreira F. 2005. Analysis of
46
47 volatile compounds in some typical Brazilian fruits and juices by SPME-GC
48
49 method. *Food Additives and Contaminants* 22:508-513.
50
51
52 Diaz P, Señorans FJ, Reglero G, Ibañez E. 2002. Truffle aroma analysis by headspace
53
54 solid phase microextraction. *Journal of Agriculture and Food Chemistry* 50: 6468-
55
56 6472.
57
58
59 Diaz-Maroto M, Sanchez-Palomo E, Perez-Coello MS. 2004. Fast screening method for
60

1
2
3
4 volatile compounds of oak wood used for aging wines by headspace SPME-GC-MS
5 (SIM). *Journal of Agriculture and Food Chemistry* 52:6857-6861.
6
7

8
9 Ezquerro O, Pons B, Tena MT. 2002. Development of a headspace solid-phase
10 microextraction-gas chromatography-mass spectrometry method for the
11 identification of odour-causing volatile compounds in packaging materials. *Journal*
12 *of Chromatography A* 963:381-392.
13
14

15
16 Ezquerro O, Pons B, Tena MT. 2003. Direct quantitation of volatile organic compounds
17 in packaging materials by headspace solid-phase microextraction-gas
18 chromatography-mass spectrometry. *Journal of Chromatography A* 985:247-257.
19
20

21
22 Fenaille F, Visani P, Fumeaux R, Milo C, Guy PA. 2003. Comparison of mass
23 spectrometry-based electronic nose and solid phase microextraction gas
24 chromatography-mass spectrometry technique to asses infant formula oxidation.
25 *Journal of Agriculture and Food Chemistry* 51:2790-2796.
26
27

28
29 Garcia-Gonzalez DL, Barie N, Rapp M, Aparicio R. 2004. Analysis of virgin olive oil
30 volatiles by a novel electronic nose based on miniaturized SAW sensor array
31 coupled with SPME enhanced headspace enrichment. *Journal of Agriculture and*
32 *Food Chemistry* 52:7475-7479.
33
34

35
36 Goodridge CF, Beaudry RM, Pestka JJ, Smith DM. 2003. Solid phase microextraction-
37 gas chromatography for quantifying headspace hexanal above freeze-dried chicken
38 myofibrils. *Journal of Agriculture and Food Chemistry* 51:4185-4190.
39
40

41
42 Jensen PN, Danielsen B, Bertelsen G, Skibsted LH, Andersen ML. 2005. Storage
43 stabilities of pork scratchings, peanuts, oatmeal and muesli: comparison of ESR
44 spectroscopy, headspace-GC and sensory evaluation for detection of oxidation in
45 dry foods. *Food Chemistry* 91:25-38.
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

- 1
2
3
4 Kataoka H, Lord HL, Pawliszyn J. 2000. Applications of solid-phase microextraction in
5
6 food analysis. *Journal of Chromatography A* 880:35-62.
7
8
9 Lee SY, Krochta JM. 2002. Accelerated shelf life testing of whey-protein-coated
10
11 peanuts analyzed by static headspace gas chromatography. *Journal of Agriculture*
12
13 and Food Chemistry 50:2022-2028.
14
15
16 Natera-Marin R, Castro-Mejias R, Garcia-Moreno MV, Garcia-Rowe F, Garcia-Barroso
17
18 C. 2002. Headspace solid-phase microextraction analysis of aroma compounds in
19
20 vinegar validation study. *Journal of Chromatography A* 967: 261-267.
21
22
23 Pillonel L, Bosset JO, Tabacchi R. 2002. Rapid preconcentration and enrichment
24
25 techniques for the analysis of food volatile. A Review. *Lebensmittel Wissenschaft*
26
27 und Technology 35:1-14.
28
29
30 Ramirez MR, Estevez M, Morcuende D, Cava R. 2004. Effect of the type of frying
31
32 culinary fat on volatile compounds isolated in fried pork loin chops by using SPME-
33
34 GC-MS. *Journal of Agriculture and Food Chemistry* 52:7637-7643.
35
36
37 Romeu-Nadal M, Castellote AI, Lopez-Sabater MC. 2004. Headspace gas
38
39 chromatographic method for determining volatile compounds in infant formulas.
40
41 *Journal of Chromatography A* 1046:235-239.
42
43
44 Shahidi F. 2001. Headspace volatile aldehydes as indicators of lipid oxidation in foods.
45
46 *Advances in Experimental Medicine and Biology* 488:113-123.
47
48
49 Snow NH. 2002. Head-space analysis in modern gas chromatography. *Trends*
50
51 in analytical chemistry 21:605617.
52
53
54 Stashenko EE, Puertas MA, Martinez JR. 2002. SPME determination of volatile
55
56 aldehydes for evaluation of in-vitro antioxidant activity. *Analytical and*
57
58 *Bioanalytical Chemistry* 373:70-74.
59
60

1
2
3
4 Subcommittee on environmental analytical chemistry (ACS). 1980. Guidelines for data
5 acquisition and data quality evaluation in environmental chemistry. *Analytical*
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

Chemistry 52:2242-2249.

Vas G, Vékey K. 2004. Solid-phase microextraction: a powerful sample preparation
tool prior to mass spectrometric analysis. *Journal of Mass Spectrometry* 39:233-
254.

Table I. Conditioning recommendations for different fibres

Fibre	Temperature (°C)	Time (h)
PDMS 100 µm	250°C	0.5
CAR/PDMS 75 µm	300°C	1-2
PDMS/DVB 65 µm	250°C	0.5
CAR/DVB 65 µm	220°C	0.5
DVB/PDMS/CAR 50-30 µm	270°C	2-4

Table II Evaluating oxygen scavengers: test results.

Parameters	Samples stored with oxygen scavenger	Samples stored without oxygen scavenger
O ₂ (%)	0.001	18.0
Hexanal content in HS vial (ng/mL)*	0.55	0.63

*Hexanal content analysed after 3 days of storage

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

Figure legends

Figure 1. - Normalized extraction efficiencies N_x (x = SPME fibre) measured for extraction with different fibres

Figure 2. –Chromatogram of hexanal content in headspace vial with and without oxygen scavenger

For Peer Review Only

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

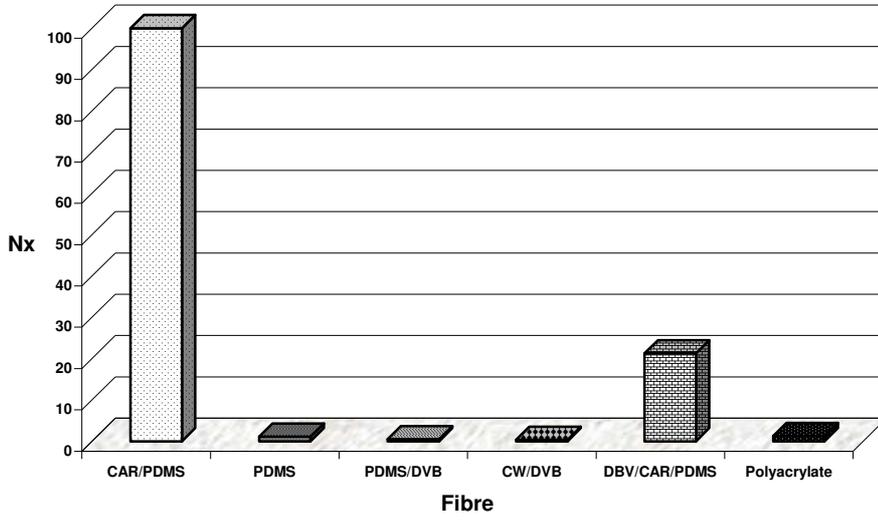


Figure 1

er Review Only

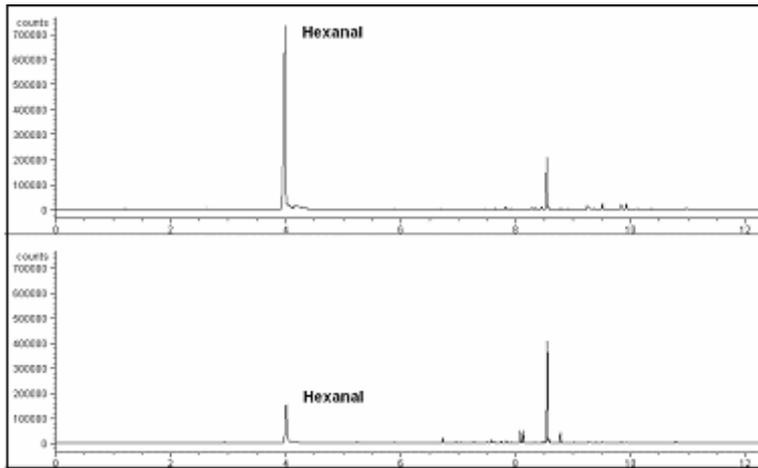


Figure 2