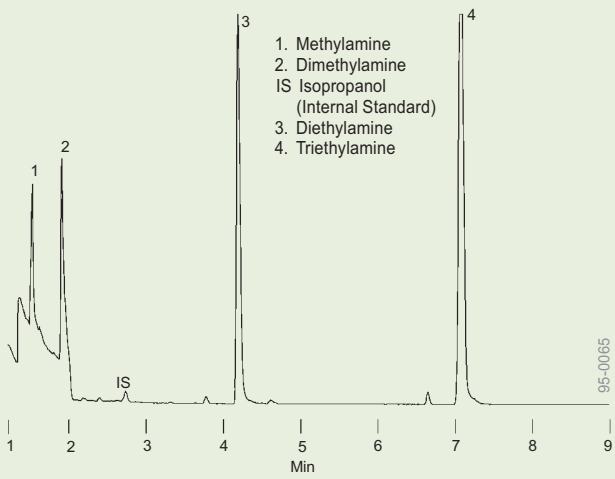
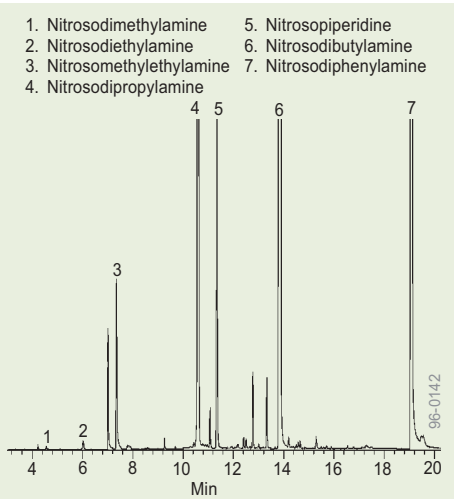
SPME Applications

Amines, Aromatics, Explosives



Amines (SPME/GC)

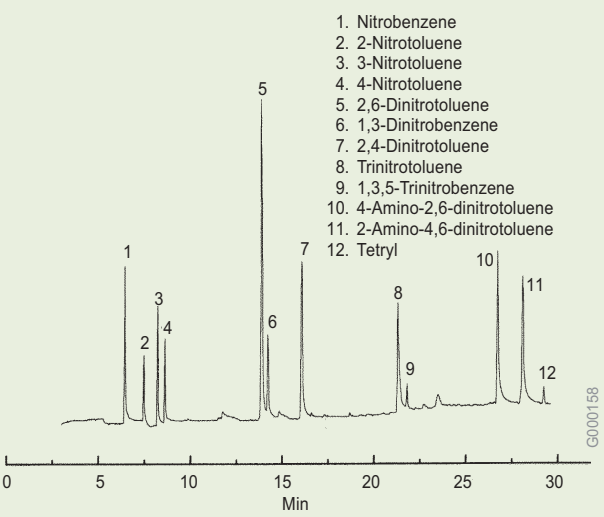
Sample:	amines at 5ppm in water, 27% NaCl, pH 9.5
SPME Fiber:	polydimethylsiloxane/divinylbenzene, 65µm film
Cat. No.:	57310
Extraction:	20 min, fiber immersed in water, rapid stirring
Desorption:	5 min, 270°C
Column:	SPB-1 SULFUR, 30m x 0.32mm ID, 4.0µm film
Cat. No.	24158
Oven:	50°C (2min) to 150°C at 10°C/min
Carrier:	helium, 25cm/sec (set at 50°C)
Det.:	GC/MS, selected ions at 0.6 sec/scan
Inj.:	splitless/split, closed initial 5 min, 270°C, 0.75mm ID splitless liner



Chromatogram courtesy of J. Clark, Liggett Group, Inc.

Nitrosamines (SPME/GC)

Sample:	analytes in water + 25% KCl, pH 10.0
SPME Fiber:	polydimethylsiloxane/divinylbenzene, 65µm film
Cat. No.:	57310-U
Extraction:	immersion, 15min (rapid stirring)
Desorption:	270°C, 1min
Column:	PTA-5, 30m x 0.32mm ID, 0.5µm film
Cat. No.:	24331
Oven:	50°C (1min) to 250°C at 10°C/min, hold 2 min
Carrier:	helium, 30cm/sec
Det.:	GC/MS (quadrupole, selected ion mode)
Inj.:	splitless, 250°C (0.75mm ID liner)



Reproduced from The Analysis of High Explosives Residues by Solid-Phase Microextraction Followed by HPLC, GC/ECD and GC/MS, José R. Almirall, Metro-Dade Police Crime Laboratory, Miami, FL, US. Presented at Academy of Forensic Science 2/13/98.

Explosives in Water (SPME/GC)

Sample:	50ng/mL each explosive in water
SPME Fiber:	polydimethylsiloxane/divinylbenzene, 65µm film
Cat. No.:	57310-U
Sampling:	immersion, 30 min
Desorption:	5 min, 250°C
GC Column:	cyanopropyl silicone, 30m x 0.25mm ID, 0.25µm film
Supelco Equivalent:	SPB-1701 (Cat. No. 24113)
Oven:	95°C (3 min) to 182°C at 8°C/min (4 min) to 250°C at 8°C/min (6 min)
Carrier:	nitrogen, 60mL/min
Det.:	ECD, 250°C
Inj.:	split/splitless, 180°C

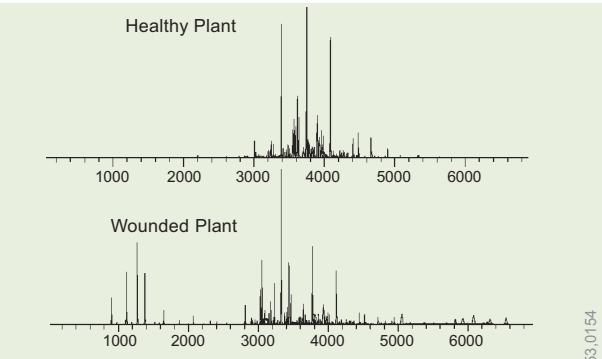


Figure provided by A.-K. Borg-Karlson, TheRoyal Institute of Technology, Department of Chemistry, Stockholm, Sweden.

Mono- and Sesquiterpenes in Spruce Seedlings (SPME/GC)

Sample:	headspace surrounding Picea abies seedlings
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	headspace, 60 min
Desorption:	30 sec, 225°C
Column:	poly(5% diphenyl/95% dimethylsiloxane), 30m x 0.25mm ID, 0.25µm film
Supelco Equivalent:	Equity-5 (Cat. No. 28089-U)
Oven:	40°C (4 min) to 200°C at 4°C/min, hold 16 min
Carrier:	helium, 10psi
Det.:	EID, 70ev
Inj.:	splitless (splitter opened after 30 sec), 225°C

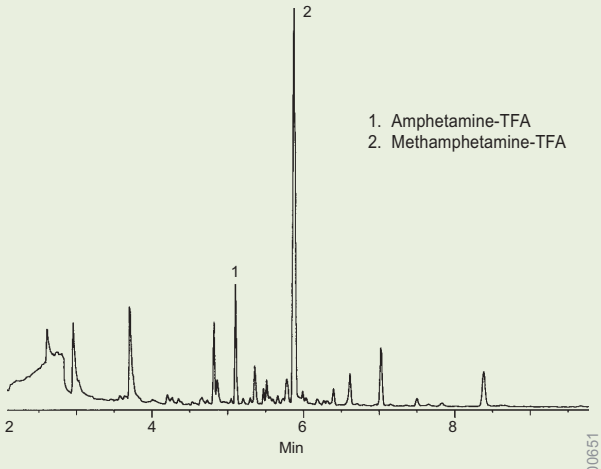


Figure provided by Thomas Brettell, New Jersey State Police Laboratory.

Amphetamines (SPME/GC)

Sample:	1mL urine + 0.7g K_2CO_3 in 20mL headspace vial, equilibrated at 80°C, 30 min
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	headspace, 3-5 min, 80°C
Derivatization:	methyl bis-trifluoroacetamide (headspace, 0.5 min, ambient)
Desorption:	1 min, 270°C
Column:	methylsiloxane, 12.5m x 0.2mm ID, .033µm film
Supelco Equivalent:	Equity-1 (available on request)
Oven:	60°C (1 min) to 140°C (4 min) at 30°C/min, then to 276°C at 20°c/min, 4 min
Det.:	MS, full scan
Inj.:	splitless (closed 1 min), 270°C

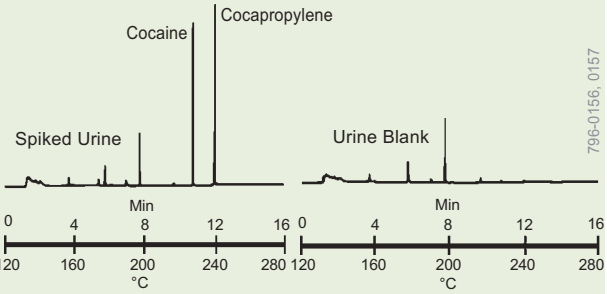


Figure provided by T. Kumazawa and K. Sato, Dept. Legal Medicine, Showa University School of Medicine, Tokyo, Japan and K. Watanabe, H. Seno, A. Ishii, and O. Suzuki, Dept. Legal Medicine, Hamamatsu University School of Medicine, Hamamatsu, Japan.

Used with permission of Japanese Journal of Forensic Toxicology.

Cocaine in Urine (SPME/GC)

Sample:	0.5mL urine (250ng each analyte, 20µL 2.5% NaF) in 1mL vial
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	immersion, 30 min
Desorption:	3 min, 240°C
Column:	polydimethylsiloxane, 30m x 0.32mm ID, 0.25µm film
Supelco Equivalent:	Equity-1 (Cat. No. 28055-U)
Oven:	120°C to 280°C at 10°C/min
Carrier:	helium, 3mL/min
Det.:	NPD, 280°C
Inj.:	splitless (splitter opened after 1 min), 240°C

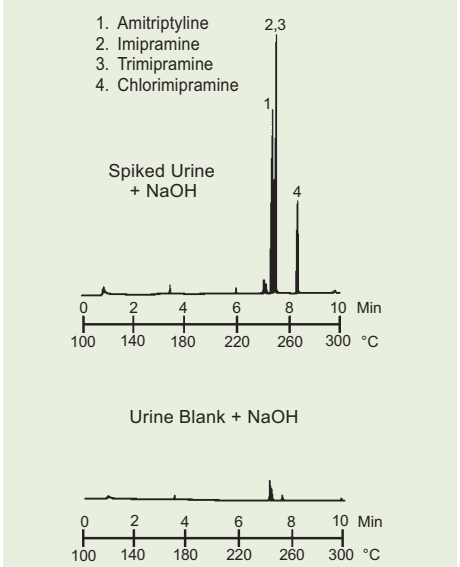


Figure provided by T. Kumazawa, X.-P. Lee, M.-C. Tsai, and K. Sato, Dept. Legal Medicine, Showa University School of Medicine, Tokyo, Japan and H. Seno and A. Ishii, Dept. Legal Medicine, Hamamatsu University School of Medicine, Hamamatsu, Japan.

Used with permission of Japanese Journal of Forensic Toxicology.

Tricyclic Antidepressants in Urine (SPME/GC)

Sample:	1mL urine (1µg each analyte + 50µL 5M NaOH) in 7.5mL vial
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	headspace, 15 min, 100°C (sample incubated 30 min)
Desorption:	3 min, 280°C
Column:	polydimethylsiloxane, 30m x 0.32mm ID, 0.25µm film
Supelco Equivalent:	Equity-1 (Cat. No. 28055-U)
Oven:	100°C to 300°C at 20°C/min
Carrier:	helium, 3cm/sec
Det.:	FID, 280°C
Inj.:	splitless (splitter opened after 1 min), 280°C

SPME Applications
Foods, Flavors and Fragrances

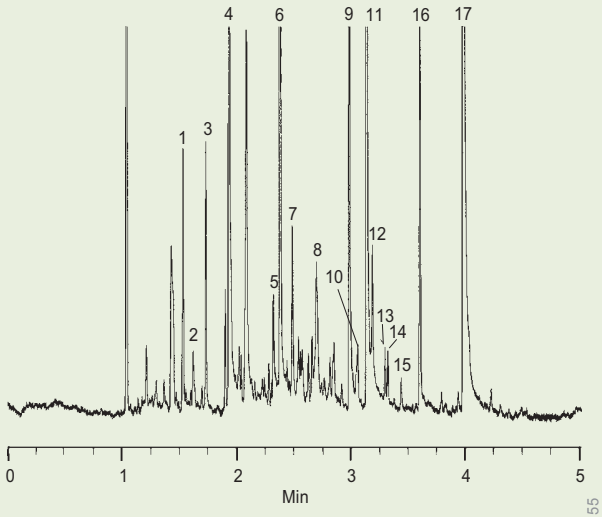
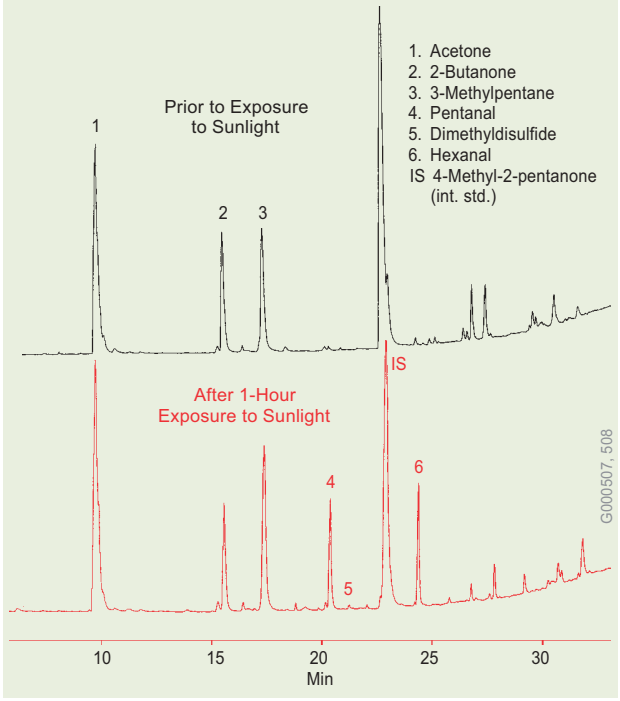


Figure provided by Jun Song, Department of Horticulture, Michigan State University, East Lansing, MI 48824 USA.

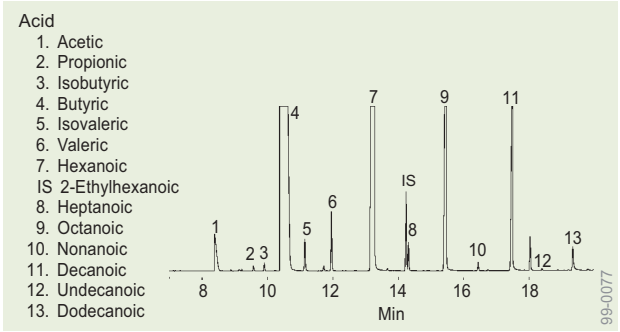
Aroma Volatiles from Stored Apples (SPME/GC)

Sample:	300-450g intact fruit in a 3 liter flask at ambient temp., exposed to an air flow of 25-30mL/min.
SPME Fiber:	polydimethylsiloxane/divinylbenzene, 65µm film headspace extraction (as described for Sample), 4 min; analytes desorbed (250°C) to a cool (<100°C) precolumn, then volatilized by heating to 150°C in 15 sec.
Cat. No.:	57310-U
GC Column:	poly(5% diphenyl/95% dimethylsiloxane) type, 25m x 0.25mm ID, 0.34µm film
Supelco Equivalent:	Equity-5 (available on request)
Oven:	40°C (1.5 min) to 250°C at 50°C/min, hold 1 min
Carrier:	helium, 1.5mL/min (44cm/min)
Det.:	mass spectrometer, m/z = 40-300 (40 spectra/sec)



Milk Sample Off-Flavors (SPME/GC)

Sample:	3g of 2% milk + 10µL internal standard solution (20µg/mL 4-methyl-2-pentanone) (9mL GC vial)
SPME Fiber:	PDMS/Carboxen, 75µm film
Cat. No.:	57318
Extraction:	headspace, 15 min with constant stirring at 45°C
Desorption:	5 min, 250°C
Column:	Supel-Q PLOT, 30m x 0.32mm ID
Cat. No.:	24242
Oven:	70°C (2 min) to 140°C at 6°C/min (2 min hold) then to 220°C at 6°C/min (5 min hold)
Carrier:	helium, 35cm/sec
Det.:	GC/MS ion trap, m/z = 33-300



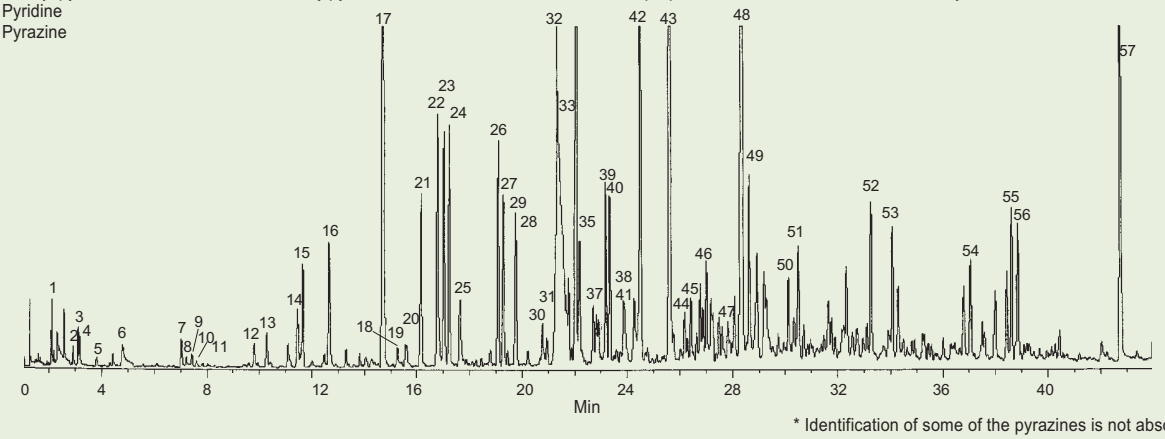
Free Fatty Acids in Parmesan Cheese (SPME/GC)

Sample:	100mg cheese in 40mL vial
SPME Fiber:	65µm Carbowax/divinylbenzene StableFlex
Cat. No.:	57336-U
Sampling:	65°C 15 min (headspace)
Desorption:	250°C, 1 min
GC Column:	Nukol, 15m x 0.25mm, 0.25µm film
Cat. No.:	24106-U
Oven:	50°C (2 min) to 220°C at 10°C/min
Carrier:	helium, 30cm/sec
Det.:	FID, 260°C
Inj.:	splitless/split (closed 1 min), 250°C

SPME Applications
Foods, Flavors and Fragrances

Coffee Components*

1. 2-Methyl furan
2. 2-Butanon
3. 2-Pentanone
4. 3-Methyl butanal
5. 2,5-Dimethylfuran
6. 2-Acetyloxy-2-propanone
7. 2-Ethyl hexanol
8. Dimethylsulfoxide
9. Phenol
10. Hexanal
11. 2-Methyl thiophene
12. n-Methyl pyrrole
13. 4-Methylphenol
14. 2-Ethyl pyrrole
15. Pyridine
16. Pyrazine
17. Methyl pyrazine
18. 4-Methyl thiazole
19. 3-Hydroxy butanone
20. Dimethyl phenol (isomer)
21. 1,2-Ethanediol monoacetate
22. 2,5-Dimethylpyrazine
23. 2,3-Dimethylpyrazine
24. 2-Ethylpyrazine
25. 2,6-Dimethylpyrazine
26. 2-Ethyl-6-methylpyrazine
27. 2-Ethyl-5-methylpyrazine
28. Trimethylpyrazine
29. 2-Ethyl-3-methylpyrazine
30. 2,6-Diethylpyrazine
31. 2-Ethenylpyrazine
32. 2-Ethyl-3,5-dimethylpyrazine
33. Glycerol
34. 2,3-Diethylpyrazine
35. 2-Ethyl-3,6-dimethylpyrazine
36. 2-Furancarboxaldehyde
37. 2-Isopropenylpyrazine
38. 3,5-Diethyl-2-methylpyrazine
39. Furfural formate
40. 2-Furonyl ethanone
41. Methyl benzoylformate
42. Furanmethanol acetate
43. 5-Methyl-2-furancarboxaldehyde
44. Furanmethanol propionate
45. Furfanyl furan
46. Pyridine methanol
47. 2-Methyl-5-propenylpyrazine
48. Furanmethanol
49. 3-Ethyl-4-methyl-2,5-furandione
50. Pyrazine carboxamide
51. 2-Ethyl-3-hydroxy-4H-pyran-4-one
52. 1-(2-Furanylmethyl)pyrrole
53. 2-Methoxyphenol
54. 1-(1H-Pyrrole-2-yl)ethanone
55. 4-Ethyl-2-methoxy phenol
56. 3-Phenylpropenal or 2-Methylbenzofuran
57. 3,5-Dimethylbenzoic acid



* Identification of some of the pyrazines is not absolute.

Ground Coffee (SPME/GC)

Sample: 5g ground coffee in 40mL vial
SPME Fiber: 50/30µm DVB/Carboxen/PDMS (StableFlex fiber)
Cat. No.: 57348-U
Extraction: 30 min, 65°C (headspace)
Desorption: 270°C, 5 min

GC Column: SUPELCOWAX 10, 30m x 0.25mm ID, 0.25µm film
Cat. No.: 24079
Oven: 40°C (5 min) to 230°C at 4°C/min
Det.: ion trap MS, m/z = 30-350, 0.6 sec/scan
Inj.: splitless/split (closed 0.5 min), 270°C (0.75mm ID inlet liner)

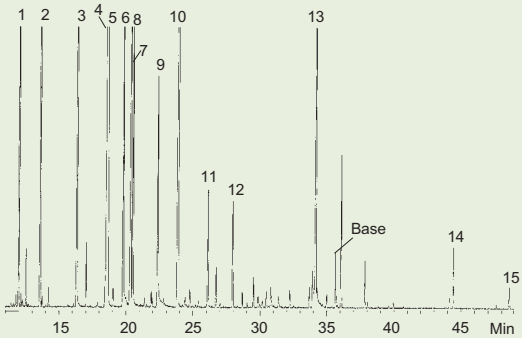


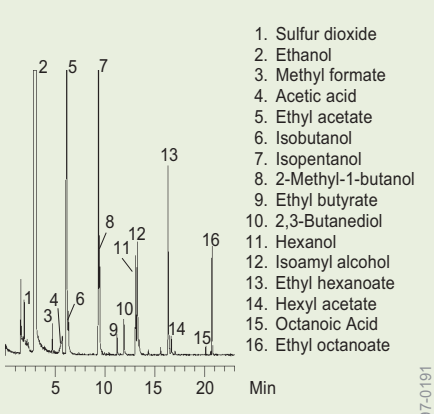
Figure provided by Eleanor Wiesenfeld, Manager, Analytical Services, Noville, Inc., 3 Empire Boulevard, South Hackensack, NJ 07606-1806 USA.

	Analyte	Boiling Pt. (°C)
1.	Myrcene	167
2.	Limonene	177
3.	Linalool	198
4.	Citronellal	206
5.	Benzyl acetate	215
6.	Menthol	216
7.	Methyl salicylate	223
8.	α-Terpineol	219
9.	Citronellol	225
10.	Linalyl acetate	220
11.	Heliotropine	263
12.	Eugenol	253
13.	Methyl ionone gamma	255
14.	Hexyl cinnamic aldehyde	305
15.	Benzyl salicylate	300

797-0627

Shampoo Base with Fragrance (SPME/GC)

Sample: 0.5g shampoo base with 1% fragrance test mix, diluted 1:1 with NaCl/water in a 10mL vial
SPME Fiber: polydimethylsiloxane, 100µm film
Cat. No.: 57300-U
Extraction: headspace, 5 min, 80°C (with sample stirring)
Desorption: 5 min, 250°C
Column: methyl silicone, 50m x 0.25mm ID, 0.25µm film
Supelco Equivalent: Equity-1 (available on request)
Oven: 35°C to 240°C at 3°C/min
Cryo: -50°C w/liquid CO_2
Flow: 0.5mL/min
Det.: MSD, scan m/z = 29-350 AMU
Inj.: split, 250°C



1. Sulfur dioxide
2. Ethanol
3. Methyl formate
4. Acetic acid
5. Ethyl acetate
6. Isobutanol
7. Isopentanol
8. 2-Methyl-1-butanol
9. Ethyl butyrate
10. 2,3-Butanediol
11. Hexanol
12. Isoamyl alcohol
13. Ethyl hexanoate
14. Hexyl acetate
15. Octanoic Acid
16. Ethyl octanoate

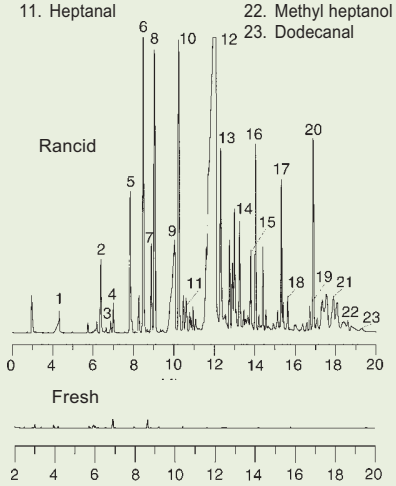
97-0191

Volatiles in White Wine (SPME/GC)

Sample: White wine + 25% NaCl
SPME Fiber: Carboxen/PDMS
Cat. No.: 57318
Extraction: 10 min, 40°C (headspace)
Desorption: 3 min at 290°C
Column: VOCOL, 30m x 0.25mm ID, 1.5µm film
Cat. No.: 24205-U
Det.: GC/MS, quadrupole, m/z = 31-240

SPME Applications
Foods, Flavors and Fragrances

1. Acetic acid
2. Pentanal
3. Butanoic acid
4. Propyl acetate
5. Methyl butyrate
6. Hexanal
7. Octane
8. Methyl hexanal
9. Hexanoic acid
10. Heptanone
11. Heptanal
12. Heptanoic acid
13. Octanal
14. Octanoic acid
15. Nonanone
16. Nonanal
17. Butyl hexanoate
18. Decanal
19. Undecanone
20. Pentyl hexanoate
21. Dodecanone
22. Methyl heptanol
23. Dodecanal



G000075, 76

Potato Chips (SPME/GC)

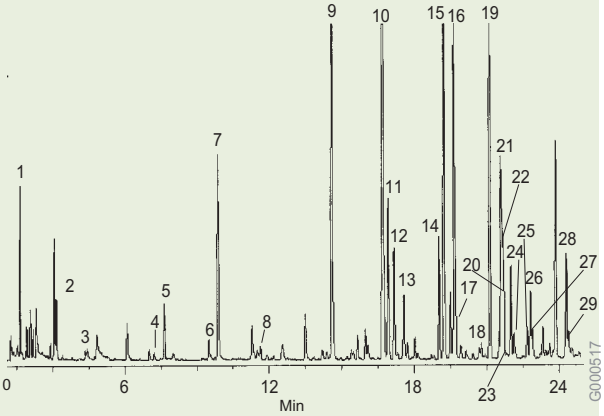
Sample:	3g crushed potato chips in 15mL vial
SPME Fiber:	DVB/Carboxen/PDMS, 50/30µm film, StableFlex
Cat. No.:	57328-U
Extraction:	headspace, 65°C, 20 min, with stirring
Desorption.:	3 min, 250°C
GC Column:	SPB-1 SULFUR30m x 0.32mm ID, 4.0µm film
Cat. No.:	24158
Oven:	45°C (1.5 min) to 250°C at 12°C/min, hold 10 min
Carrier:	helium, 40cm/sec
Det.:	quadrupole mass spectrometer, m/z = 35-290 at 0.6 sec/scan

Some Volatile Components in Peanut Butter

1. Carbon disulfide
2. 3-Methylbutanal
3. Pentanal
4. Dimethyl disulfide
5. Hexanal
6. 1-Methyl pyrrole
7. 4-Methyl-pentene-2-one
8. Heptanal

Pyrazines in Peanut Butter

9. 2-Methyl pyrazine
10. 2,5-Dimethyl pyrazine
11. 2,3-Dimethyl pyrazine
12. 2-Ethyl pyrazine
13. 2,6-Dimethyl pyrazine
14. 2-Ethyl-6-methyl pyrazine
15. 2-Ethyl-5-methyl pyrazine
16. Trimethyl pyrazine
17. 2-Ethyl-3-methyl pyrazine
18. 2,6-Diethyl pyrazine
19. 2-Ethyl-3,5-dimethyl pyrazine
20. 2,3-Diethyl pyrazine
21. 2-Methyl-5-isopropyl pyrazine
22. 3-Ethyl-2,5-dimethyl pyrazine
23. 5-Methyl-2-propyl pyrazine
24. 2-Methyl-5-propyl pyrazine
25. 2-Ethenyl-6-methyl pyrazine
26. 3,5-Diethyl-2-methyl pyrazine
27. 2-Ethenyl-5-methyl pyrazine
28. 2-Methyl-6-cis propenyl pyrazine
29. 2-Allyl-5-methyl pyrazine



G000517

Pyrazines in Peanut Butter (SPME/GC)

Sample:	5g peanut butter in 40mL vial
SPME Fiber:	divinylbenzene-Carboxen-polydimethylbenzene StableFlex
Cat. No.:	57328-U
Extraction:	headspace, 30 min at 65°C in heating block
Desorption:	270°C for 5 min
Column:	SUPELCOWAX 10, 30m x 0.25mm, 0.25µm film
Cat. No.:	24079
Oven:	40°C (5 min) to 230°C at 4°C/min
Inj.:	splitless/split, closed 0.5 min, 270°C, with 0.75mm liner
Det.:	ion trap mass spectrometer, m/z = 30-350 at 0.6 sec/scan Selected ions used for quantitation.

SPME Applications
Hydrocarbons and Pesticides

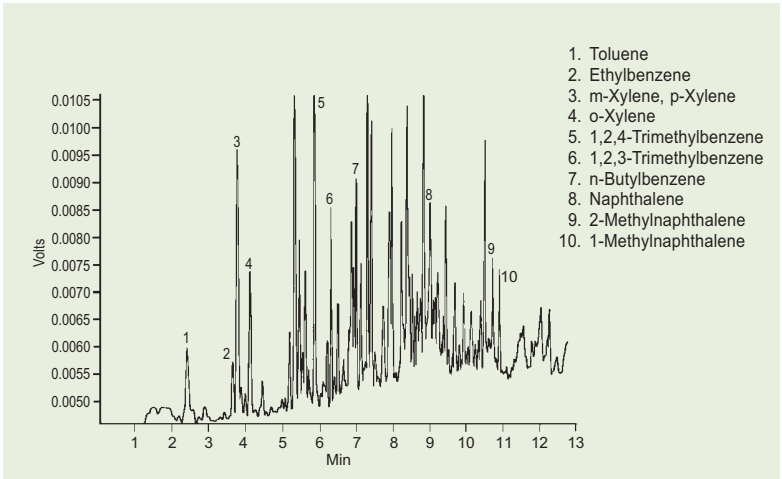
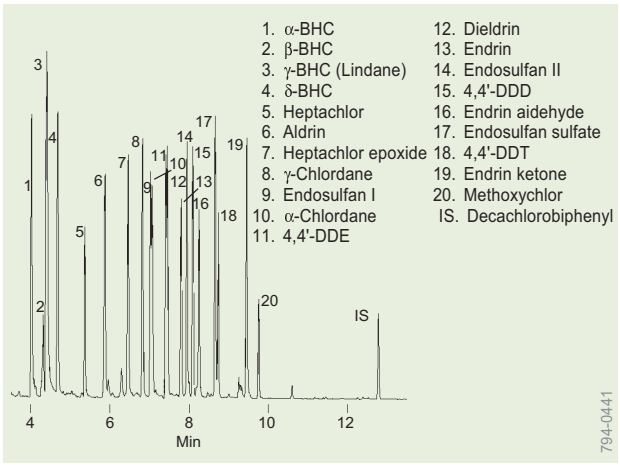


Figure courtesy of José Almirall, Crime Laboratory Bureau, Metro-Dade Police Department, Miami, FL, USA, and Kenneth Furton and Juan Bruna, Department of Chemistry, Florida International University, Miami.

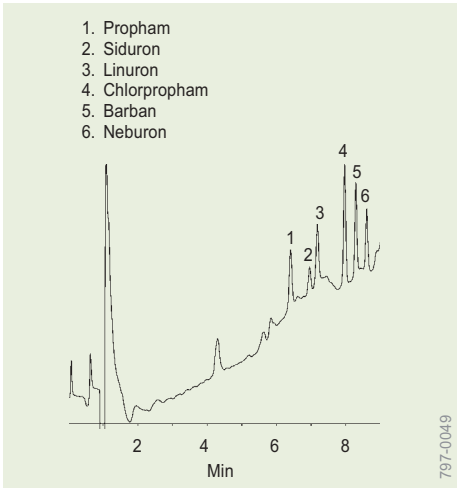
Gasoline in Arson Samples (SPME/GC)

SPME: polydimethylsiloxane phase fiber, 100µm film
headspace sampling (20 min) 10 sec desorption (splitless mode)
Cat. No.: 57300-U
Column: polydimethylsiloxane phase, 30m x 0.25mm ID, 0.25µm film
Supelco Equivalent: Equity-1 (Cat. No. 28046)
Oven: 35°C (2 min) to 220°C at 10°C/min, hold 2 min, to 300°C at 30°C/min, hold 5 min
Carrier: helium, 1mL/min (split 50:1)
Det.: FID, 300°C
Inj.: splitless (closed 3 min), 220°C (2mm ID injector liner)



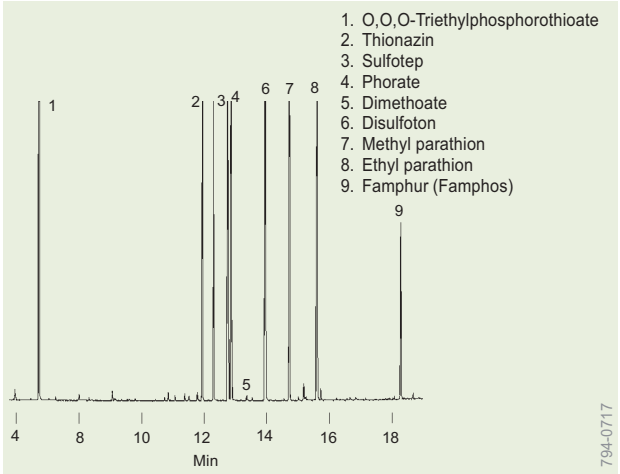
Chlorinated Pesticides (SPME/GC)

Sample: 200ppt each in 2mL water
Extraction: 15 min immersed in water with rapid stirring
SPME Fiber: polydimethylsiloxane, 100µm film
Cat. No.: 57300-U
Column: SPB-5, 15m x 0.20mm ID, 0.20µm film
Cat. No.: 24165-U
Oven: 120°C (1 min) to 180°C at 30°C/min, then to 290°C at 10°C/min
Carrier: helium, 37cm/sec (set at 120°C)
Det.: ECD, 300°C
Inj.: 260°C (splitless - closed 3min)



Carbamate and Urea Pesticides (SPME/HPLC)

Sample: 3mL water containing 8ng/mL of each analyte in 10% NaCl
SPME Fiber: PDMS/DVB, 60µm film
Cat. No.: 57317
Extraction: immersion, 40 min, rapid stirring
Desorption: static, 5 min in acetonitrile:water (65:35); dynamic, valve open during run
Column: SUPELCOSIL LC-8, 15cm x 4.6mm ID, 5µm particles
Cat. No.: 58220-U
Mobile Phase: acetonitrile:water (18:82 to 65:35 in 9 min, hold 3 min)
Flow Rate: 2.0mL/min
Temp.: 35°C
Det.: UV, 240nm

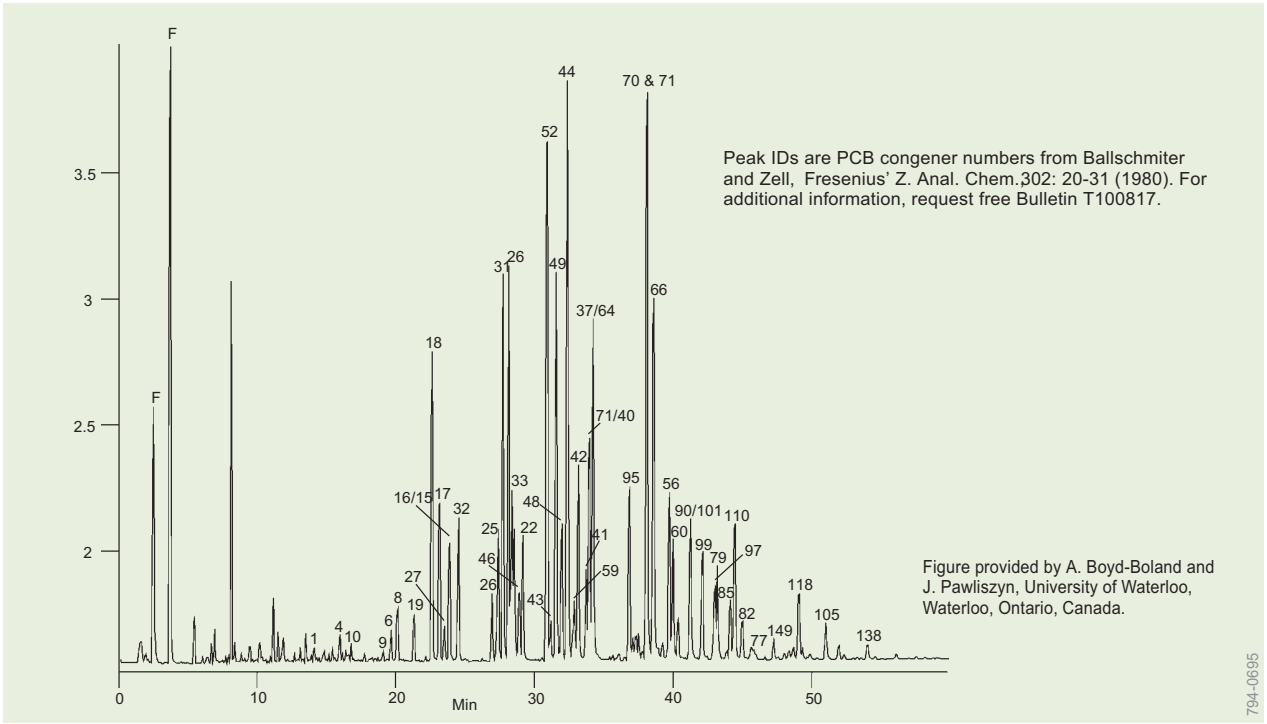


Organophosphorous Pesticides (SPME/GC)

Sample: 50ppb each in 1.5mL saturated salt water, pH 7.2
Extraction: 20 min immersion with rapid stirring
SPME Fiber: polydimethylsiloxane, 100µm film
Cat. No.: 57300-U
Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Cat. No.: 24135-U
Oven: 60°C (1 min) to 300°C at 12°C/min, hold 5 min
Carrier: helium, 30cm/sec
Det.: MS (m/z = 45-400, 0.6sec/scan)
Inj.: splitless (0.75mm ID liner), 270°C

SPME Applications

Herbicides, PCBs, and Phenols



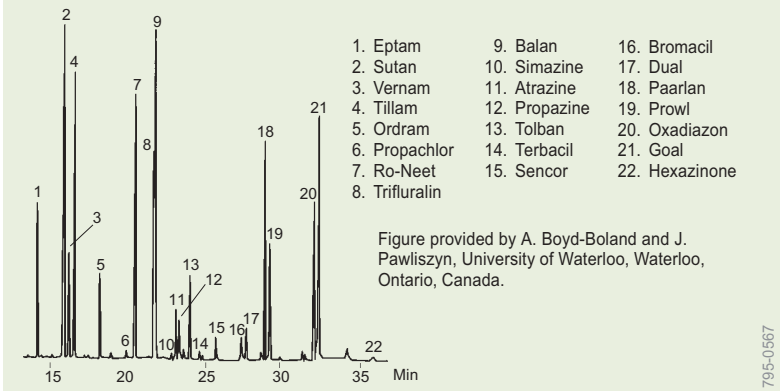
Peak IDs are PCB congener numbers from Ballschmiter and Zell, Fresenius' Z. Anal. Chem.302: 20-31 (1980). For additional information, request free Bulletin T100817.

Figure provided by A. Boyd-Boland and J. Pawliszyn, University of Waterloo, Waterloo, Ontario, Canada.

794-0695

PCB Congeners in Stream Sediment (SPME/GC)

SPME Fiber: PDMS, 100µm film
Cat. No.: 57300-U
Extraction: headspace, 90°C/30 min
Desorption: 280°C/2 min, splitless
Column: SPB-Octyl, 30m x 0.25mm ID, 0.25µm film
Cat. No.: 24218-U
Oven: 50°C/1 min at 10°C/min to 150°C/0 min
Sample: 5g stream sediment, downstream from industrial sites



1. Eptam
2. Sutan
3. Vernam
4. Tillam
5. Ordram
6. Propachlor
7. Ro-Neet
8. Trifluralin
9. Balan
10. Simazine
11. Atrazine
12. Propazine
13. Tolban
14. Terbacil
15. Sencor
16. Bromacil
17. Dual
18. Paarlan
19. Prowl
20. Oxadiazon
21. Goal
22. Hexazinone

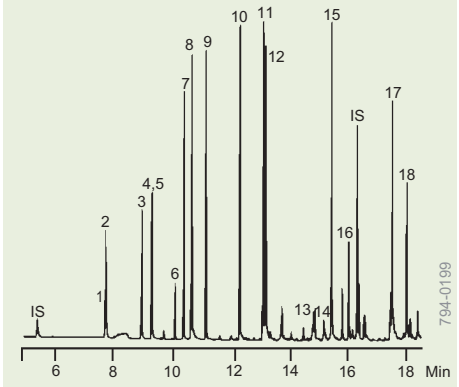
Figure provided by A. Boyd-Boland and J. Pawliszyn, University of Waterloo, Waterloo, Ontario, Canada.

795-0567

Nitrogen-Containing Herbicides Extracted from Water (SPME/GC)

Sample: 4mL (water + 100ng/mL each analyte 1g/mL NaCl), pH 2, in 4.6mL vial
SPME Fiber: polyacrylate, 85µm film
Cat. No.: 57304
Extraction: immersion, ambient temp., 50 min (constant stirring)
Desorption: 5 min, 230°C
Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Cat. No.: 24135-U
Oven: 40°C (5 min) to 100°C at 30°C/min, to 275°C at 5°C/min
Carrier: helium, 40cm/sec, set at 40°C
Det.: MS (Ion Trap Manifold: 250°C;
Mass Scan Range: m/z = 45-400 at 0.6 sec/scan)
Inj.: splitless, 230°C

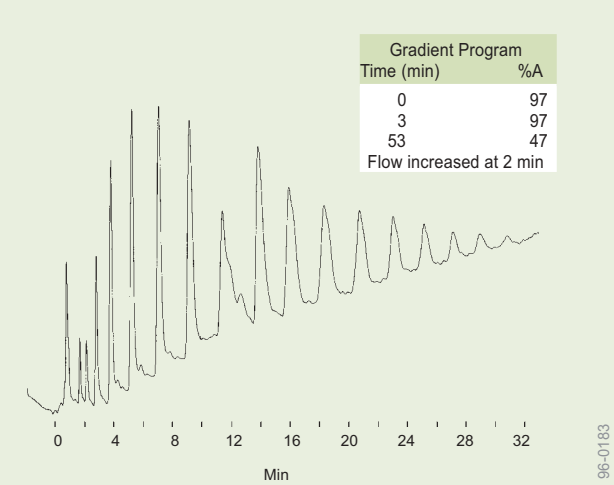
IS 2-Fluorophenol (int. std.)
1. Phenol
2. 2-Chlorophenol
3. 2-Methylphenol
4. 3-Methylphenol
5. 4-Methylphenol
6. 2-Nitrophenol
7. 2,4-Dimethylphenol
8. 2,4-Dichlorophenol
9. 2,6-Dichlorophenol
10. 4-Chloro-3-methylphenol
11. 2,4,5-Trichlorophenol
12. 2,4,6-Trichlorophenol
13. 2,4-Dinitrophenol
14. 4-Nitrophenol
15. 2,3,4,6-Tetrachlorophenol
16. 2-Methyl-4,6-dinitrophenol
IS 2,4,6-Tribromophenol (int. std.)
17. Pentachlorophenol
18. Dinoseb



794-0199

Phenols in Water (SPME/GC)

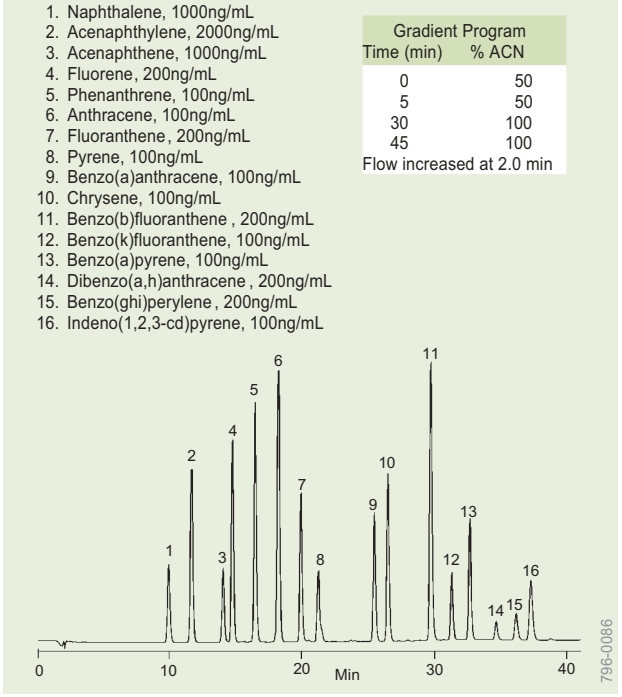
Sample: 50ppb phenols in 1.8mL saturated salt water, pH 2
Extraction: 20 min immersed in water with rapid stirring
SPME Fiber: polyacrylate, 85µm film
Cat. No.: 57304
Desorption: splitless, 280°C (closed 3 min)
Column: PTE-5, 30m x 0.25mm ID, 0.25µm film
Cat. No.: 24135-U
Oven: 40°C (4 min) to 260°C at 12°C/min
Carrier: helium, 40cm/sec at 40°C
Det.: MS, Scan Range m/z = 45-465 at 0.6 sec/scan



Nonionic Surfactants in Water (SPME/HPLC)

Sample: Triton X-100 in water (500mg/mL)
SPME Fiber: Carbowax/TPR-100, 65µm film
Cat. No.: 57315
Extraction: immersion, 50 min (rapid stirring)
Desorption: static, 200µL, mobile phase A:B (97:3), 2 min
Column: SUPELCOSIL LC-NH_2 25cm x 4.6mm, 5µm particles
Cat. No.: 58338
Mobile Phase: A = hexane:2-propanol (90:10) B = 2-propanol:water (90:10)
Flow Rate: 0.5mL/min (0-2 min) 1.5mL/min (2-53min)
Det.: UV, 220nm
Inj.: 200µL

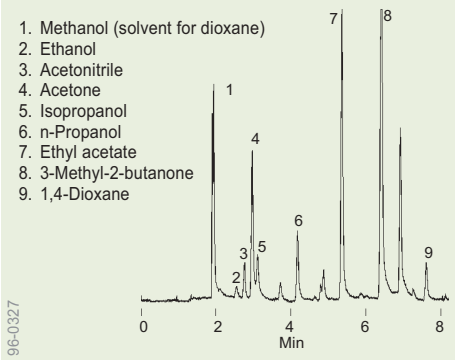
1. Naphthalene, 1000ng/mL
2. Acenaphthylene, 2000ng/mL
3. Acenaphthene, 1000ng/mL
4. Fluorene, 200ng/mL
5. Phenanthrene, 100ng/mL
6. Anthracene, 100ng/mL
7. Fluoranthene, 200ng/mL
8. Pyrene, 100ng/mL
9. Benzo(a)anthracene, 100ng/mL
10. Chrysene, 100ng/mL
11. Benzo(b)fluoranthene , 200ng/mL
12. Benzo(k)fluoranthene, 100ng/mL
13. Benzo(a)pyrene, 100ng/mL
14. Dibenzo(a,h)anthracene , 200ng/mL
15. Benzo(ghi)perylene , 200ng/mL
16. Indeno(1,2,3-cd)pyrene, 100ng/mL



PAHs in Water (SPME/HPLC)

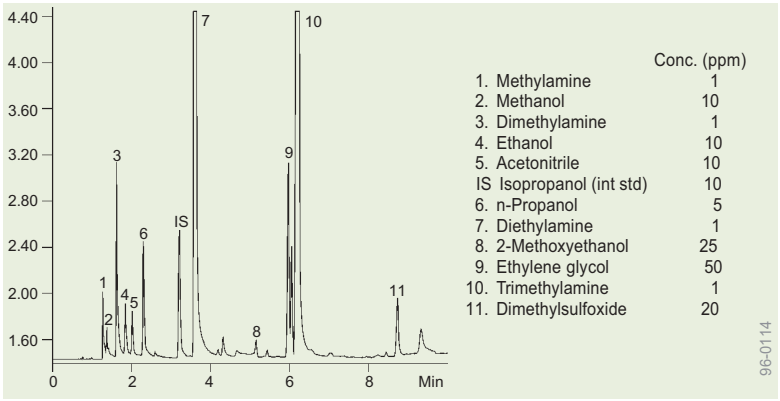
Sample: 5µL PAH mix (Cat. No. 4-8743) in 5mL water
SPME Fiber: polydimethylsiloxane, 100µm film
Cat. No.: 57301
Extraction: immersion, 30 min (rapid stirring)
Desorption: static, 200µL acetonitrile:water, 40:60, 2 min
Column: SUPELCOSIL LC-PAH, 15cm x 4.6mm ID, 5µm particles
Cat. No.: 58318
Mobile Phase: acetonitrile:water gradient (see program)
Flow Rate: 0-2 min: 0.2mL/min 2-45 min: 1.0mL/min
Det.: UV, 254nm

SPME Applications
Semivolatiles, Solvents



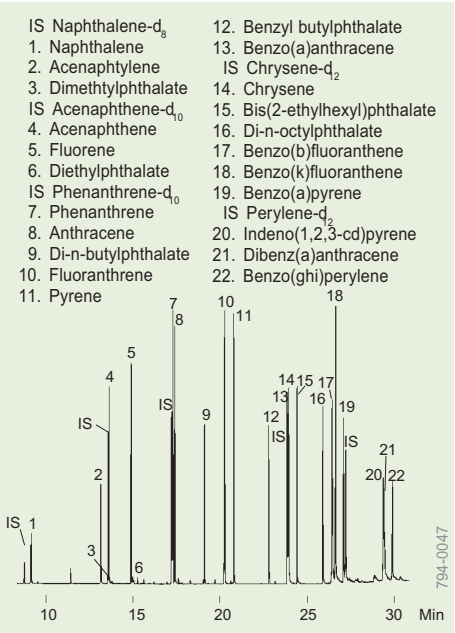
Solvents in Water at 20ppb (SPME /GC)

Sample:	solvents at 20ppb in 4mL water + 25% NaCl in 4mL vial
SPME Fiber:	Carboxen/PDMS, 75µm film
Cat. No.:	57318
Extractions:	immersion, 10 min., rapid stirring
Desorption:	3 min., 260°C
Column:	SPB-SULFUR, 30m x 0.32mm ID, 4.0µm film
Cat. No.:	24158
Oven:	50°C (2 min) to 150°C at 10°C/min
Carrier:	helium, 30cm/sec
Det.:	FID
Inj.:	splitless (closed 2 min), 260°C, 0.75mm ID liner



Pharmaceutical Solvents (SPME/GC)

Sample:	water + 25% NaCl, pH 11.0 immersion: 3.9mL sample + 0.1mL 50% Na_3PO_4 / 1g NaCl headspace: 2.4mL sample + 0.06mL 50% Na_3PO_4 / 0.6g NaCl
SPME Fiber:	polydimethylsiloxane/DVB, 65µm film
Cat. No.:	57310-U
Extraction:	immersion, 15 min (rapid stirring or headspace, 15 min, 55°C)
Desorption:	270°C, 5 min
Column:	SPB-1 SULFUR, 30m x 0.32mm ID, 4.0µm film
Cat. No.:	24158
Oven:	50°C (2 min) to 180°C at 10°C/min
Carrier:	helium, 50cm/sec
Det.:	FID
Inj.:	splitless (3 min), 270°C (0.75mm ID liner)



Semivolatiles in Water (SPME/GC)

Sample:	water spiked with PAHs and phthalates at 50ppb
SPME Fiber:	polydimethylsiloxane, 7µm film
Cat. No.:	57302
Sampling:	direct immersion in water, 15 min (rapid stirring)
Inj.:	split/splitless, 280°C (closed 4 min)
Column:	PTE-5, 30m x 0.25mm ID, 0.25µm film
Cat. No.:	24135-U
Oven.:	60°C (3 min) to 320°C at 10°C/min
Carrier:	helium, 40cm/sec at 60°C
Det.:	MS, Scan Range m/z = 45-465 at 0.6 sec/scan

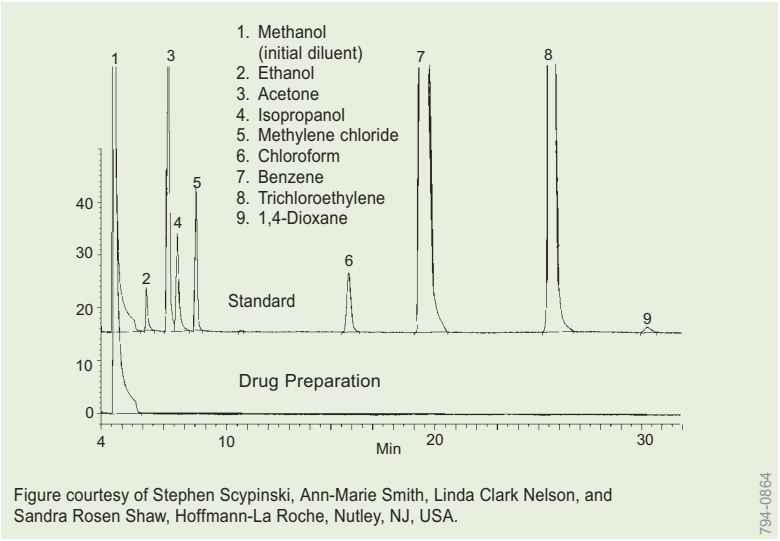
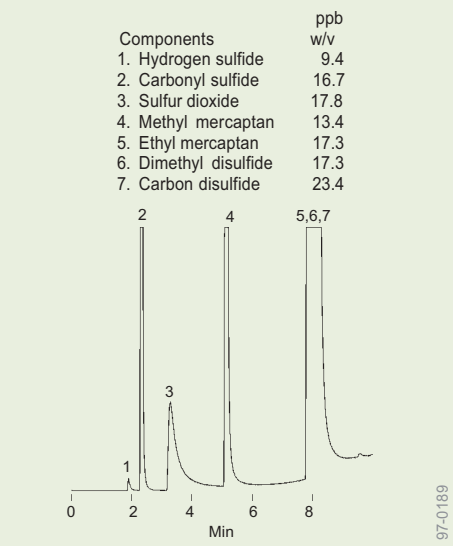


Figure courtesy of Stephen Scypinski, Ann-Marie Smith, Linda Clark Nelson, and Sandra Rosen Shaw, Hoffmann-La Roche, Nutley, NJ, USA.

Residual Solvents in Pharmaceutical Preparations (SPME/GC)

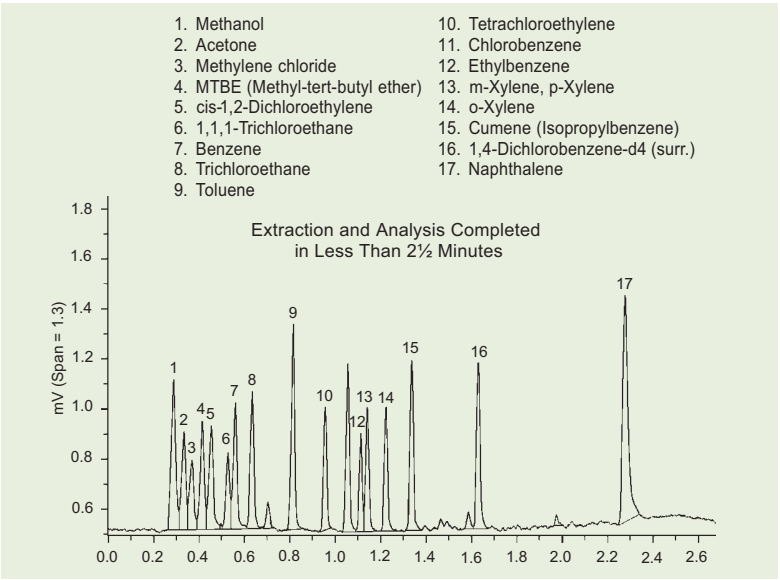
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	headspace or immersion sampling (15 min) 1 min desorption
Column:	SPB-624, 75m x 0.53mm ID, 3.0µm film
Cat. No.:	25432
Oven:	40°C (35 min) to 220°C at 40°C/min, hold 5 min
Carrier:	helium, 35cm/sec
Det.:	FID, 250°C
Inj.:	splitless (closed 3 min), 200°C

SPME Applications
Odors, Sulfur Compounds and Volatiles



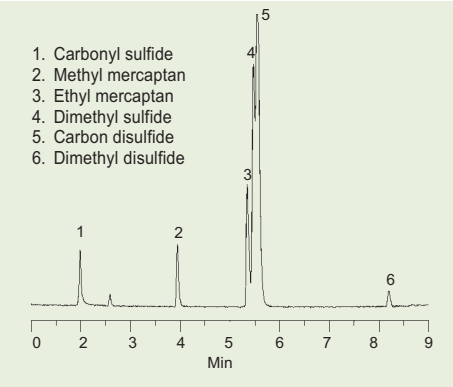
Sulfur Gases (SPME/GC)

Sample: sulfur gases in 40mL vial
SPME Fiber: Carboxen/PDMS, 75µm film
Cat. No.: 57318
Extraction: 10 min headspace at 40°C
Desorption: 3 min at 290°C
Column: Supel-Q PLOT, 30m x 0.32mm ID
Cat. No.: 24242
Oven: 45°C (1 min) to 250°C at 15°C/min



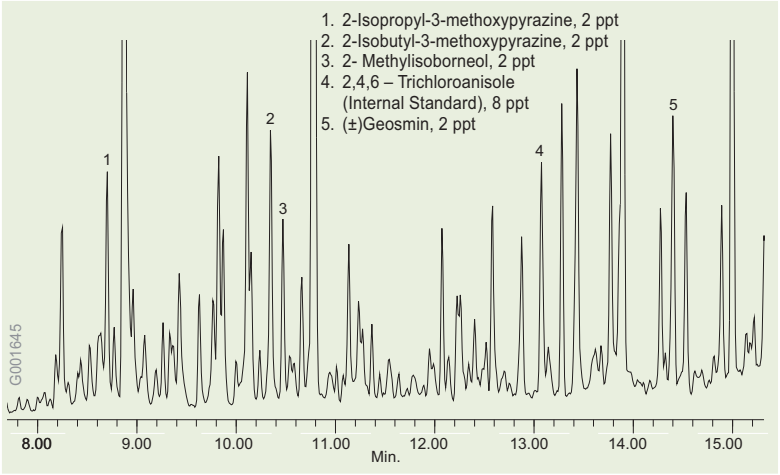
Water Sample Screened for BTEX and Other Compounds (SPME/GC)

Sample: 0.7mL water spiked with 2-600µg/liter each analyte, 0.25g NaCl added
SPME Fiber: 30µm PDMS
Cat. No.: 57309
Extraction: headspace, 12 sec (no stirring), ambient temp.
Desorption: 2-3 min, 250°C (0.75-1. 0mm ID inj. port liner)
GC Column: Equity-1 , 10m x 0.20mm ID, 1.2µm film (available on request)
Oven: 70°C (0.2 min) to 180°C at 50°C/min
Carrier: hydrogen, 12psi (+ 30mL/min nitrogen make-up gas)
Det.: FID, 250°C



Sulfur Gases at 1ppm (SPME/GC)

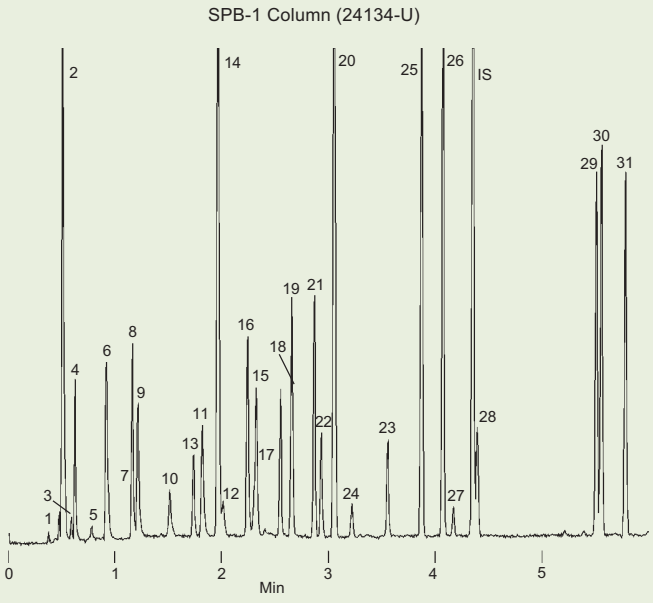
Sample: sulfur gases in air at 1ppm, 250mL bulb
SPME Fiber: Carboxen/PDMS, 75µm film
Cat. No.: 57318
Extraction: headspace, 5 min., ambient temp.
Desorption: 2 min., 250°C
Column: Supel-Q PLOT, 30m x 0.32mm ID
Cat. No.: 24242
Oven: 45°C (0.75 min.) to 250°C at 25°C/min
Carrier: helium, 25cm/sec
Injection: splitless/split (closed 2 min.), 0.75mm ID liner
Detector: MS quadrupole, m/z = 32 - 125 (0.6sec/scan)

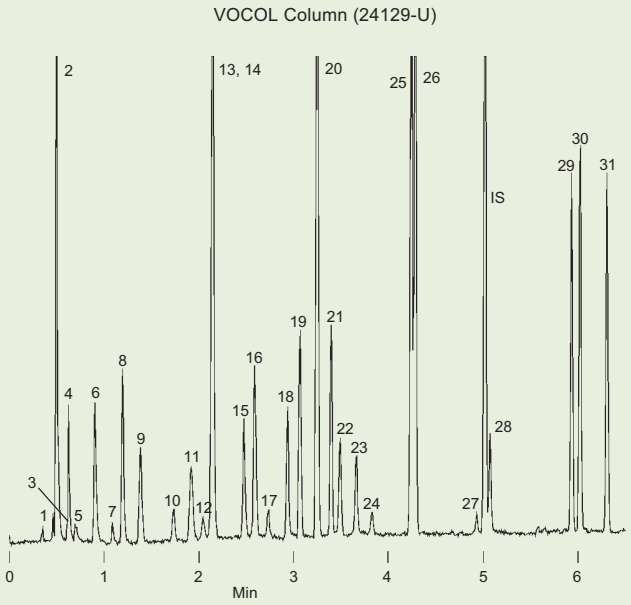


Trace Odors in Drinking Water (SPME/GC)

Sample: 25mL of water containing 25% NaCl and drinking water odors kit, Cat. No. 47529-U
SPME Fiber: 2cm StableFlex coated with 50/30µm DVB/Carboxen/PDMS
Cat. No.: 57348-U
Extraction: headspace, 65°C (30 min.)
Column: Equity-5, 30m x 0.25mm, 0.25µm film
Cat. No.: 28089-U
Desorption: 3 min. at 260°C
Oven: 60°C (2min) to 200°C at 8°C/min
GC Liner: 0.75mm SPME liner
Detector: MS, selected ions (SIM) 95, 112, 124, 137, 197; interface at 280°C
Flow: Helium, 37cm/sec @ 60°C (1mL/min constant flow)
Inj.: SPME fiber, splitless opened at after 1 min at 50mL/min.

SPME Applications
Volatiles





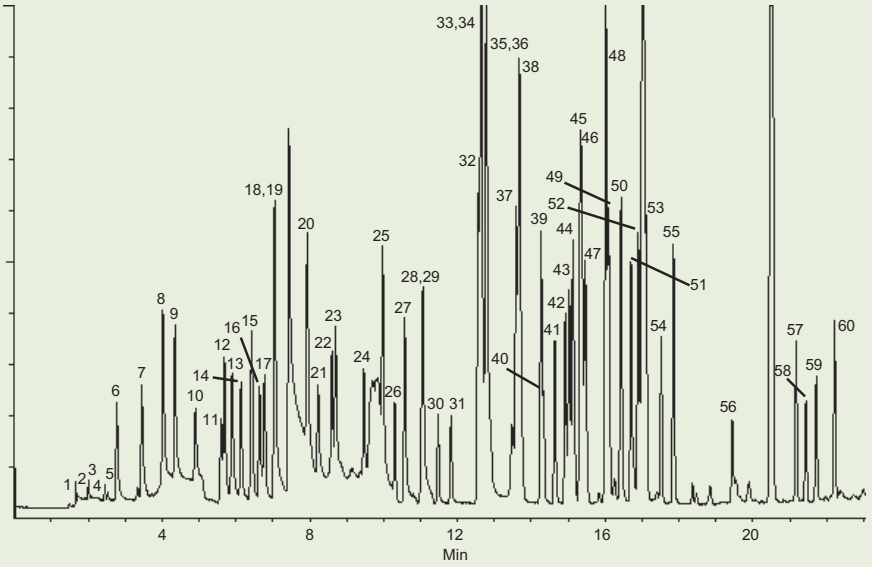
1. Chloromethane
2. Vinyl chloride
3. Bromomethane
4. Chloroethane
5. Trichlorofluoromethane
6. 1,1-Dichloroethylene
7. Methylene chloride
8. trans-1,2-Dichloroethene
9. 1,1,-Dichloroethane
10. Chloroform
11. 1,1,1-Trichloroethane
12. Carbon tetrachloride
13. Benzene
14. 1,2-Dichloroethane
15. Trichloroethylene
16. 1,2-Dichloropropane
17. Bromodichloromethane
18. 2-Chloroethyl vinyl ether
19. cis-1,3-Dichloropropene
20. Toluene
21. trans-1,3-Dichloropropene
22. 1,1,2-Trichloroethane
23. Tetrachloroethylene
24. Dibromochloromethane
25. Chlorobenzene
26. Ethylbenzene
27. Bromoform
IS 1,4-Dichlorobutane
28. 1,1,2,2-Tetrachloroethane
29. 1,3-Dichlorobenzene
30. 1,4-Dichlorobenzene
31. 1,2-Dichlorobenzene

794-0438, 0439

Volatiles (SPME/GC)

Sample:	50ppb each analyte in 1.8mL saturated salt water
SPME Fiber:	polydimethylsiloxane, 100µm film
Cat. No.:	57300-U
Extraction:	5 min immersed in water with rapid stirring
Columns:	10m x 0.20mm ID, 1.2µm film
Oven:	40°C (0.75 min) to 160°C at 20°C/min
Carrier:	helium, 40cm/sec
Det.:	FID, 260°C
Inj.:	230°C, splitless (closed 3 min)

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1-Dichloroethylene
8. Methylene chloride
9. trans-1,2-Dichloroethylene
10. 1,1,-Dichloroethane
11. 2,2-Dichloropropane
12. cis-1,2-Dichloroethylene
13. Chloroform
14. Bromochloromethane
15. 1,1,1-Trichloroethane
16. 1,1-Dichloropropene
17. Carbon tetrachloride
18. 1,2-Dichloroethane
19. Benzene
IS Fluorobenzene
20. Trichloroethylene
21. 1,2-Dichloropropane
22. Bromodichloromethane
23. Dibromomethane
24. cis-1,3-Dichloropropene
25. Toluene
26. trans-1,3-Dichloropropene
27. 1,1,2-Trichloroethane
28. 1,3-Dichloropropane
29. Tetrachloroethylene
30. Chlorodibromomethane
31. 1,2-Dibromoethane
32. Chlorobenzene
33. 1,1,1,2-Tetrachloroethane
34. Ethylbenzene
35. m-Xylene
36. p-Xylene
37. o-Xylene
38. Styrene
39. Isopropylbenzene
40. Bromoform
41. 1,1,2,2-Tetrachloroethane
42. 1,2,3-Trichloropropane
43. n-Propylbenzene
44. Bromobenzene
45. 1,3,5-Trimethylbenzene
46. 2-Chlorotoluene
47. 4-Chlorotoluene
48. tert-Butylbenzene
49. 1,2,4-Trimethylbenzene
50. sec-Butylbenzene
51. p-Isopropyltoluene
52. 1,3-Dichlorobenzene
53. 1,4-Dichlorobenzene
54. n-Butylbenzene
IS 1,2-Dichlorobenzene-d_4
55. 1,2-Dichlorobenzene
56. 1,2-Dibromo-3-chloropropane
57. 1,2,4-Trichlorobenzene
58. Hexachlorobutadiene
59. Naphthalene
60. 1,2,3-Trichlorobenzene



Volatile Compounds by US EPA 524.2 (SPME/GC)

Sample: 1ppb each analyte in 3mL water (25% NaCl added) in 4mL vial
SPME Fiber: Carboxen/PDMS, 75µm film
Cat. No.: 57318
Extraction: headspace, 40°C (20 min.), with stirring
Desorption: 5 min., 310°C

Column: VOCOL, 30m x 0.25mm ID, 1.5µm film
Cat. No.: 24205-U
Oven: 40°C (2 min.) to 210°C at 8°C/min
Carrier: helium, 35cm/sec
Injection: splitless/split (closed 2 min), 0.75mm ID liner
Detector: ion trap, MS, m/z = 45 - 260 (0.6sec/scan)