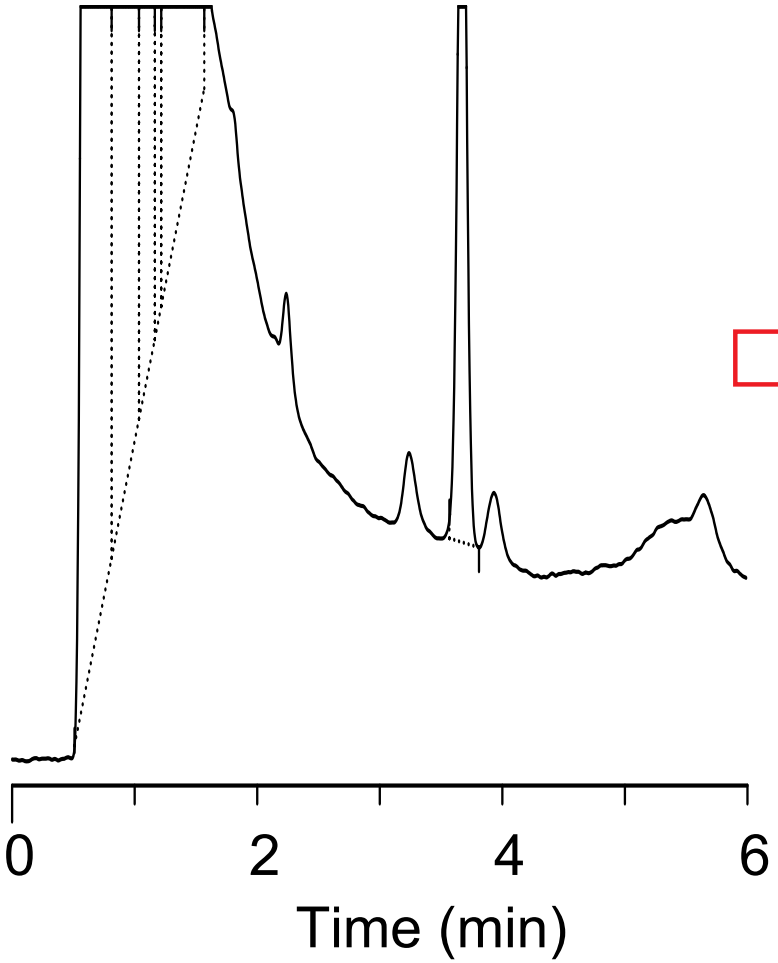
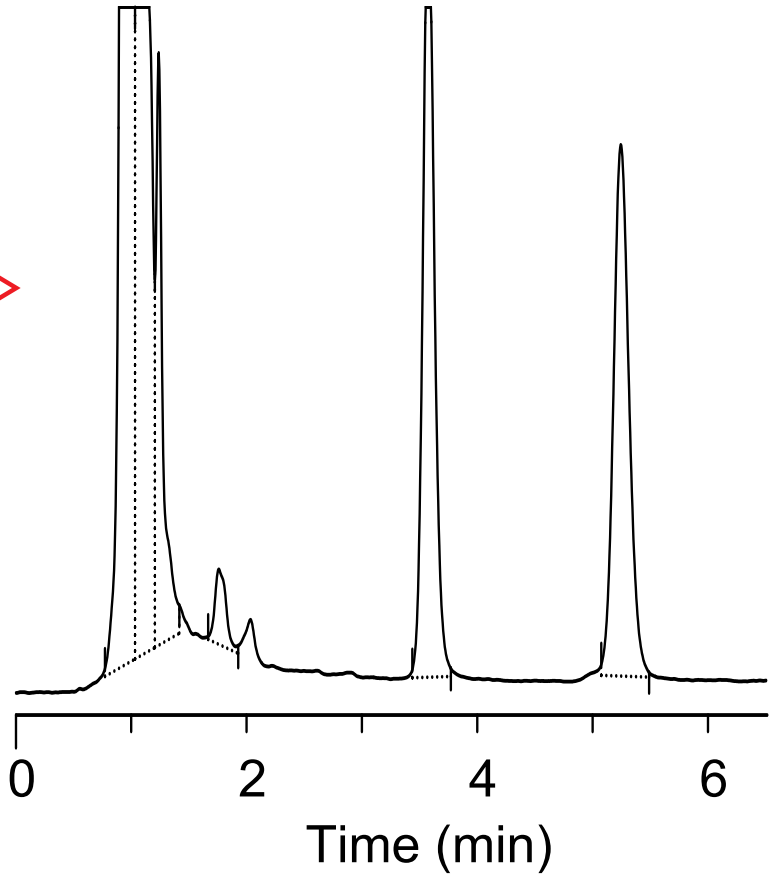
Sample Prep for Chromatographic Analysis of Difficult Matrixes

Real World & Real Samples

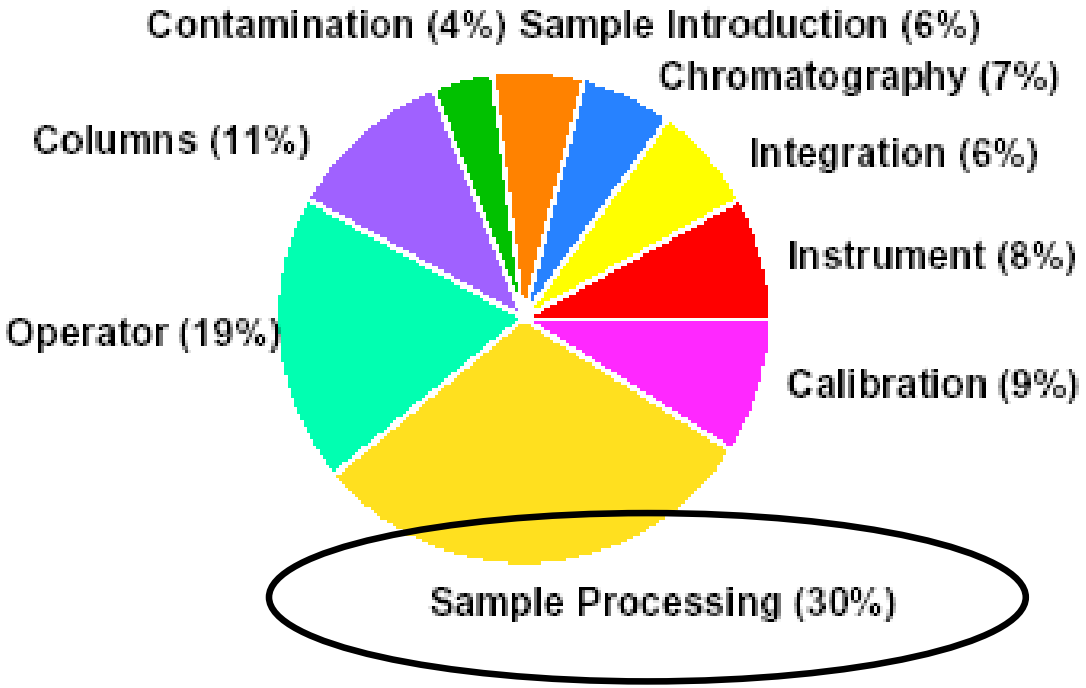
Urine Sample without sample prep



Urine Sample with sample prep

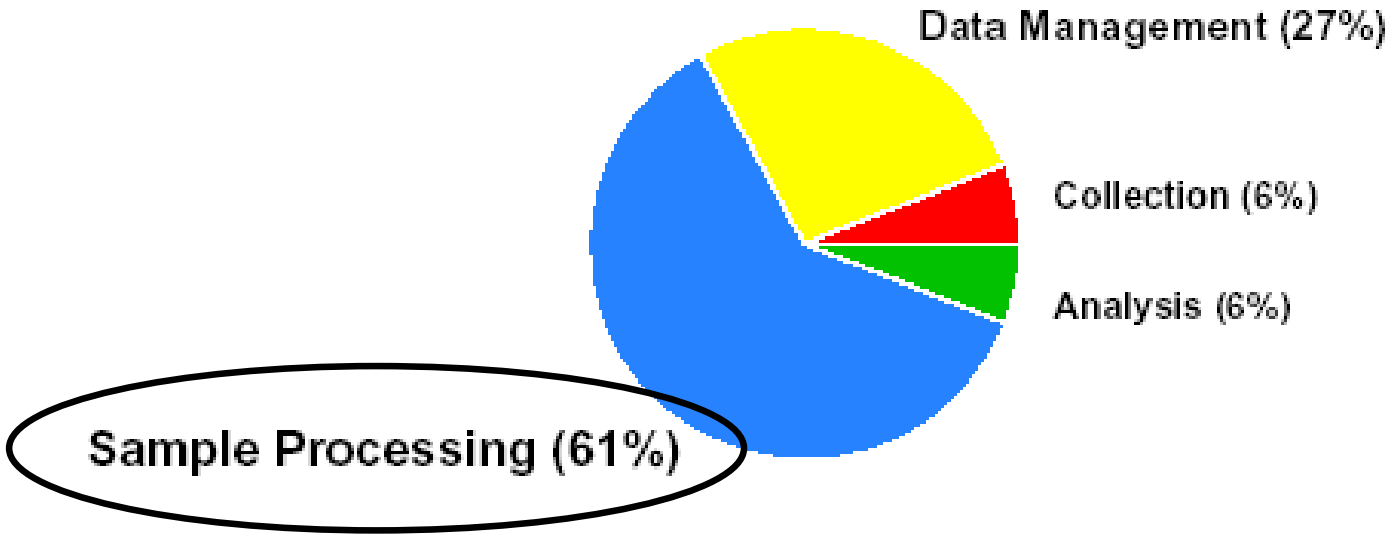


Sources of Chromatographic Errors



(R.E. Majors, LC/GC Magazine, 1991, 1997, 2002)

Time Spend on Analytical Process



(R.E. Majors, LC/GC Magazine, 1991, 1997, 2002)

Sample Prep Innovations

- Solid Phase Microextraction (SPME)
- High specificity SPE
- Dispersive SPE
- Silver Ion SPE for FAMEs
- Carbonaceous adsorbents
- Flash chromatography

Solid Phase Microextraction (SPME)

"Sample Prep Made Easy"

Enrichment technique mainly for trace analysis

Developed in collaboration with Janusz Pawliszyn, Univ. of Waterloo

Unique and proprietary to Supelco

Users are...
- **GC and GC-MS analysts (HPLC & LC-MS)**
- **Analyzing compounds in gases, liquids or solids.**

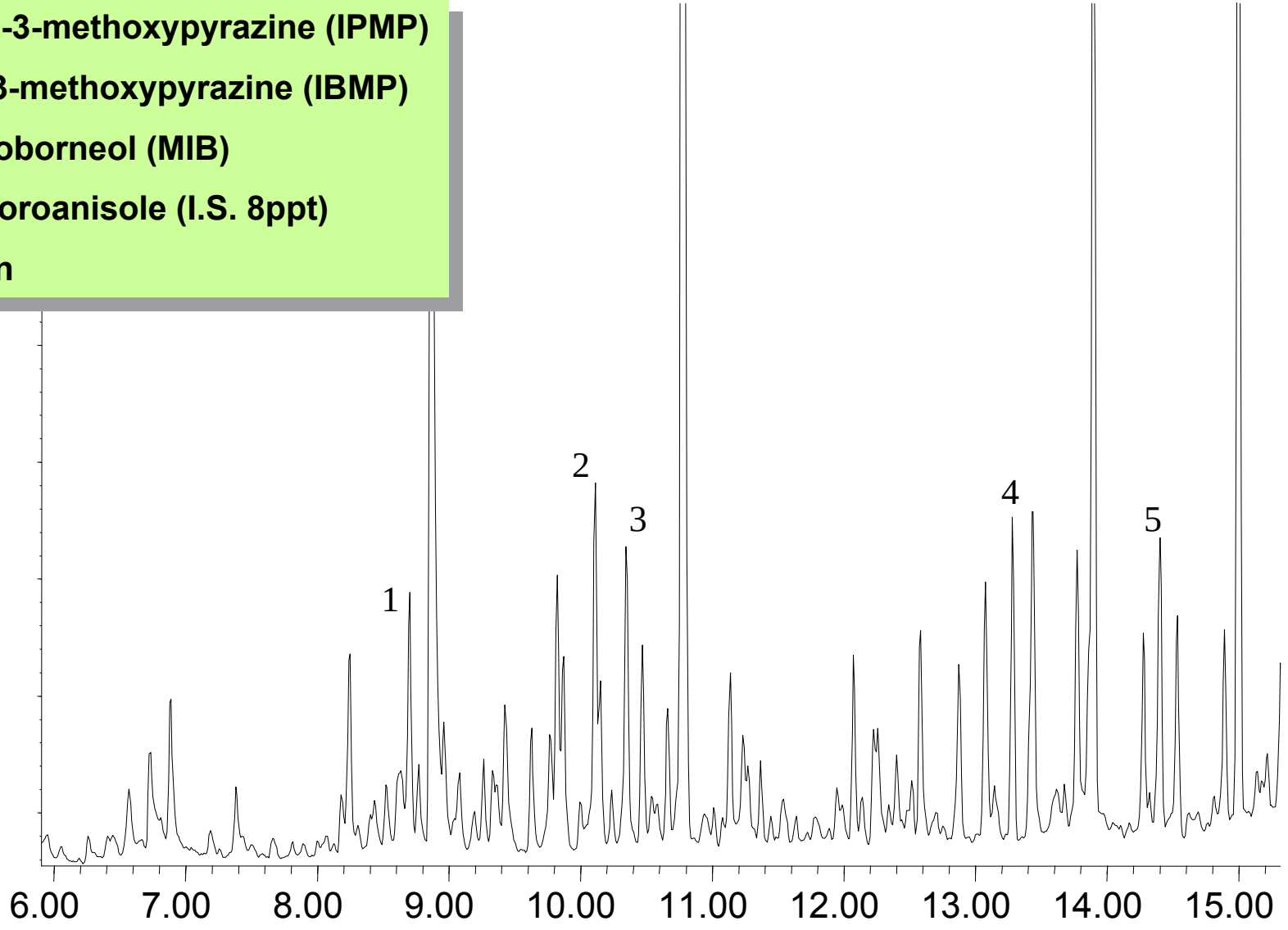
Interested in...
- **Sample enrichment**
- **Solventless extraction**
- **Using existing GC & HPLC systems**
- **Economical sample prep**
- **Reducing lab animal sacrifice**

Users can expect...
- **Highly consistent, quantifiable results from low concentrations of analytes**

Odor-Causing Compounds in Water at 2 ppt (GC/MS)

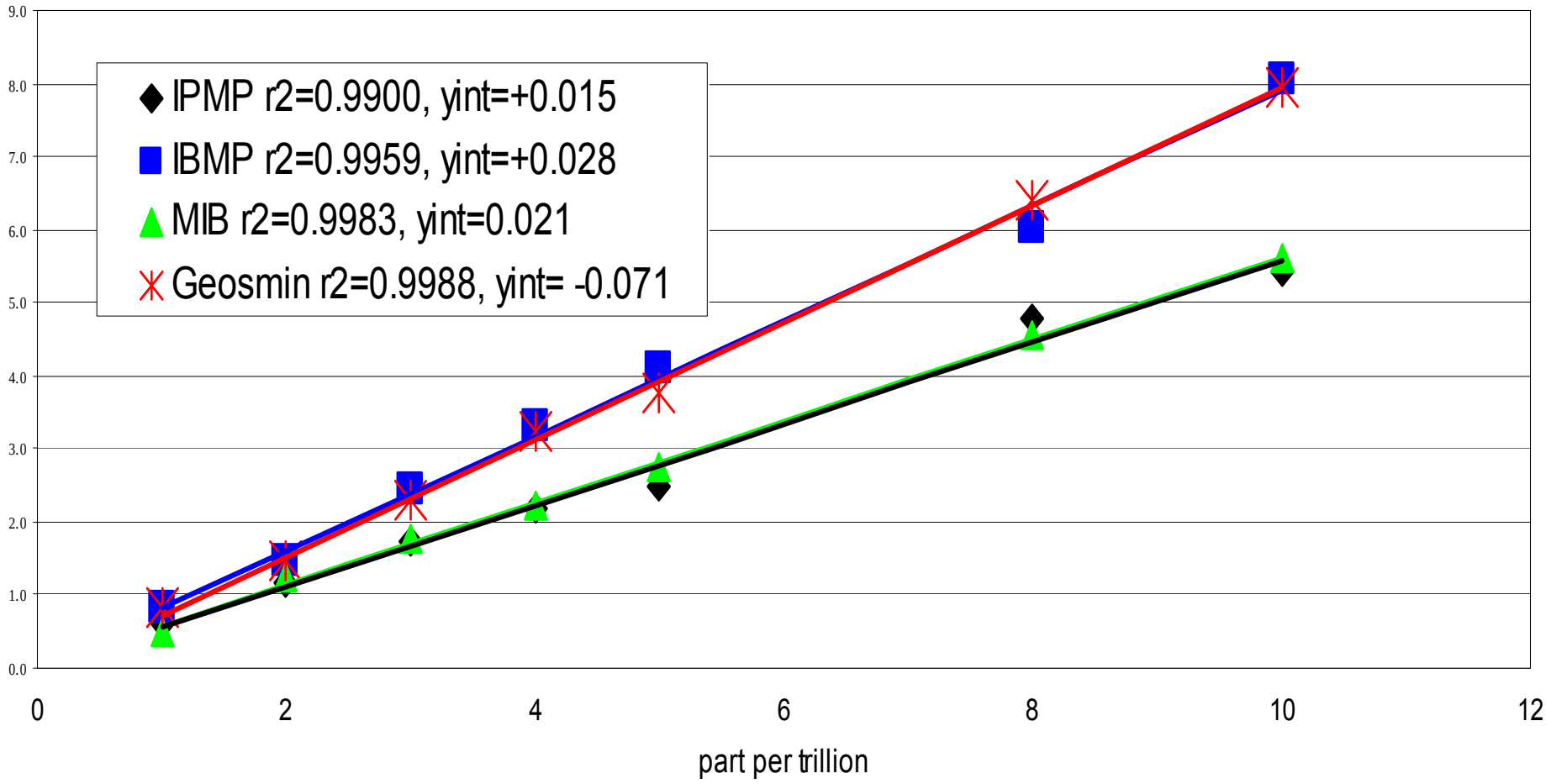
Sensitive

1. 2-Isopropyl-3-methoxypyrazine (IPMP)
2. 2-Isobutyl-3-methoxypyrazine (IBMP)
3. 2- Methylisoborneol (MIB)
4. 2,4,6-Trichloroanisole (I.S. 8ppt)
5. (±) Geosmin



Linearity of Odor-Causing Compounds from Water at ppt Levels (SPME-GC/MS)

Quantitative



SPME Overview

Solvent-free extraction technique for nearly any sample or matrix

Alternative to head-space GC and solid phase extraction (SPE) techniques

Directly interfaced with GC analysis

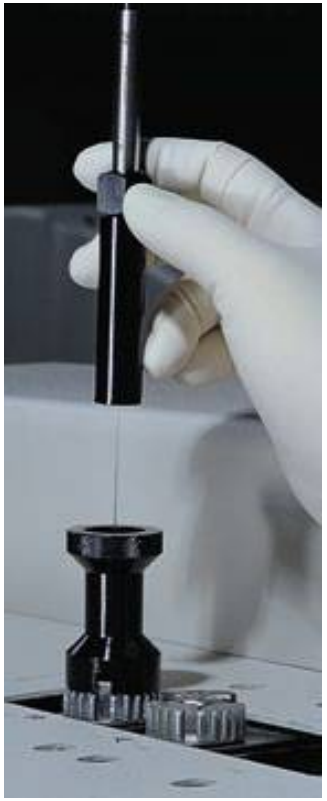
Non-destructive to sample

Reusable (100+ times)

Inexpensive

Fast

Assembled SPME fiber and holder with fiber immersed in a liquid sample.



Manual SPME holder and inlet guide.

The SPME Concept

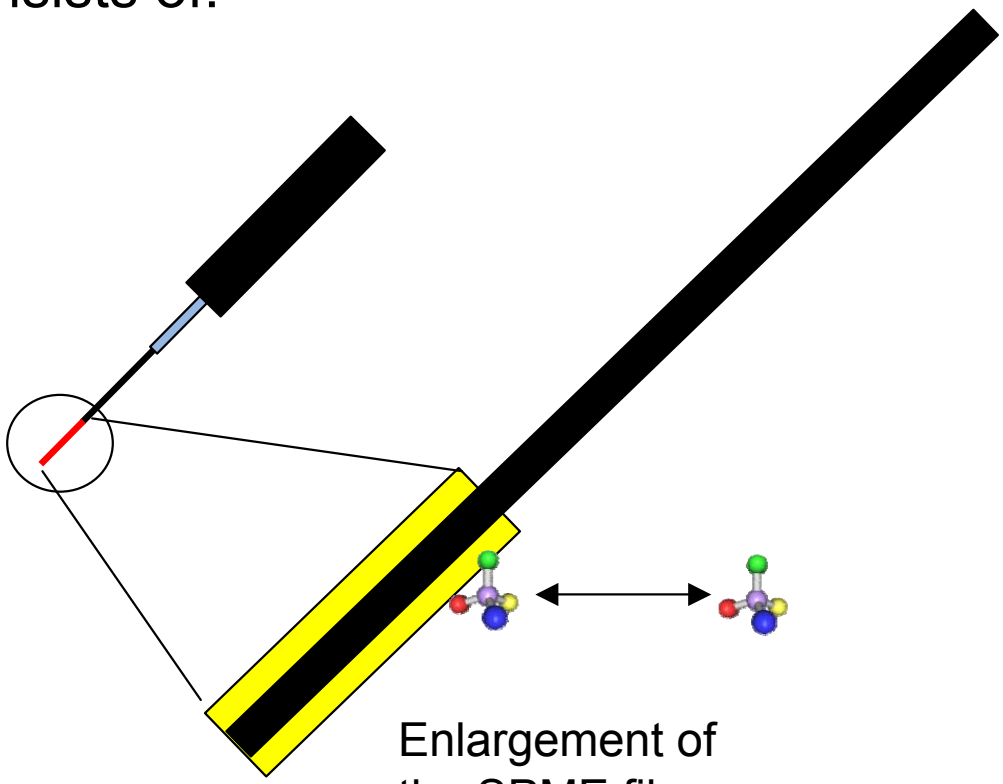
Click here for animation

SPME Fiber Coating: The Business End

An equilibrium is set up between analytes dissolved in the sample (solution or gas phase) and in the liquid coating on the fiber.

The fiber coating consists of:

- GC-type phases
- Particles



Enlargement of the SPME fiber coating

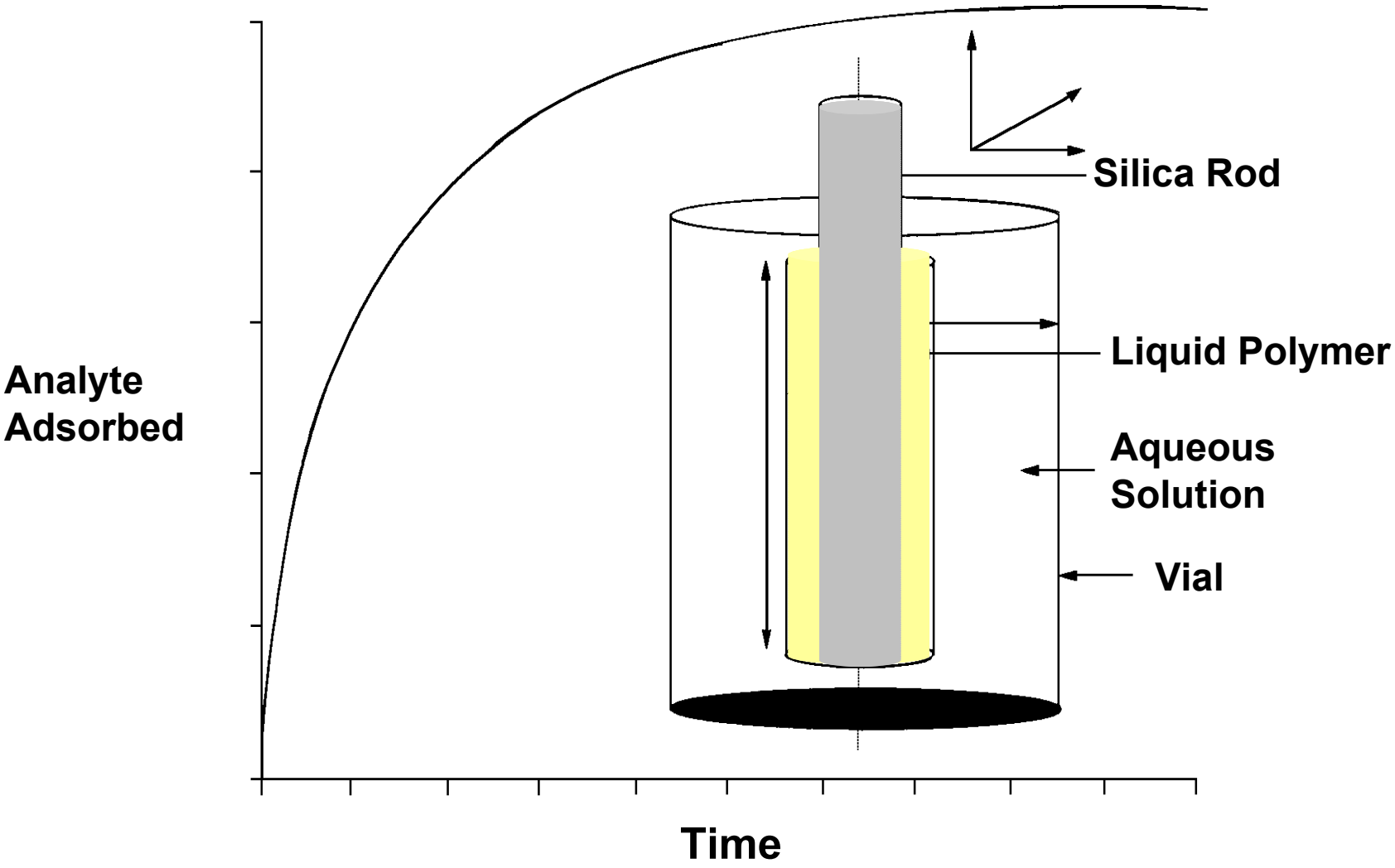
Distribution Constant

Concentration of analyte in stationary phase compared to concentration of analyte in solution:

$$K = n_s/V_1C_2^\circ$$

K = Distribution constant
n_s = Moles of analyte in stationary phase
V_1 = Volume of stationary phase
C_2° = Final analyte concentration in water

Adsorption Mechanism for SPME



Absorbent vs. Adsorbent Fibers

Absorbent-type fibers

(Film-type fibers)

Analytes are extracted by partitioning

- **Liquid phase**
- **Retains by thickness of coating**

Analytes do not compete for sites

Fibers can have high capacity

Adsorbent-type fibers

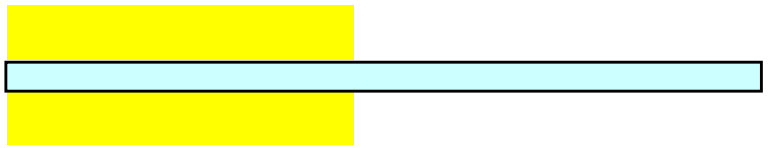
(Particle-type fibers)

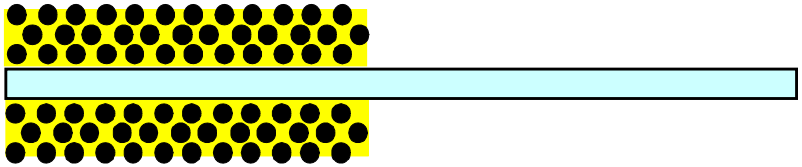
Physically traps or interacts with analytes

- **Porous particles**
- **High surface area**

Analytes may compete for sites

Fibers have limited capacity





Types of SPME Fiber Coatings

Films – Absorption:

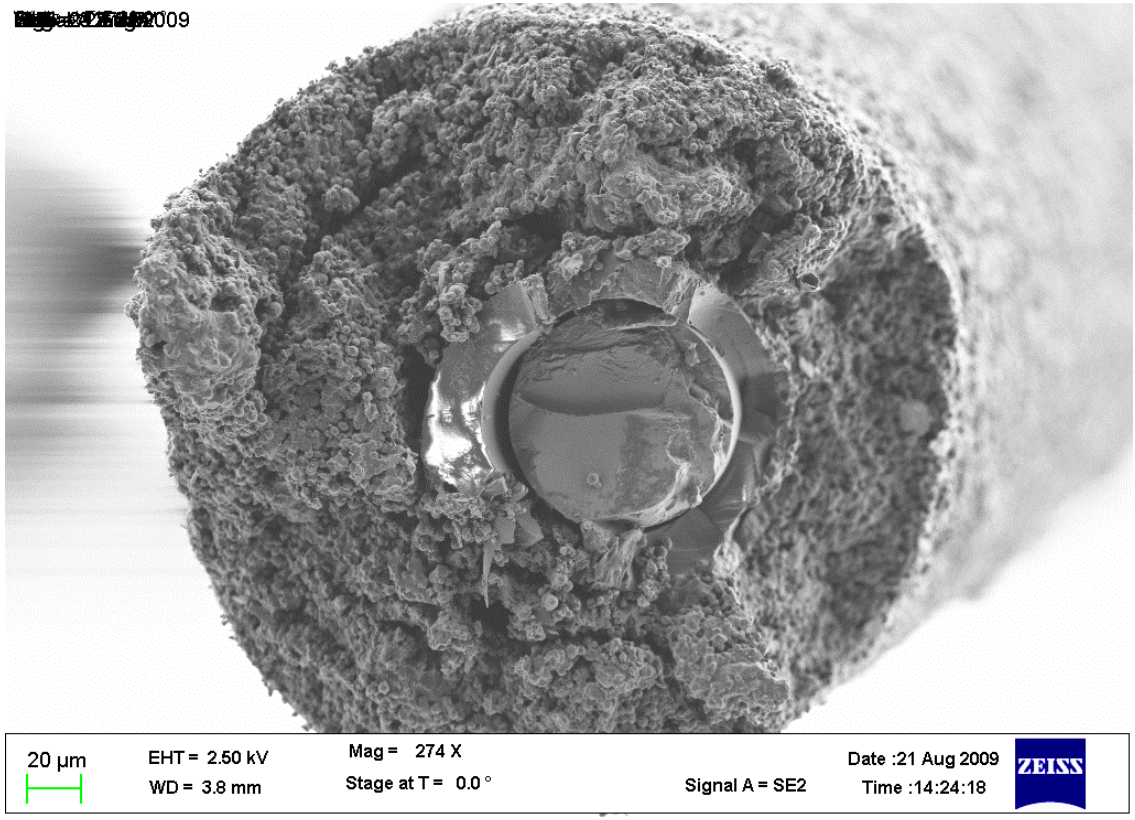
Coating	Type	Polarity
7 µm Polydimethylsiloxane (PDMS)	Absorbent	Nonpolar
30 µm PDMS	Absorbent	Nonpolar
100 µm PDMS	Absorbent	Nonpolar
85 µm Polyacrylate (PA)	Absorbent	Polar
60 µm PEG (Carbowax)	Absorbent	Polar

Particles – Adsorption:

Coating	Type	Polarity
85 µm Carboxen-PDMS	Adsorbent	Bipolar
65 µm PDMS-DVB	Adsorbent	Bipolar
55 µm/30 µm DVB/Carboxen-PDMS	Adsorbent	Bipolar
15 µm Carbopack Z-PDMS	Adsorbent	Bipolar

PDMS-DVB Fiber SEM

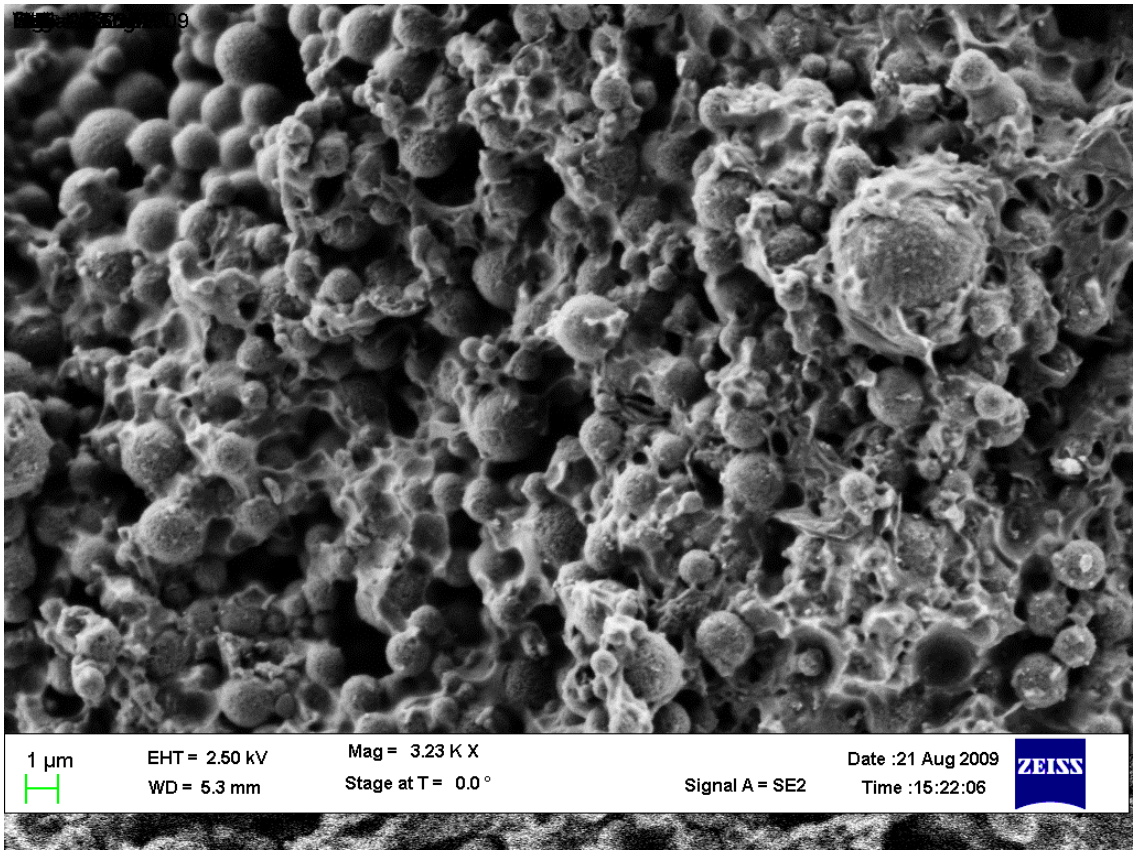
Cross section of the PDMS-DVB fiber. The center is a fused silica core, surrounded by a Stableflex core. The 3-5µm DVB particles are suspended in PDMS and layered over the cores. 275x magnification.



Photomicrograph of SPME fiber provided by Prof. Dan Armstrong, U. Texas Arlington

PDMS-Carboxen Fiber SEM

3000X magnification of the Carboxen PDMS coating. The 3-5µm Carboxen-PDMS particles are suspended in PDMS.



Photomicrograph of SPME fiber provided by Prof. Dan Armstrong, U. Texas Arlington

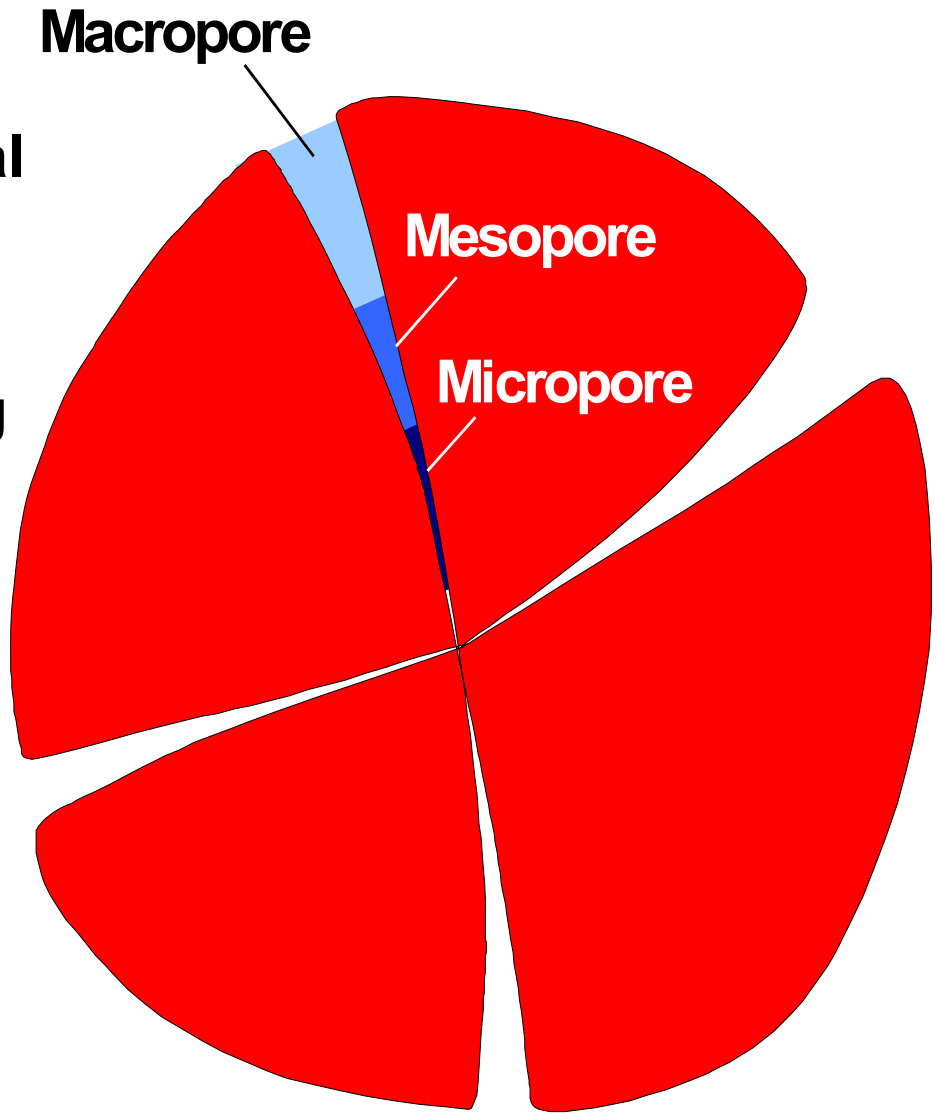
Carboxen™ Particle – Volume Contribution

Contribution of pore types to total Carboxen pore volume:

micropores (2-20Å) = 0.29 mL/g

mesopores (20-500Å) = 0.26 mL/g

macropores (>500Å) = 0.23 mL/g



97-0340

Physical Properties of Divinylbenzene and Carboxen 1006

Material	Surface Area (m^2/g)	Porosity (mL/g)* macro	meso	micro
Divinylbenzene	750	0.58	0.85	0.11
Carboxen™ 1006	720	0.23	0.26	0.29

*Macropore = >500Å
Mesopore = 20-500Å
Micropore = 2-20Å

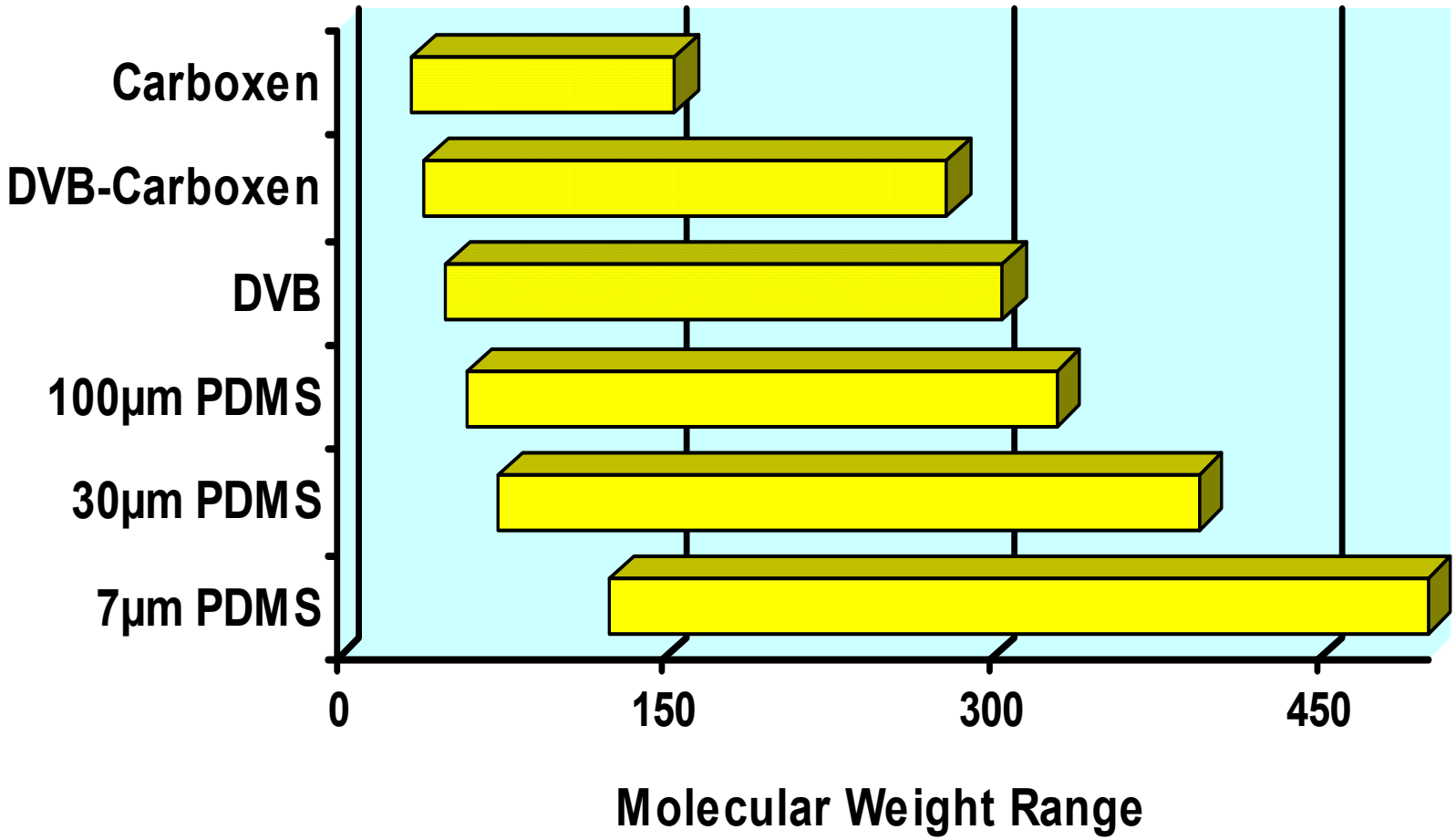
Comparison of SPME Fibers for the Extraction of Small Hydrocarbons

(Analytes at 1 ppm in air, extracted for 10 min.)

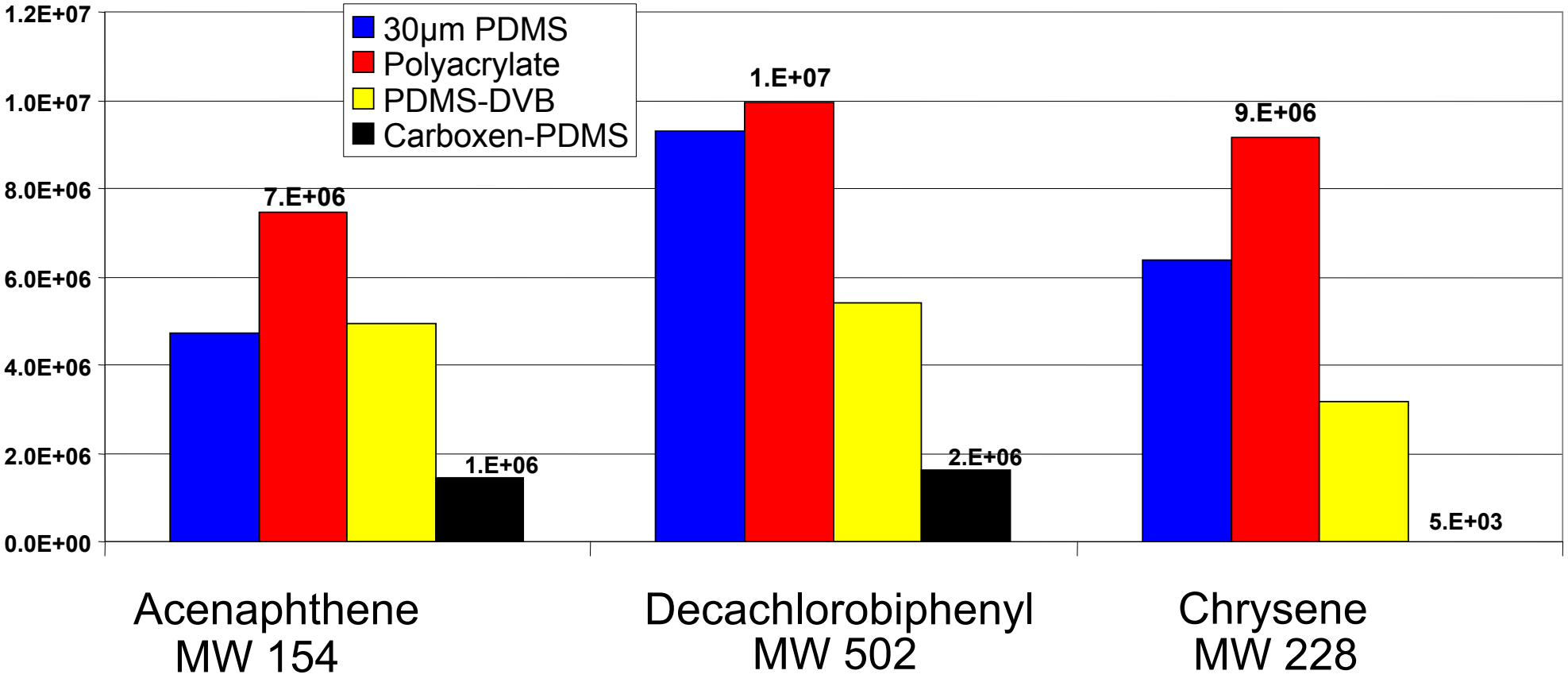
	Absorbent	Adsorbent	
Analyte	**100µm PDMS**	**PDMS/DVB**	**Carboxen/PDMS**
Ethane	**0**	**0**	**750**
Propane	**0**	**0**	**20000**
Butane	**0**	**340**	**72100**
Pentane	**230**	**2150**	**108000**
Hexane	**460**	**9280**	**105000**

(Absolute area responses)

Molecular Weight Range for SPME Fibers



Area Response vs. Fiber Type



Effects of Fiber Polarity & Coating Thickness

Fiber Polarity

- Analyte selectivity
- Better recovery of polar analytes
- PEG
- Polyacrylate

Coating Thickness

- Analyte selectivity
- Extraction time
- Sample capacity
- Desorption time and carryover

Effects of Phase Coating Thickness of PDMS on Analyte Recovery Relative to Chrysene*

Analyte	%Relative Recovery		
	100µm	30µm	7µm
Benzene	2	1	<1
Toluene	5	1	<1
Naphthalene	13	4	1
Phenanthrene	37	27	16
Anthracene	49	38	32
Pyrene	69	54	47
Benzo(a)anthracene	105	91	96
Chrysene	100	100	100
Benzo(a)pyrene	119	127	131
Indeno(1,2,3-cd)pyrene	61	140	148
Benzo(g,h,i)perylene	61	117	122

*Absolute response of chrysene set to 100%

Factors Affecting Extraction Recovery

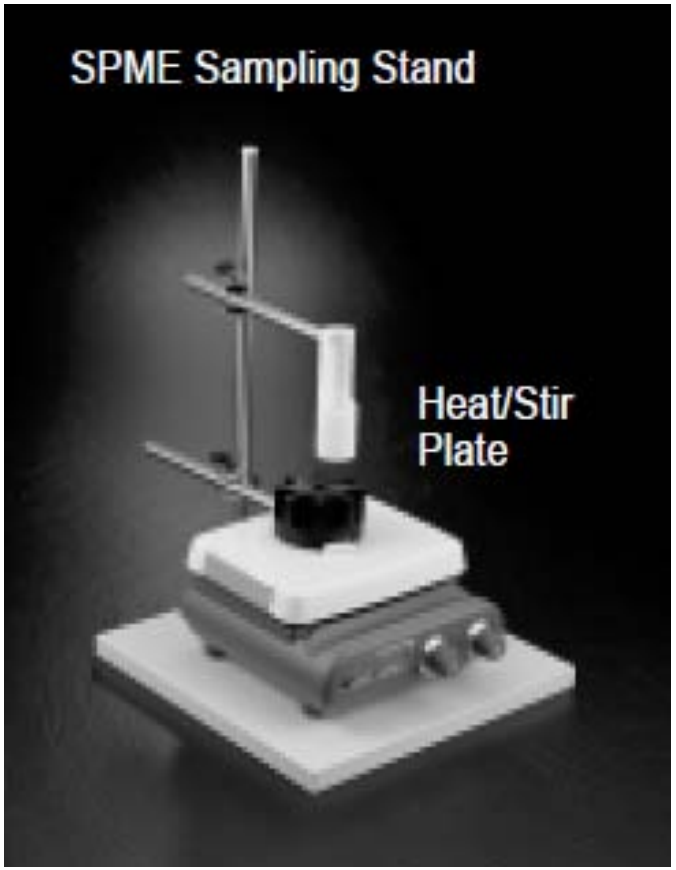
Salts and pH

Headspace vs. direct extraction

Inlet liner volume

Stirring (sample) & agitation (fiber)

- Increases precision
- Reduces time to reach equilibrium
- Must be consistent for all analyses
- Required for analytes with high distribution constants
- Sonication may increase temperature



Effects of Salt and pH

Salt usually increases analyte uptake

Use 25-30% NaCl to salt-out samples

Salt is not necessary for large non-polar analytes, such as PAHs and large hydrocarbons, and may reduce recovery

Lower pH to extract acidic compounds

Raise pH to extract basic compounds

Beware of stability of analytes at different pH levels

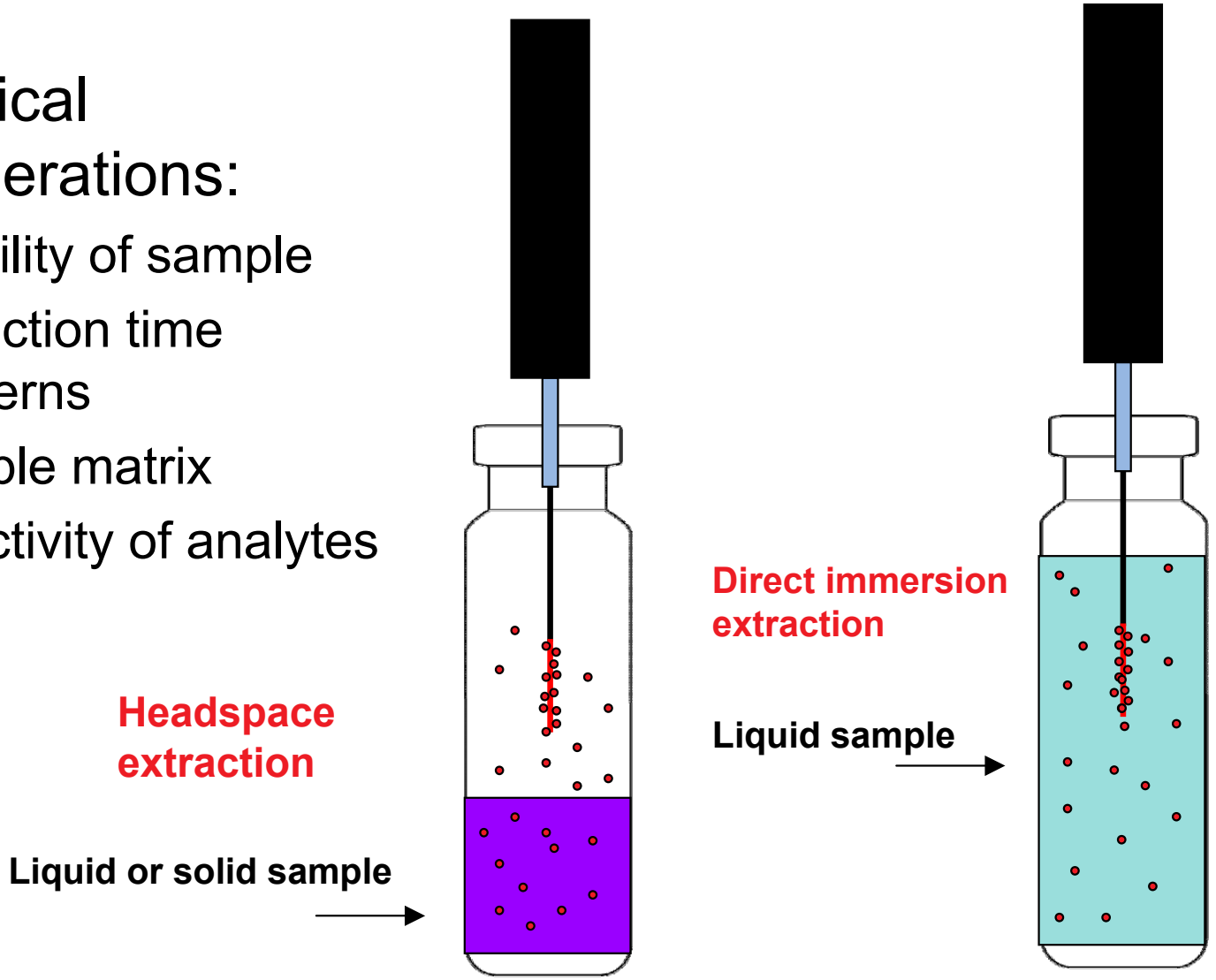
The Effect of Salt and pH on Extraction of Phenols by SPME

	No Salt Neutral	No Salt pH = 2	Salt Neutral	Salt pH = 2
Phenol	810	1003	6425	6150
Methylphenol	761	882	5485	7434
2-Nitrophenol	422	474	311	2315
2,4-Dimethylphenol	1344	1476	15000	20710
2,4-Dichlorophenol	5396	8138	19803	61664
2,4,5-Trichlorophenol	3115	11097	24270	96333
2,4-Dinitrophenol	0	11	765	1182
4-Nitrophenol	626	730	6536	11438
2,3,4,6-Tetrachlorophenol	3108	27683	33938	70440
2-Methyl-4,6-dinitrophenol	55	47	920	1685
Pentachlorophenol	2305	40582	22056	143905

Headspace vs. Direct Immersion

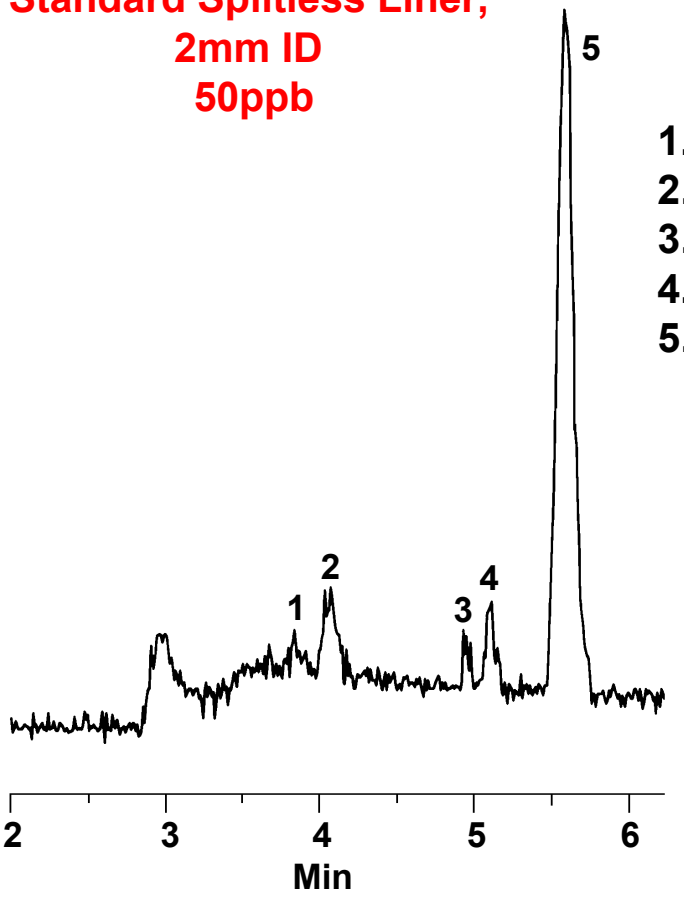
Analytical considerations:

- Volatility of sample
- Extraction time concerns
- Sample matrix
- Selectivity of analytes



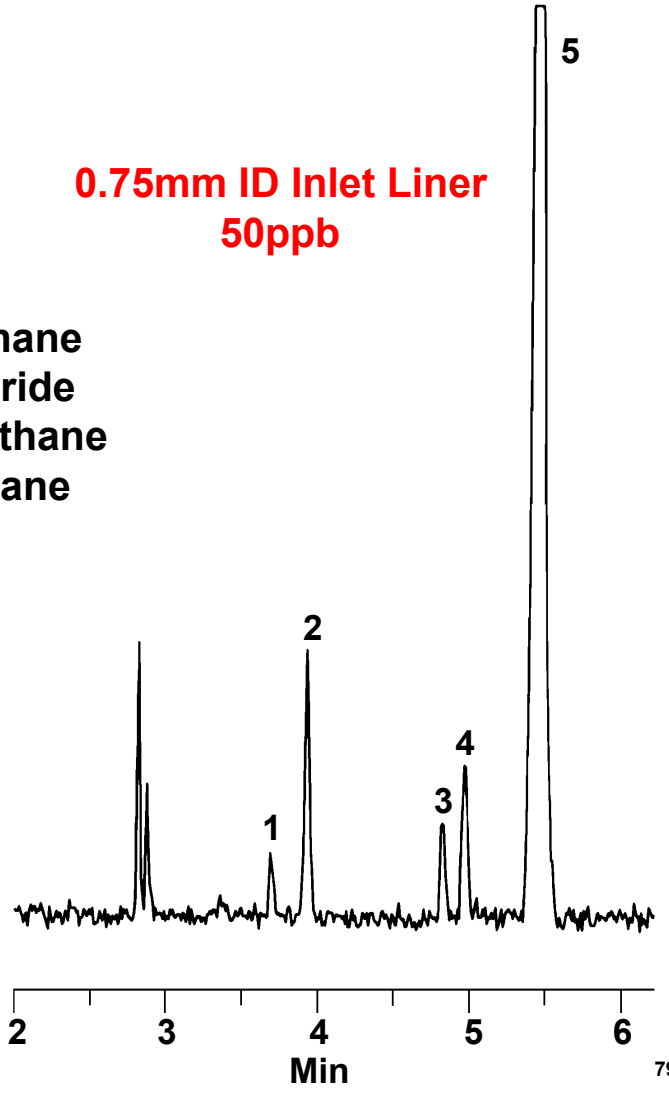
Inlet Liner Volume: Comparison for Analysis of Gaseous VOCs by SPME

Standard Splitless Liner, 2mm ID 50ppb



1. Chlormethane
2. Vinyl chloride
3. Bromomethane
4. Chloroethane
5. Freon 11

0.75mm ID Inlet Liner 50ppb



794-0050, 0051

SPME Automation

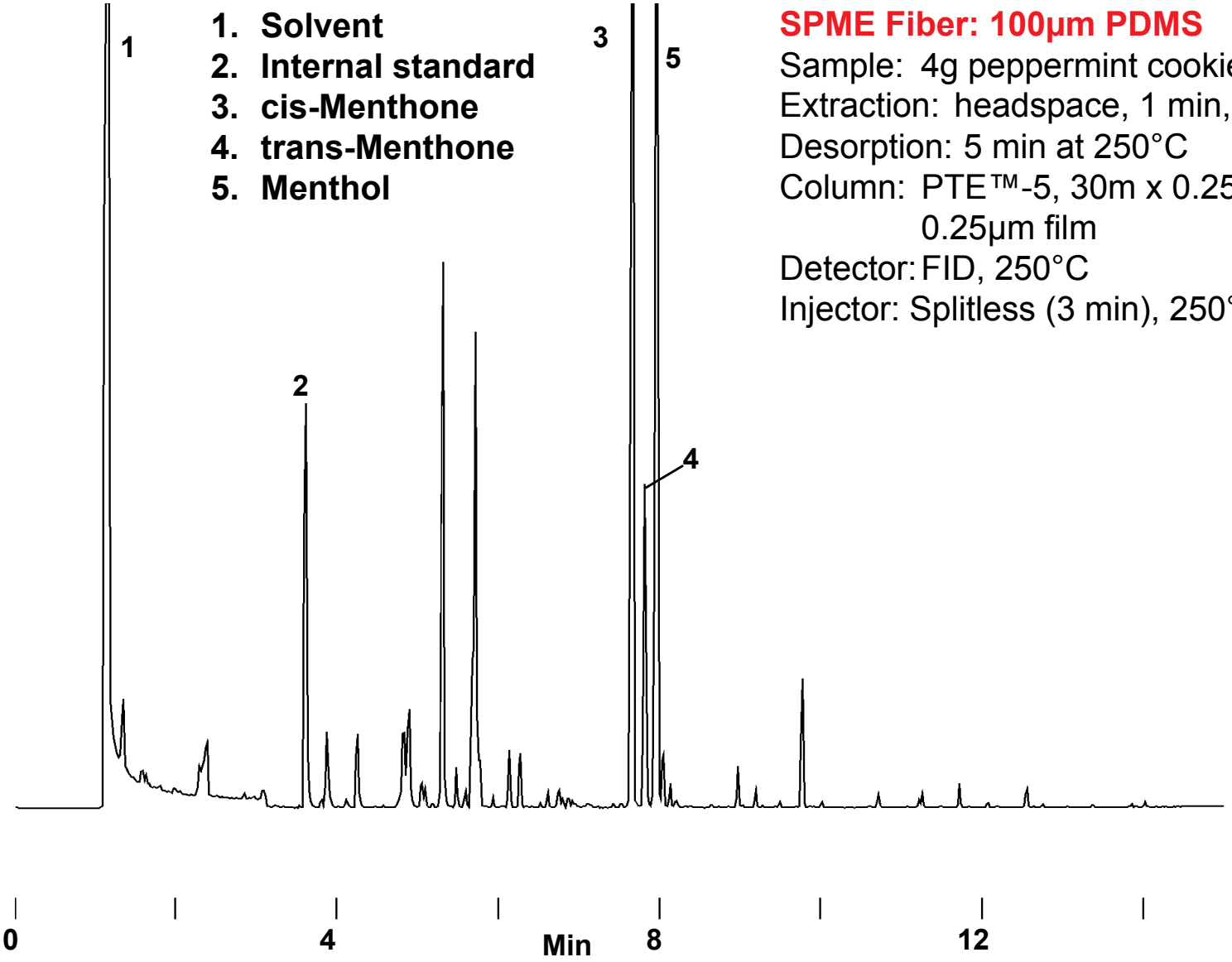
Compatible with common GC autosamplers (Gerstel MPS, CTC Combi PAL, *etc*.)

Improves reproducibility by automating important variables:

- Heating
- Agitation
- Equilibration time

SPME automation video (~2 mins.)

Peppermint Oil in Chocolate Cookie Bar



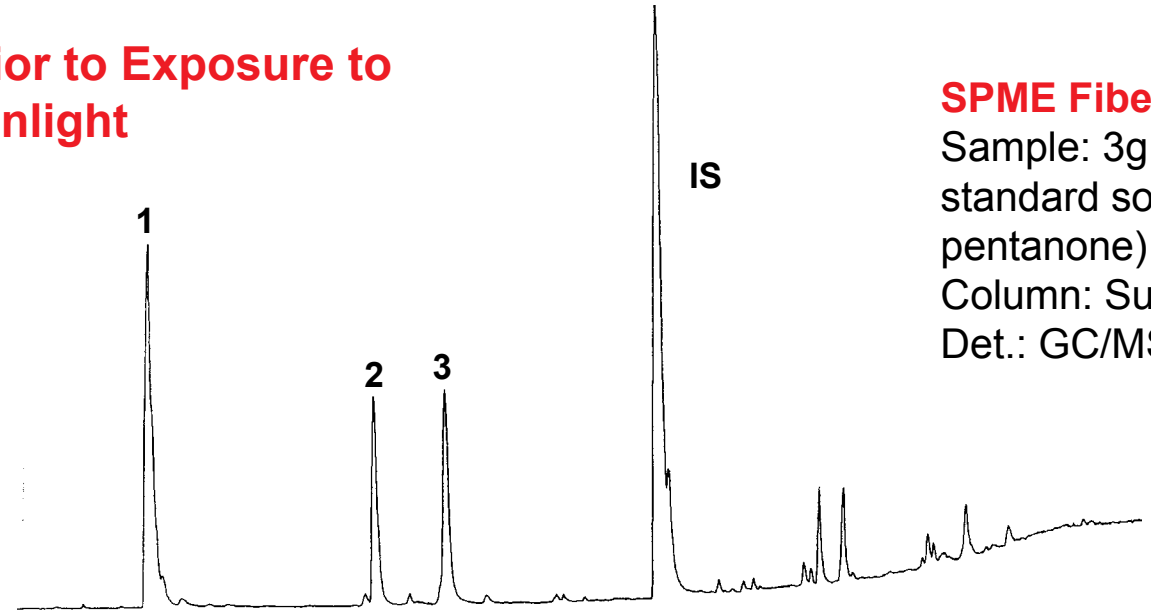
1. **Solvent**
2. **Internal standard**
3. **cis-Menthone**
4. **trans-Menthone**
5. **Menthol**

SPME Fiber: 100µm PDMS
Sample: 4g peppermint cookie bar
Extraction: headspace, 1 min, 45°C
Desorption: 5 min at 250°C
Column: PTE™-5, 30m x 0.25mm ID, 0.25µm film
Detector: FID, 250°C
Injector: Splitless (3 min), 250°C

794-0862

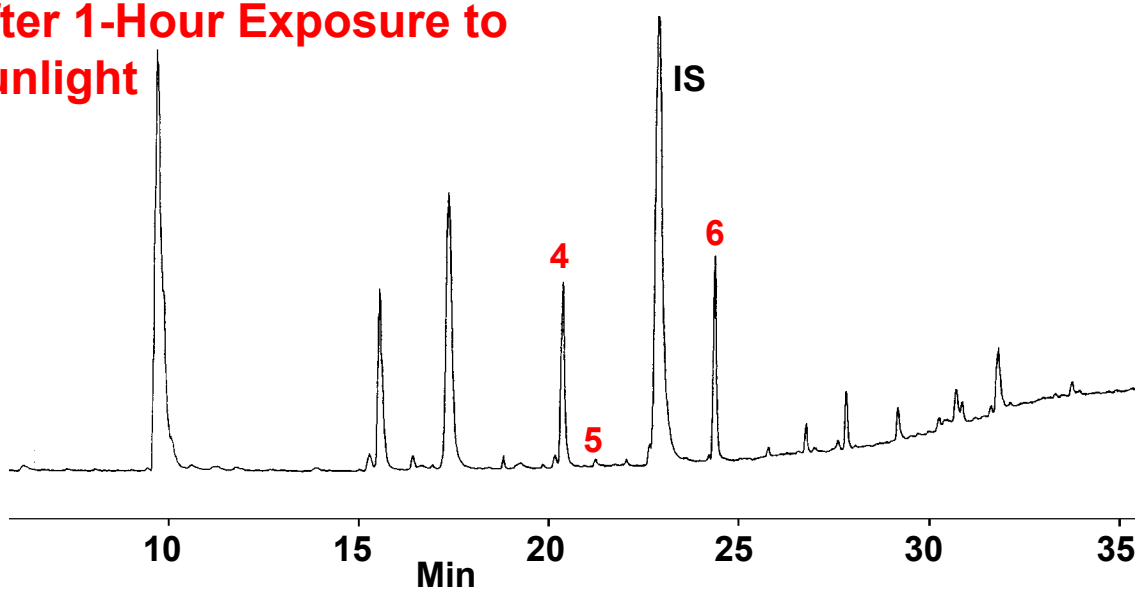
Milk Sample Off-Flavors by SPME-GC/MS

Prior to Exposure to Sunlight



SPME Fiber: 75 µm PDMS/Carboxen
Sample: 3g of 2% milk + 10µL internal standard solution, (20µg/mL 4-methyl-2-pentanone) (9mL GC vial)
Column: Supel-Q™ PLOT, 30m x 0.32mm ID
Det.: GC/MS ion trap, m/z = 33-300

After 1-Hour Exposure to Sunlight



1. Acetone
2. 2-Butanone
3. 3-Methylpentane
4. Pentanal
5. Dimethyldisulfide
6. Hexanal

IS. 4-Methyl-2-pentanone

Chromatogram provided by Ray Marsili, Dean Foods Technical Center, Rockford, IL, USA.

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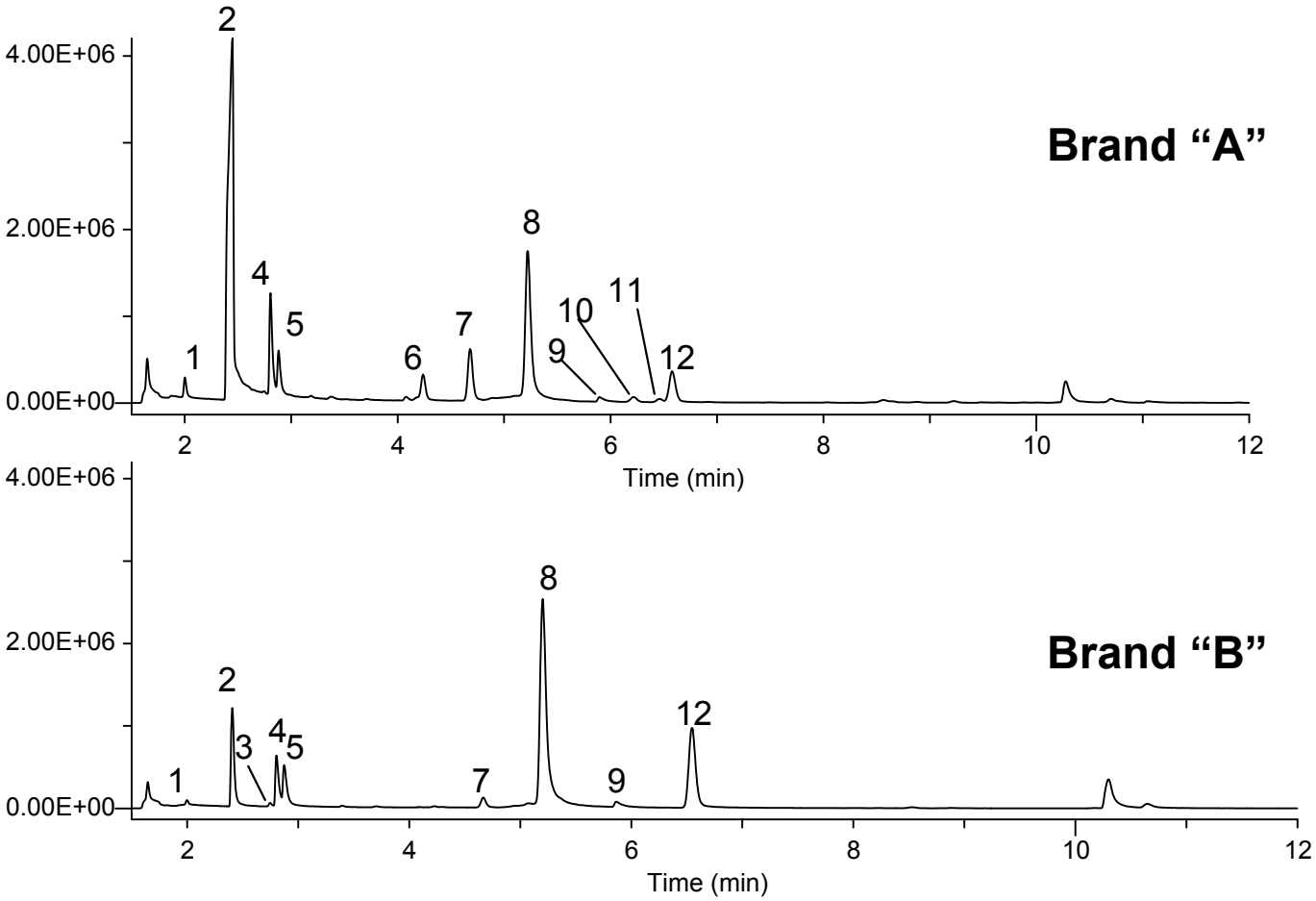
98-0385

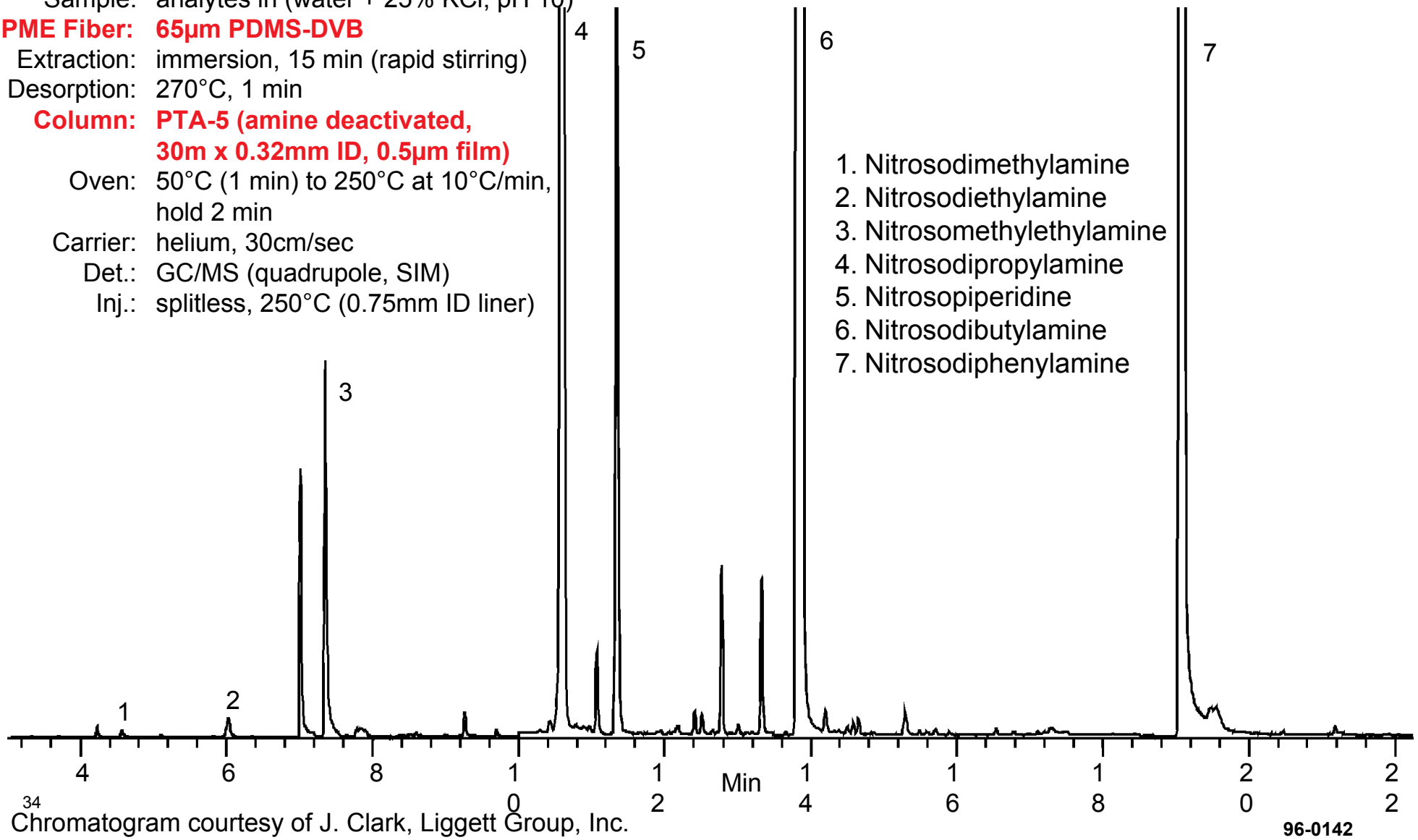
Residual Solvents in Commercial Ibuprofen



1. Acetaldehyde
2. Ethanol
3. Acetonitrile
4. Acetone
5. 2-Propanol
6. 2-Methylpentane
7. 3-Methyl pentane
8. Hexane
9. Ethyl acetate
10. 2,2-Dimethylpentane
11. 2,4-Dimethylpentane
12. Methylcyclopentane

10ppb Nitrosamines in Water: SPME-GC/MS

Sample: analytes in (water + 25% KCl, pH 10)
SPME Fiber: 65µm PDMS-DVB
Extraction: immersion, 15 min (rapid stirring)
Desorption: 270°C, 1 min
Column: PTA-5 (amine deactivated, 30m x 0.32mm ID, 0.5µm film)
Oven: 50°C (1 min) to 250°C at 10°C/min, hold 2 min
Carrier: helium, 30cm/sec
Det.: GC/MS (quadrupole, SIM)
Inj.: splitless, 250°C (0.75mm ID liner)



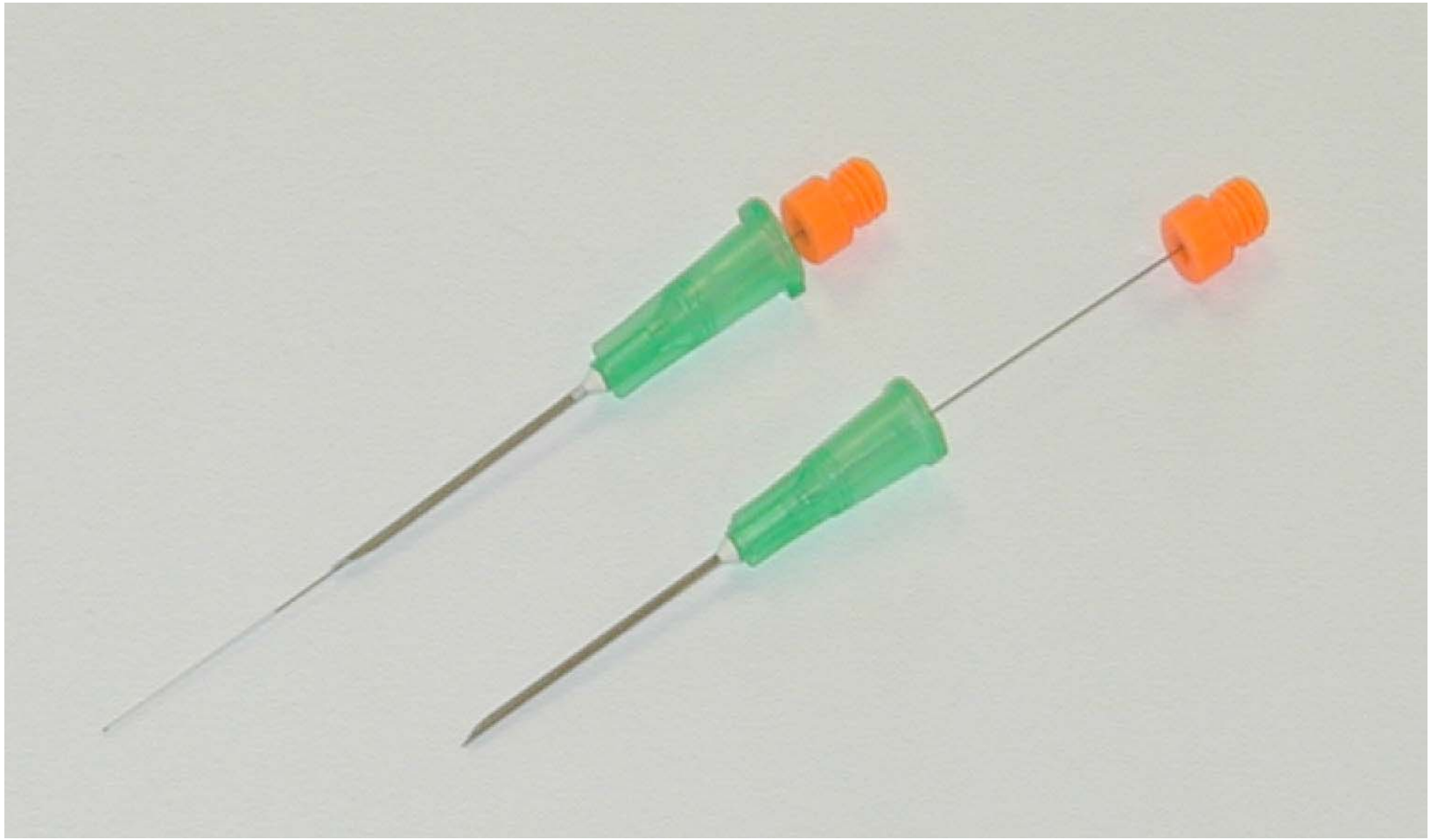
Chromatogram courtesy of J. Clark, Liggett Group, Inc.

96-0142

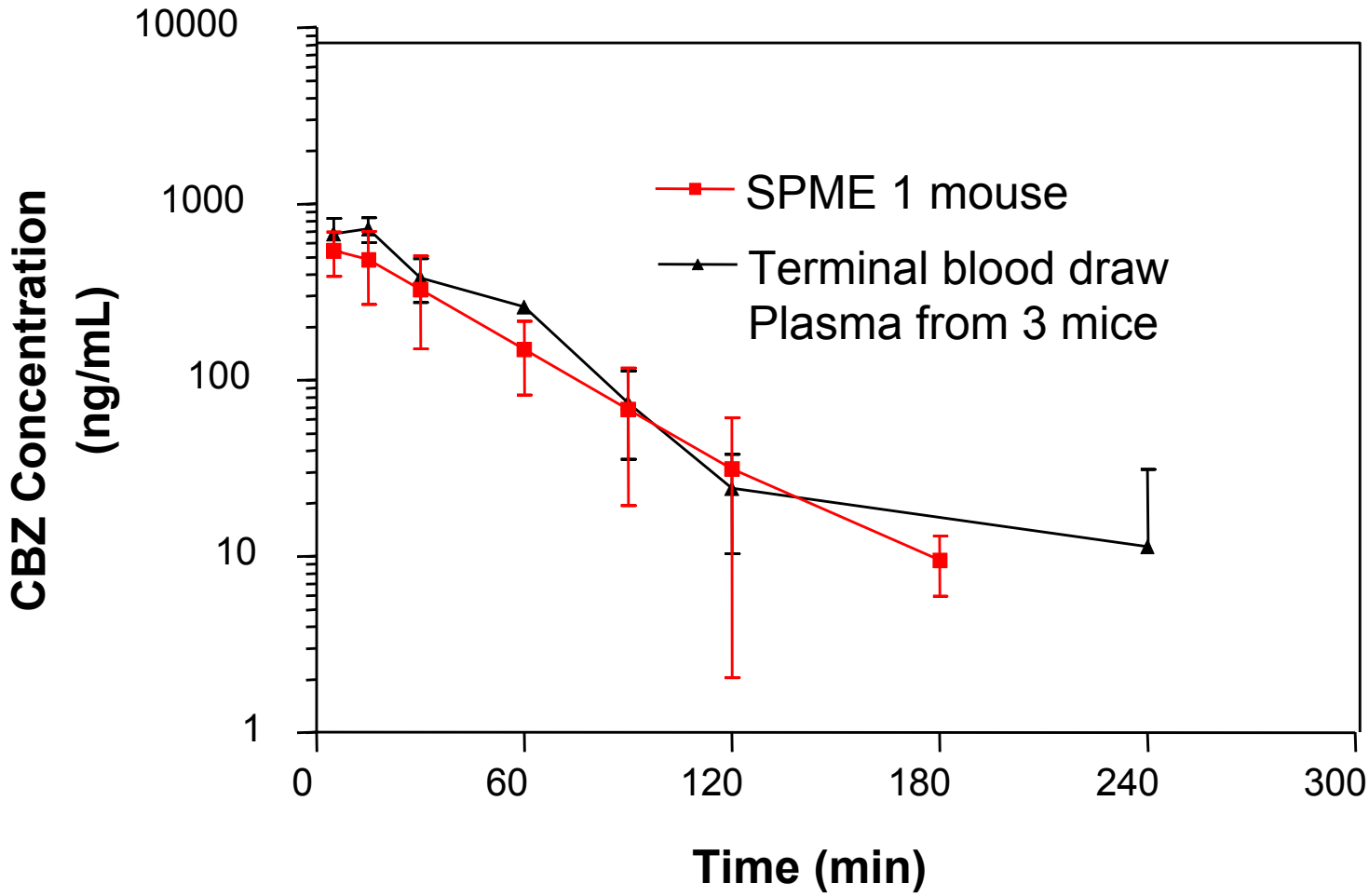
SIGMA-ALDRICH®

New Development: Biocompatible Fiber Pipette Tips for Solvent Extraction

Single Use Biocompatible Fiber Probes for *in vivo* Analysis

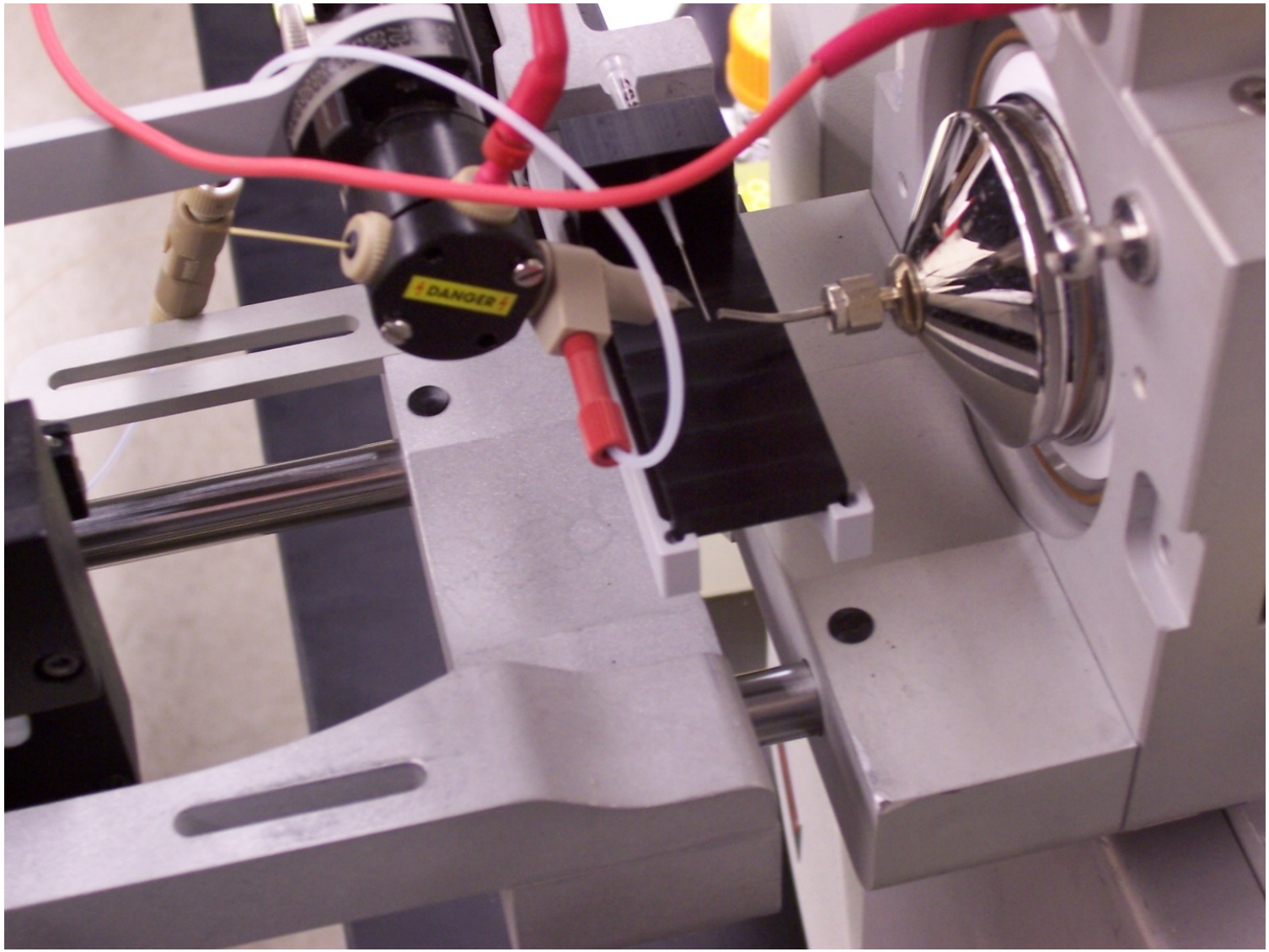


Comparison of SPME *in-vivo* PK Study of Carbamazepine from Mice Whole Blood to Extracts of Plasma Removed from Mice



Slide Courtesy of Ines de Lannoy-NoAb BioDiscoveries

SPME fiber Holder with Automated DESI-1D Source



Courtesy of Joseph Kennedy of Prosolia

Solid Phase Microextraction (SPME) Products

Fibers

Holders

- **Manual**
- **For autosamplers**

Accessories

Instructions

Applications on CD

sigma-aldrich.com/spme

Sample Prep Innovations

- Solid Phase Microextraction (SPME)
- **High specificity SPE (SupelMIPs)**
- Dispersive SPE
- Silver Ion SPE for FAMEs
- Carbonaceous adsorbent
- Flash chromatography

Users are...
- **Analytical chemists (LC, LC-MS, GC...)**

Interested in...
- **Very selective extraction**
- **Analysis at extremely low concentrations (ppb, ppt)**
- **Increasing specificity of sample prep from complex matrixes**

Users can expect...
- **More rigorous washing to remove matrix**
- **Detect at lower levels**

High Specificity Sample Prep

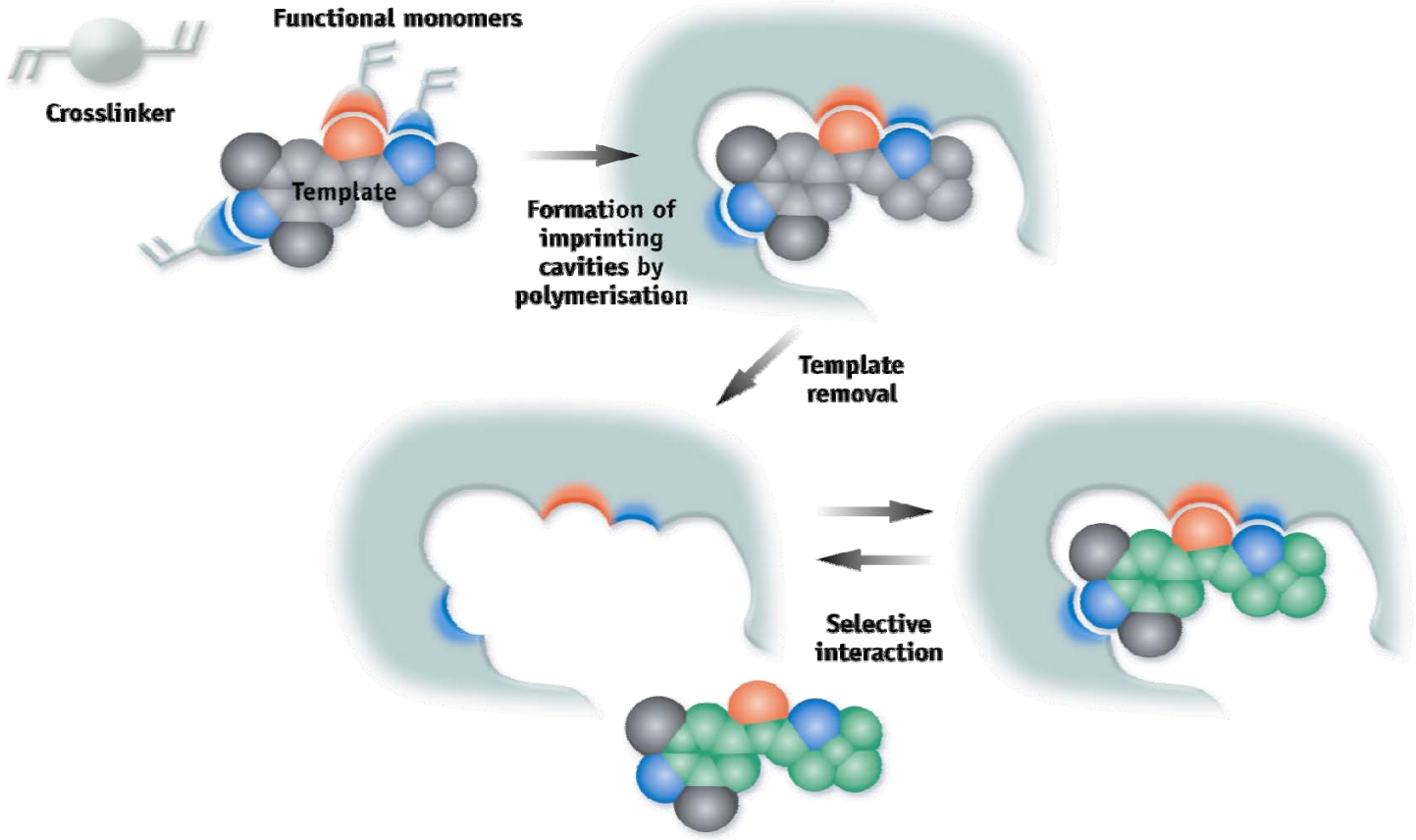
The specific innovation we will describe:

SupelMIP Molecularly Imprinted Polymers

- SPE tubes
- 96-well plates

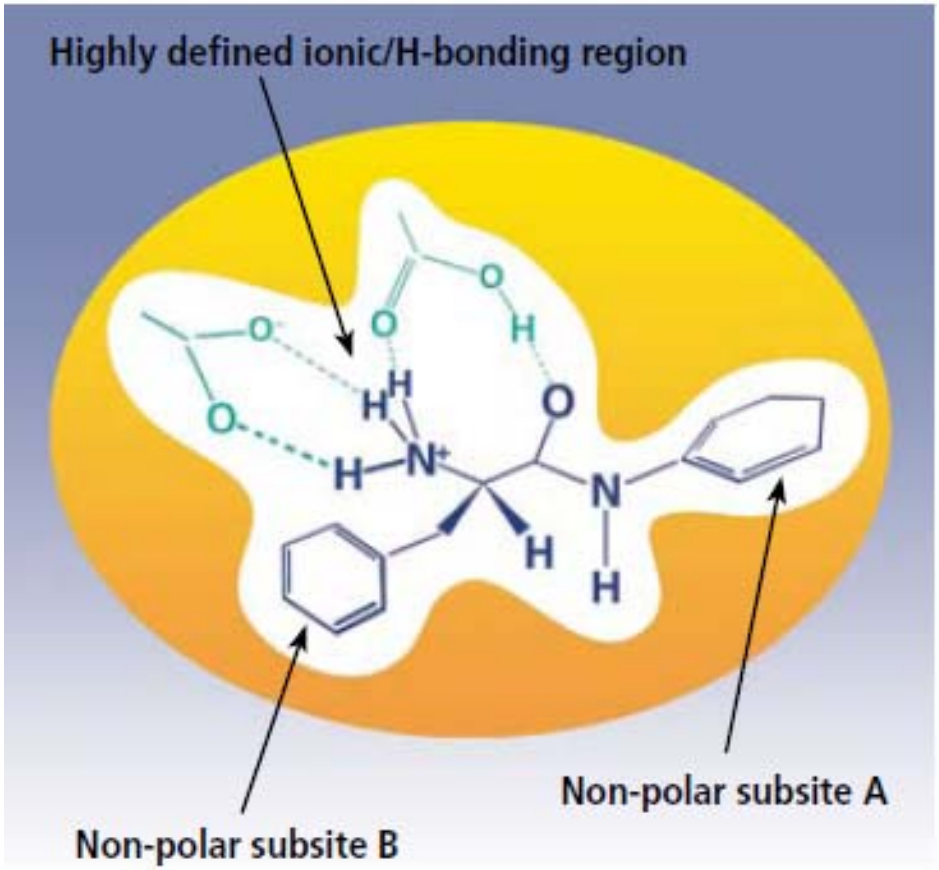
The Molecular Imprinting Process

Molecularly imprinted polymers (MIPs) are polymers that have been prepared by polymerizing either pre-formed or self-assembled monomer-template complexes together with a cross-linking monomer. After removal of the template molecule, a polymer with binding sites for the template is obtained.



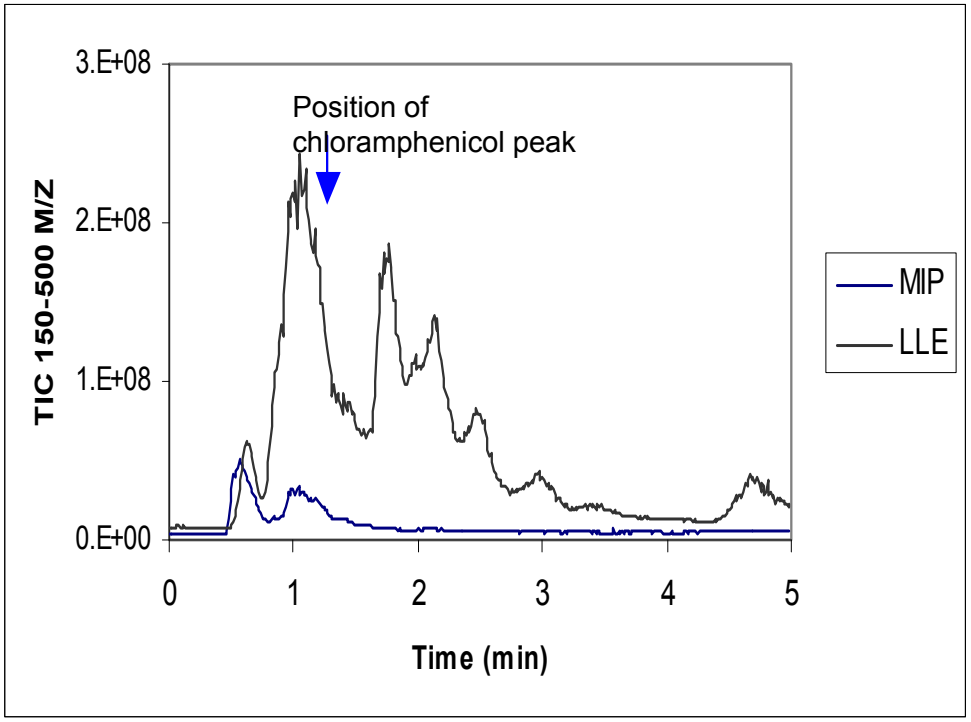
The MIP Binding Site

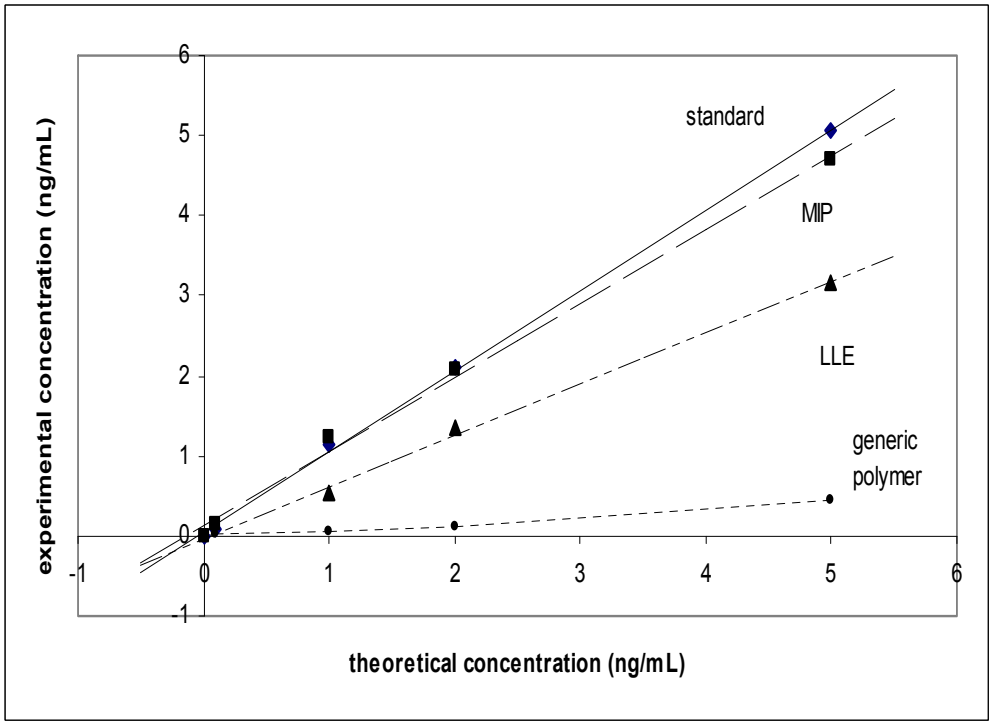
Graphical representation of the MIP binding site, which contains a cavity of the right size and attractive molecular features that can bind to the target molecule(s).



SupelMIP Chloramphenicol: Analysis in Honey

Chloramphenicol is an antibiotic that is monitored in honey.



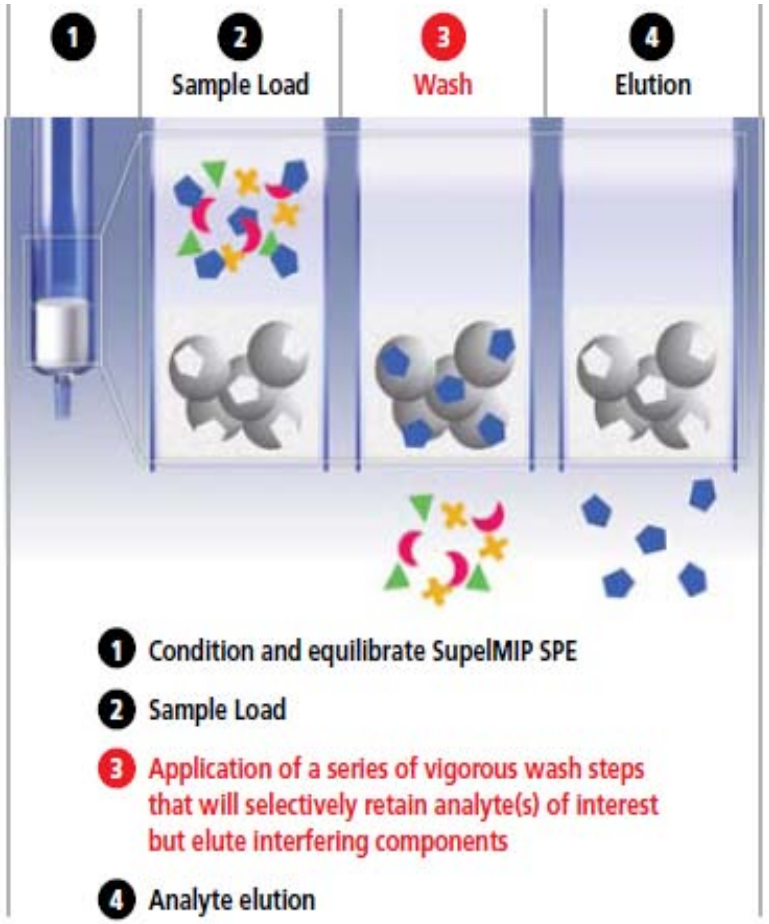


Background from honey sample cleaned by SupelMIP SPE and LLE for Chloramphenicol analysis

Comparison of ion suppression effect between different clean-up methods for honey. Samples were post-spiked with CAP prior to analysis.

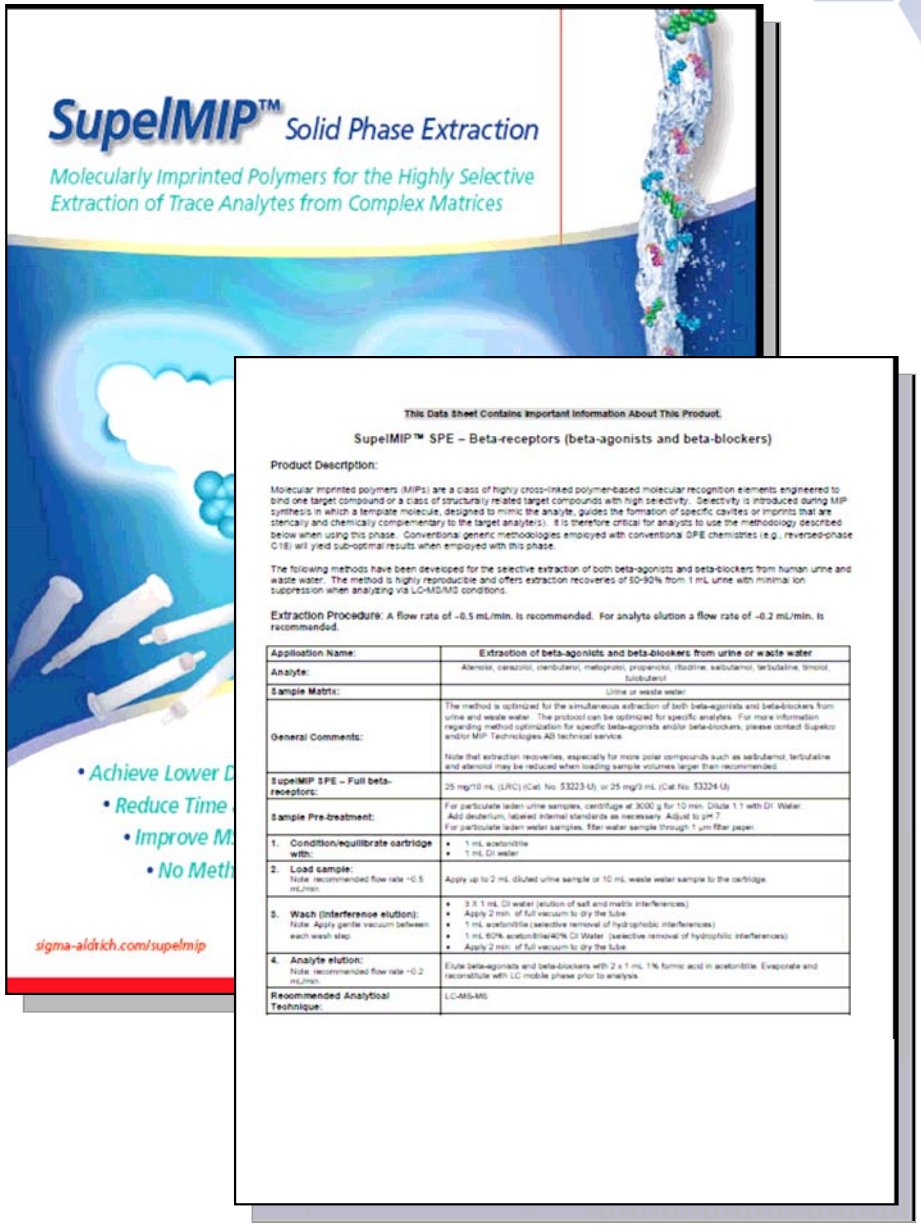
Overview of a Typical SupelMIP SPE Procedure

- Very simple methods.
- Full protocols are included with each MIP product.
- Protocols may require optimization depending on the sample matrix.



SupelMIP Products

- **PAHs** in edible oils
- **Nitroimidazoles** in milk, eggs and other foods
- Nonsteroidal anti-inflammatory drugs (**NSAIDS**) in wastewater and other matrices
- **Fluoroquinolones** in bovine kidney, honey and milk
- **Amphetamines** and related compounds in urine
- **Chloramphenicol** in plasma, urine, milk, honey and shrimp
- **NNAL** - nitroso compound in urine
- **TSNAs** - tobacco specific nitrosamines in urine and tobacco
- **β-agonists** and **β-blockers** in tissue, urine and wastewater
- **Clenbuterol** in urine
- **Triazines** in water
- **Riboflavin** in milk



sigma-aldrich.com/supelmip

SIGMA-ALDRICH®

Topics: Sample Prep Innovations

- SPME (solid phase microextraction)
- High specificity SPE (SupelMIP)
- **Dispersive SPE**
- Silver Ion SPE for FAMEs
- Carbonaceous adsorbent
- Flash chromatography

Users are...
- **Food safety analysts**

Interested in...
- **Multi-residue pesticide analysis in food and agricultural products**

Users can expect...
- **Quick, easy, inexpensive extraction method**

Dispersive SPE (dSPE or QuEChERS) Multiresidue Pesticide Method

Multi-residue (100's) pesticide analysis

Retains/removes key interferences in food samples

Analytes are un-retained

Quick (~30 min./6 samples)

Easy (no laborious steps)

Cheap

Effective (wide scope, low consumption)

Rugged (minimal sources of errors)

Safe (solvents and techniques)

Dispersive SPE Procedure

Procedure:

1. Food initially extracted with aq. miscible solvent (e.g. ACN)
2. High amounts of salts (NaCl, Mg-sulfate) and buffering agents added to induce phase separation and stabilize acid/base labile pesticides
3. Shake/centrifuge. Isolate aliquot of sup for SPE clean-up.
4. Transfer supernatant to centrifuge tube. Add bulk SPE phase(s) and salts. Shake/vortex. Centrifuge and analyze supernatant.

Standard dSPE product line configured for:

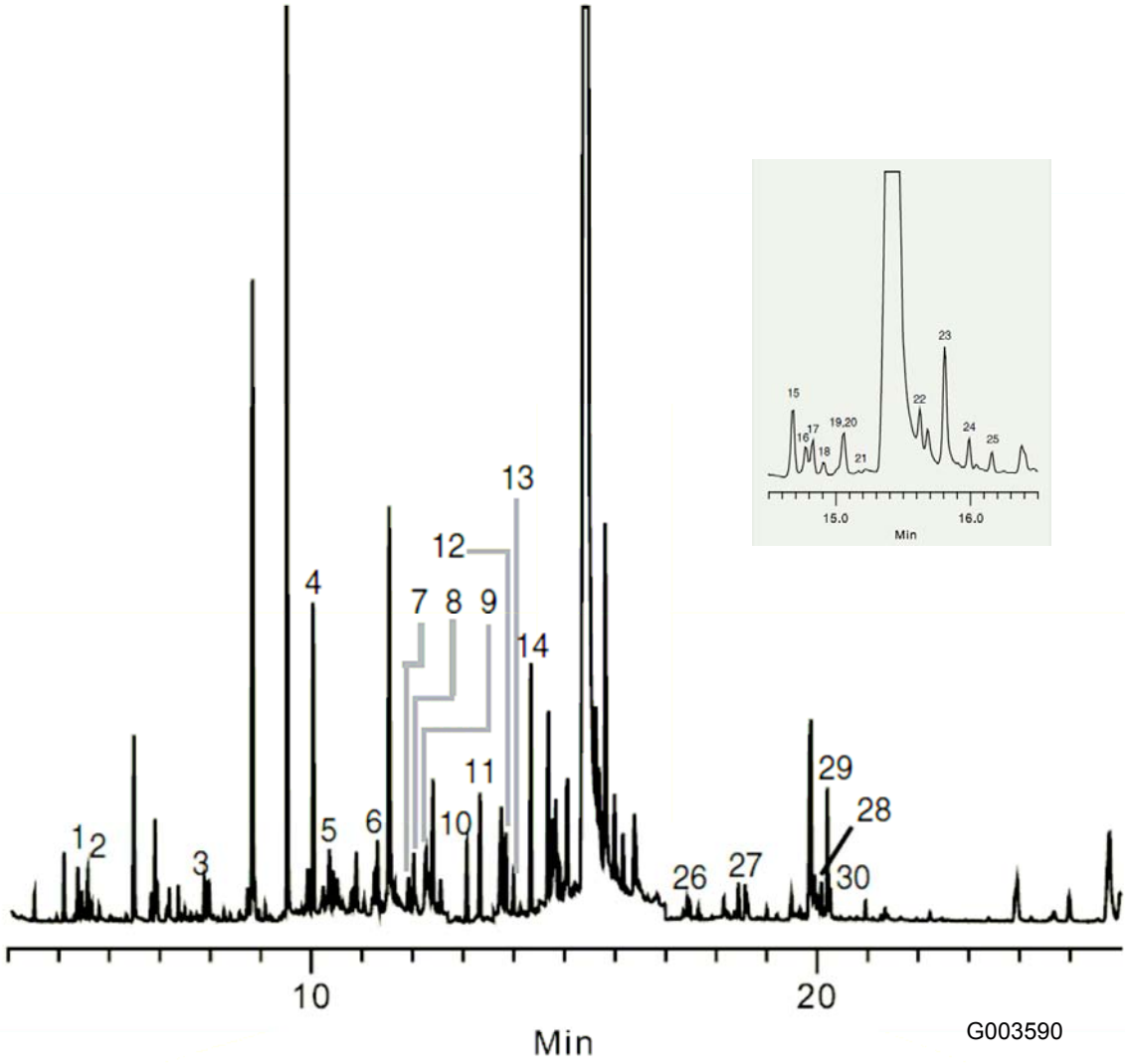
- CEN Standard Method EN – 15662
- AOAC Method 2007.01

Full details of the simple protocol is included with the product.

GC-MS of Pesticides from Oranges Following Extraction with dSPE

column: SLB-5ms, 30 m x 0.25 mm I.D, 0.25 µm (28471-U)
oven: 100 °C (1 min.), 10 °C/min. to 300 °C (5 min.)
inj.: 250 °C
MSD interface: 300 °C
scan range: selected ion monitoring (SIM), 7 monitoring groups used
carrier gas: helium, 1 mL/min constant
injection: 1 µL, pulsed (20 psi until 0.20 min.), splitless (1.0 min.)
liner: 4 mm I.D., single taper

1. Methamidiphos
2. Dichlorvos
3. Acephate
4. Propoxur
5. Ethoprophos (I.S.)
6. Hexachlorobenzene
7. γ-BHC
8. Diazinon
9. Chlorothalonil
10. Methyl chlorpyrifos
11. Carbaryl
12. Dichlofluanid
13. Chlorpyrifos
14. p-Dichlorobenzophenone
15. Cyprodinil
16. Pencanazole
17. Tolylfluanid
18. Heptachlor epoxide
19. Captan
20. Thiabendazole
21. Folpet
22. cis-Chlordane
23. Imazalil
24. 4,4'-DDE
25. Dieldrin
26. Endosulfan sulfate
27. Dicofol
28. cis-Permethrin
29. trans-Permethrin
30. Coumaphos



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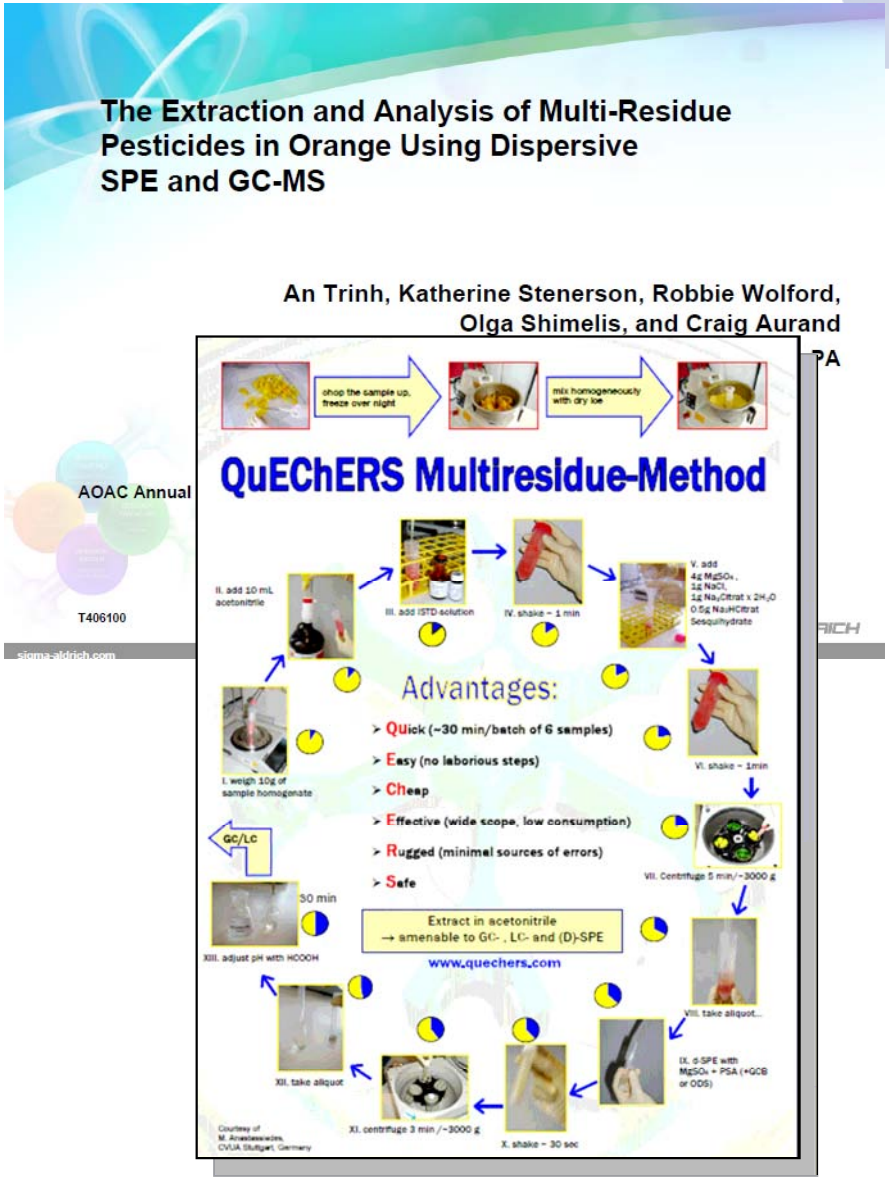
SIGMA-ALDRICH®

Dispersive SPE Products

Centrifuge tubes containing pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today

Product #	Description
55227-U	Dispersive SPE (dSPE) Citrate Extraction Tube, pk of 50
55237-U	Dispersive SPE (dSPE) Citrate/Sodium Bicarbonate Extraction Tube, pk of 50
55234-U	Dispersive SPE (dSPE) $MgSO_4$ Extraction Tube, pk of 50
55228-U	Dispersive SPE (dSPE) PSA SPE Clean Up Tube 1, pk of 50
55229-U	Dispersive SPE (dSPE) PSA/C18 SPE Clean Up Tube 1, pk of 50
55230-U	Dispersive SPE (dSPE) PSA/ENVI-Carb SPE Clean Up Tube 1, pk of 50
55233-U	Dispersive SPE (dSPE) PSA/ENVI-Carb SPE Clean Up Tube 2, pk of 50



Also available:

- Sample packs
- Custom tubes and packing materials

sigma-aldrich.com/spe

Topics: Sample Prep Innovations

- SPME (solid phase microextraction)
- High specificity SPE (SupelMIP)
- Dispersive SPE
- **Silver Ion SPE for FAMEs (Discovery Ag-Ion)**
- Carbonaceous adsorbents
- Flash chromatography

Users are...
- **Food analysts**

Interested in...
- **Measuring cis/trans fats or degree of unsaturation**

Users can expect...
- **To fractionate FAME samples prior to GC analysis, simplifying analytical chromatography and improving method accuracy**

Discovery Ag-Ion SPE for FAME Fractionation

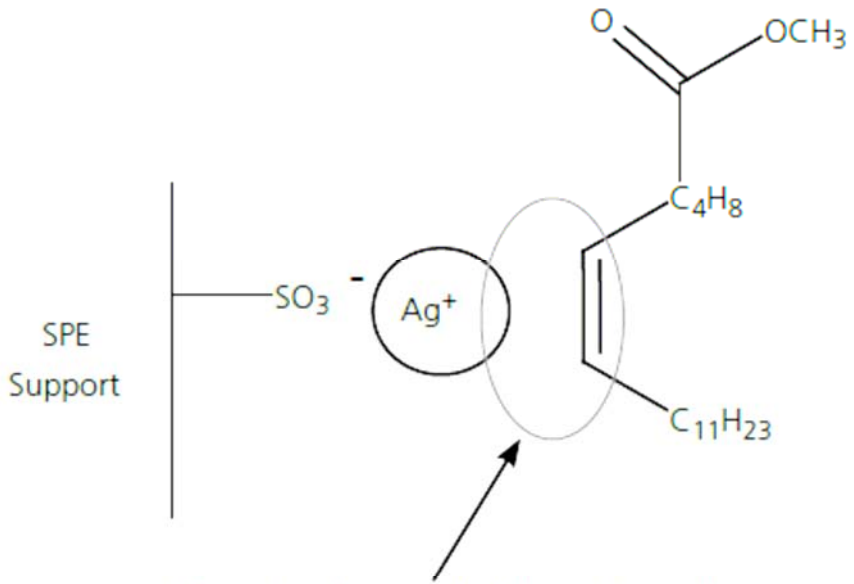
Silver ion anchored onto SCX SPE support

Ag+ forms a charge transfer complex with unsaturated FAME double bond

- Ag+ = electron acceptor; double bond = electron donor

Cis configuration offers greater steric accessibility = stronger retention

Strength of interaction increases with no. of double bonds



Overview Discovery Ag-Ion SPE Procedure

1) Fatty acids (FA) extracted from food sample

2) FA converted to FAMEs using BF_3

3) FAMEs are extracted into hexane

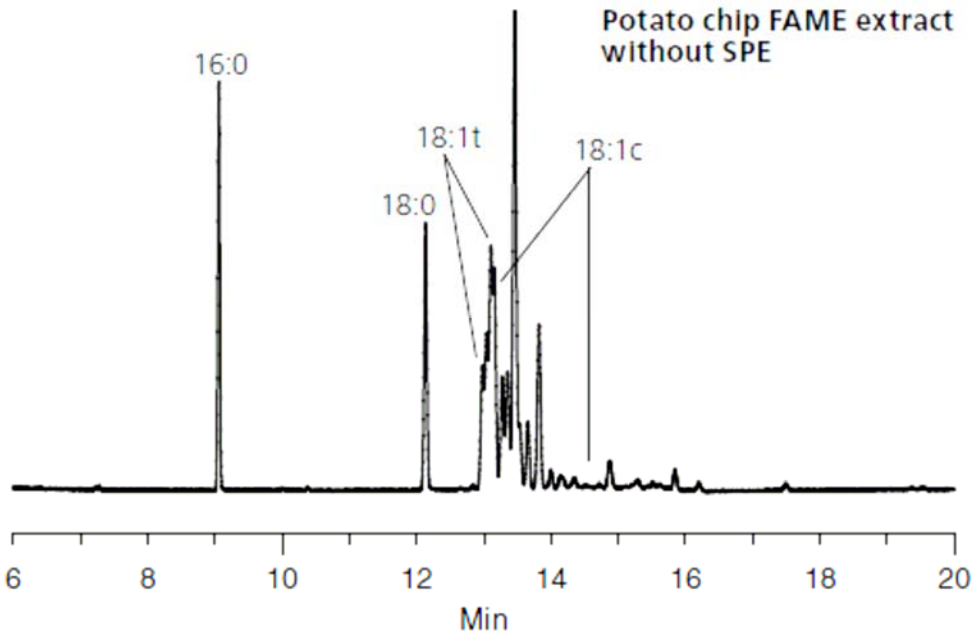
4) Hexane sample applied to Discovery Ag-Ion SPE cartridge

5) FAMEs separated using different mixtures of hexane:acetone to extract from cartridge

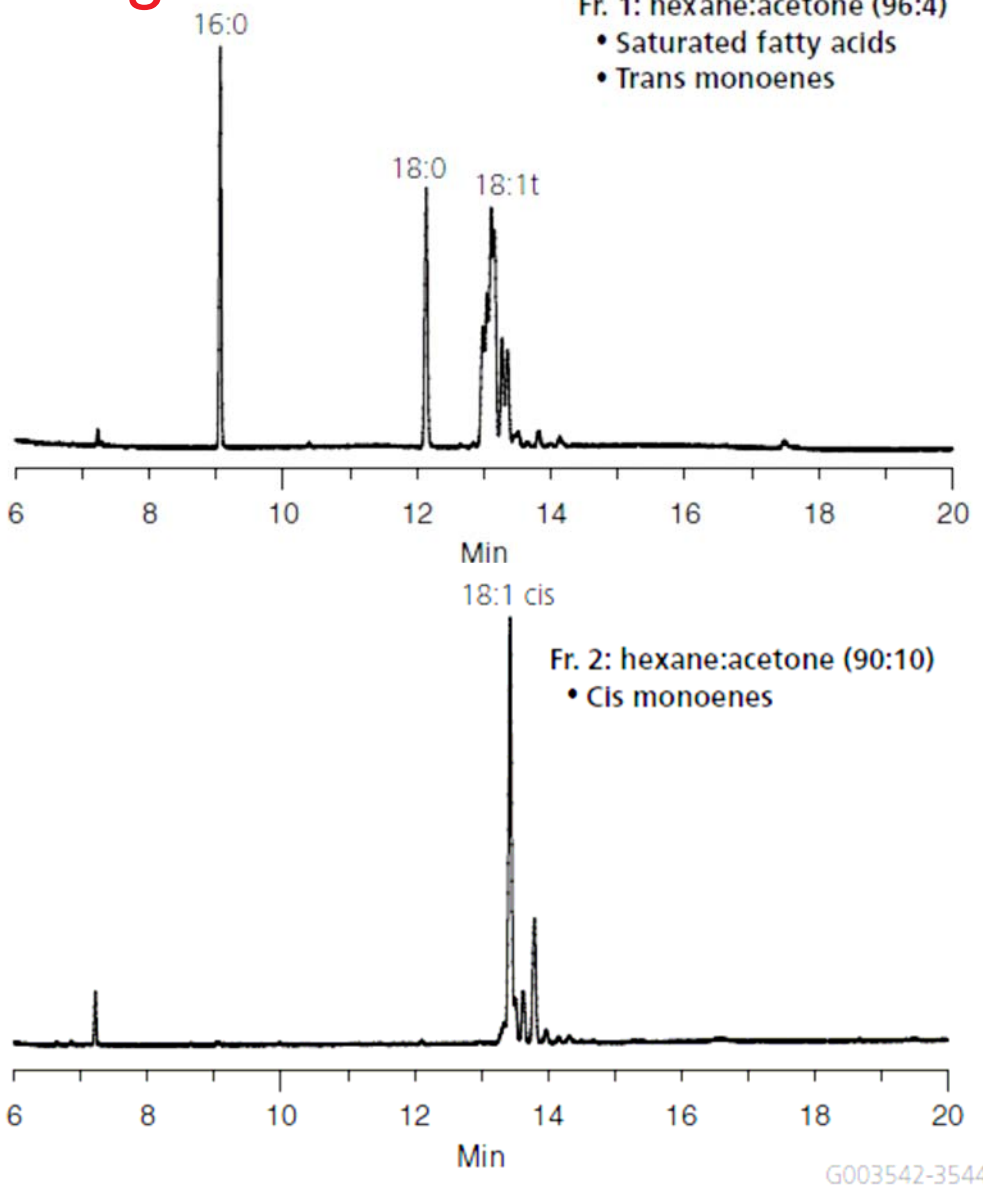
- Increasing % acetone disrupts retention of strongly retained FAMEs (cis and higher number of double bonds)

6) Fractions analyzed by GC

Cis/Trans Fractionation of FAMEs from Potato Chips with and without Ag-Ion SPE



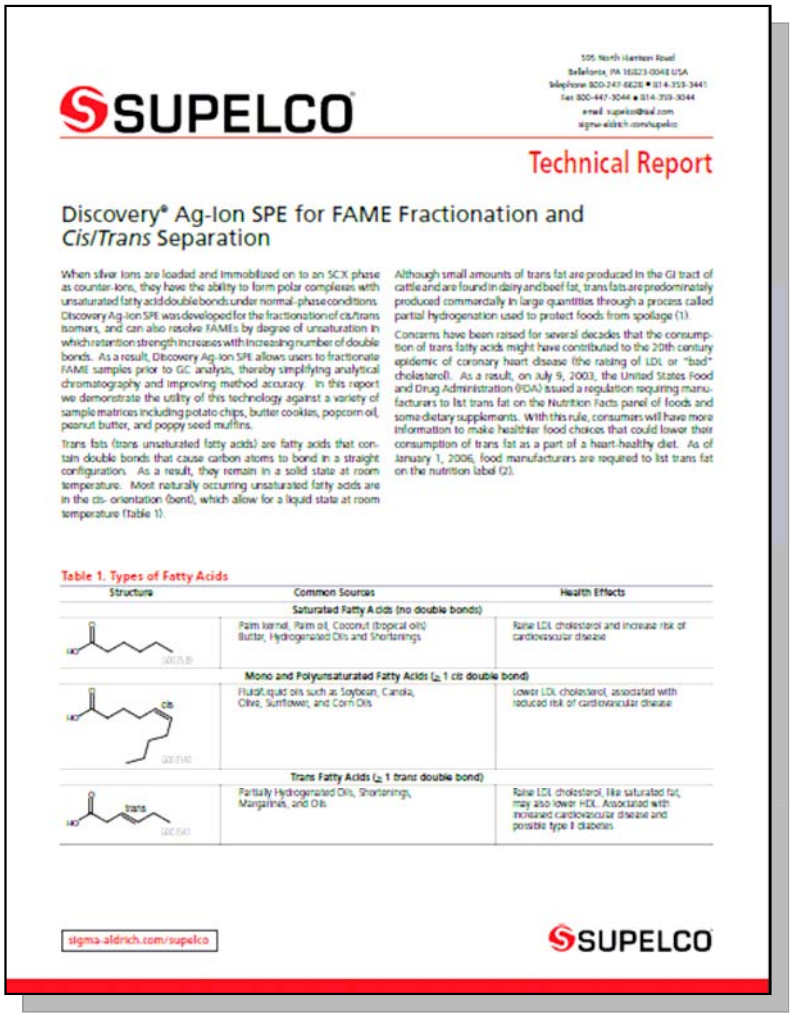
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 µm (23348-U)
oven: 180 °C, isothermal
inj.: 220 °C
det.: FID, 220 °C
carrier gas: hydrogen, 40 cm/sec. at 180 °C
injection: 0.5 µL, 100:1 split
liner: 4 mm I.D., split, cup design



Discovery Ag-Ion SPE Products

Description	Qty.	Cat. No.
Discovery Ag-Ion SPE		
750 mg/6 mL SPE Tube	30	**54225-U**
750 mg/1 mL Rezorian™ Cartridge	10	**54226-U**

http://tinyurl.com/agionspe



SUPELCO

Technical Report

Discovery® Ag-Ion SPE for FAME Fractionation and *Cis/Trans* Separation

When silver ions are loaded and immobilized on to an SCX phase as counter-ions, they have the ability to form polar complexes with unsaturated fatty acid double bonds under normal-phase conditions. Discovery Ag-Ion SPE was developed for the fractionation of cis/trans isomers, and can also resolve FAMEs by degree of unsaturation in which retention strength increases with increasing number of double bonds. As a result, Discovery Ag-Ion SPE allows users to fractionate FAME samples prior to GC analysis, thereby simplifying analytical chromatography and improving method accuracy. In this report we demonstrate the utility of this technology against a variety of sample matrices including potato chips, butter cookies, popcorn oil, peanut butter, and poppy seed muffins.

Trans fats (trans unsaturated fatty acids) are fatty acids that contain double bonds that cause carbon atoms to bond in a straight configuration. As a result, they remain in a solid state at room temperature. Most naturally occurring unsaturated fatty acids are in the cis- orientation (bent), which allow for a liquid state at room temperature (Table 1).

Although small amounts of trans fat are produced in the GI tract of cattle and are found in dairy and beef fat, trans fats are predominately produced commercially in large quantities through a process called partial hydrogenation used to protect foods from spoilage (1).

Concerns have been raised for several decades that the consumption of trans fatty acids might have contributed to the 20th century epidemic of coronary heart disease (the raising of LDL or "bad" cholesterol). As a result, on July 9, 2003, the United States Food and Drug Administration (FDA) issued a regulation requiring manufacturers to list trans fat on the Nutrition Facts panel of foods and some dietary supplements. With this rule, consumers will have more information to make healthier food choices that could lower their consumption of trans fat as a part of a heart-healthy diet. As of January 1, 2006, food manufacturers are required to list trans fat on the nutrition label (2).

Table 1. Types of Fatty Acids

Structure	Common Sources	Health Effects
	Saturated Fatty Acids (no double bonds)	
	Palm kernel, Palm oil, Coconut (tropical oils) Butter, Hydrogenated Oils and Shortenings	Raise LDL cholesterol and increase risk of cardiovascular disease
	Mono and Polyunsaturated Fatty Acids (≥ 1 cis double bond)	
	Fluid/Liquid oils such as Soybean, Canola, Olive, Sunflower, and Corn Oils	Lower LDL cholesterol, associated with reduced risk of cardiovascular disease
	Trans Fatty Acids (≥ 1 trans double bond)	
	Partially Hydrogenated Oils, Shortenings, Margarines, and Oils	Raise LDL cholesterol, like saturated fat, may also lower HDL. Associated with increased cardiovascular disease and possible type II diabetes

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SUPELCO

Topics: Sample Prep Innovations

- SPME (solid phase microextraction)
- High specificity SPE (SupelMIP)
- Dispersive SPE
- Silver Ion SPE for FAMEs
- **Carbonaceous adsorbents (ENVI-Carbs)**
- Flash chromatography

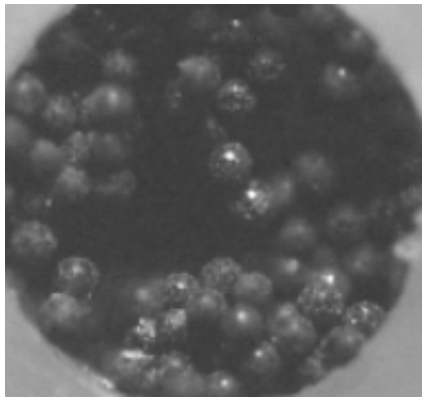
Users are...
• Analytical chemists, HPLC, GC doing sample prep

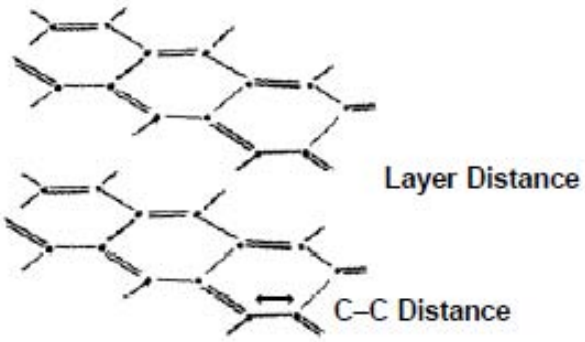
Interested in...
• Extraction of highly polar compounds from water samples, and many others...

Users can expect...
• High extraction efficiency

Structural Classification of Carbons

Carbon Class	C–C Distance (nm)	Layer Distance (nm)
Amorphous (hexagonal)	0.139	—
Turbostratic	0.142	0.365
Graphitic	0.142	0.335
Diamond (cubic)	0.155	—



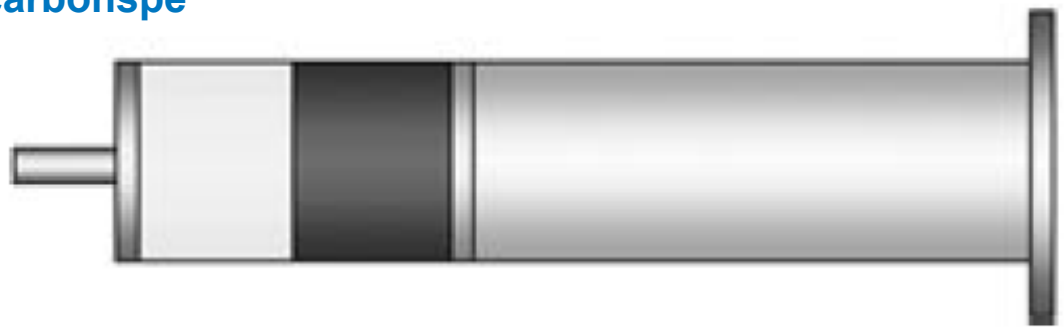


Carbon Sorbents for Sample Prep

Packed SPE tubes:

- **Supelclean™ ENVI-Carb PLUS** – spherical carbon molecular sieve for extraction of highly polar compounds from water samples
- **Supelclean ENVI-Carb-II/PSA SPE** – multilayer SPE tubes for multiresidue pesticide analysis in foods
- **Supelclean ENVI-Carb-II SPE** – isolation/removal of pigments (e.g., chlorophyll and carotenoids) and sterols commonly present in fruits, vegetables, and other natural products
- **Supelclean ENVI-Carb-II/SAX/PSA SPE** – additional ion exchange capability
- **Supelclean PSA SPE** – polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines

http://tinyurl.com/carbonspe



Dual Layer Supelclean ENVI-Carb-II/PSA SPE Tube

Supelclean ENVI-Carb PLUS

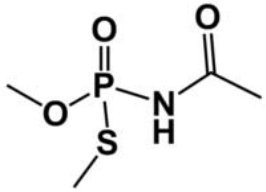
Spherical Carbon Molecular Sieve

- Extraction of highly polar compounds from water samples
- > 70% Abs Recovery from 0.5 L drinking water (1-100 ng/mL)

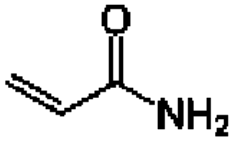
Procedure:

1. Condition w/ 10 mL MeOH & 10 mL DI water
2. Load up to 1 L sample
3. Reverse tube & elute w/ 4-5 mL MeOH in opposite direction

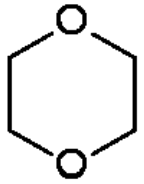
Examples of polar compounds:



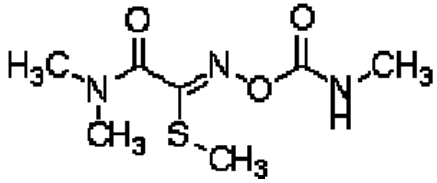
Acephate
log P -0.85



Acrylamide
log P -0.67

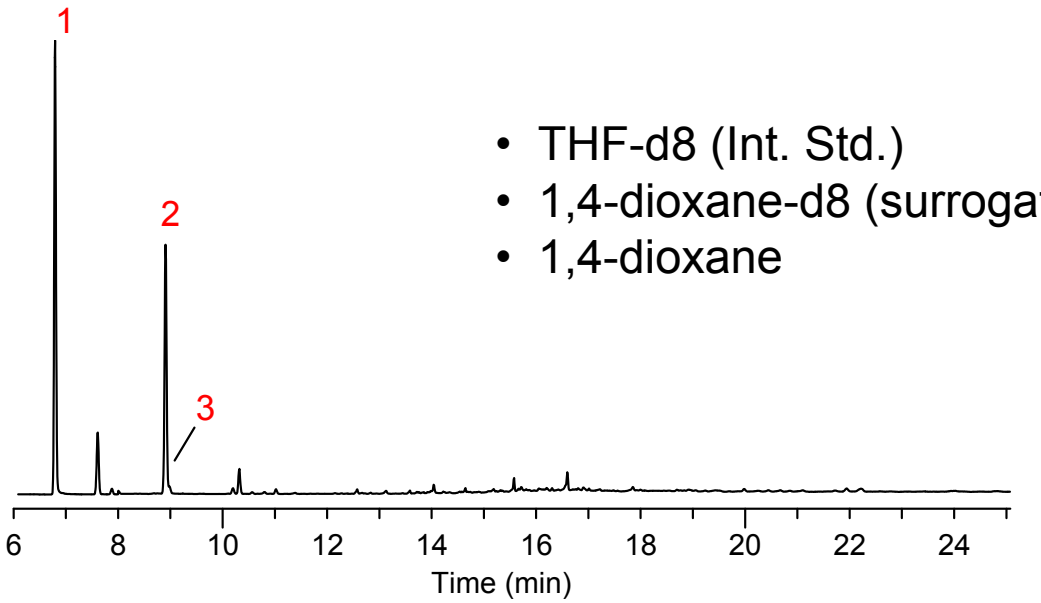


1,4-Dioxane
log P -0.27



Oxamyl
log P -1.2

GC-MS Analysis of 1,4-dioxane in water extracted using ENVI-Carb Plus



- THF-d8 (Int. Std.)
- 1,4-dioxane-d8 (surrogate)
- 1,4-dioxane

Column: SPB-624, 30 m x 0.25 mm I.D. ,1.4 µm
Oven: 30 °C (1 min.), 7 °C/min. to 90 °C, 20 °C/min. to 200 °C (3 min.)
Inj: 200 °C
Carrier: helium, 1 mL/min constant flow
Injection: 2 µL, splitless
MS interface: 220 °C
Scan range: SIM

Topics: Sample Prep Innovations

- SPME (solid phase microextraction)
- High specificity SPE (SupelMIP)
- Dispersive SPE
- Silver Ion SPE for FAMEs
- Carbonaceous adsorbents (ENVI-Carbs)
- **Flash Chromatography**

Users are...
- **Synthetic, organic chemists**
- **Medicinal chemists**

Interested in...
- **Purification of relatively large samples from reaction mixtures or other samples**

Users can expect...
- **Fast, simple, inexpensive purifications**
- **High N (spherical particles)**

Performance of Spherical vs. Irregular Silicas in Flash Application

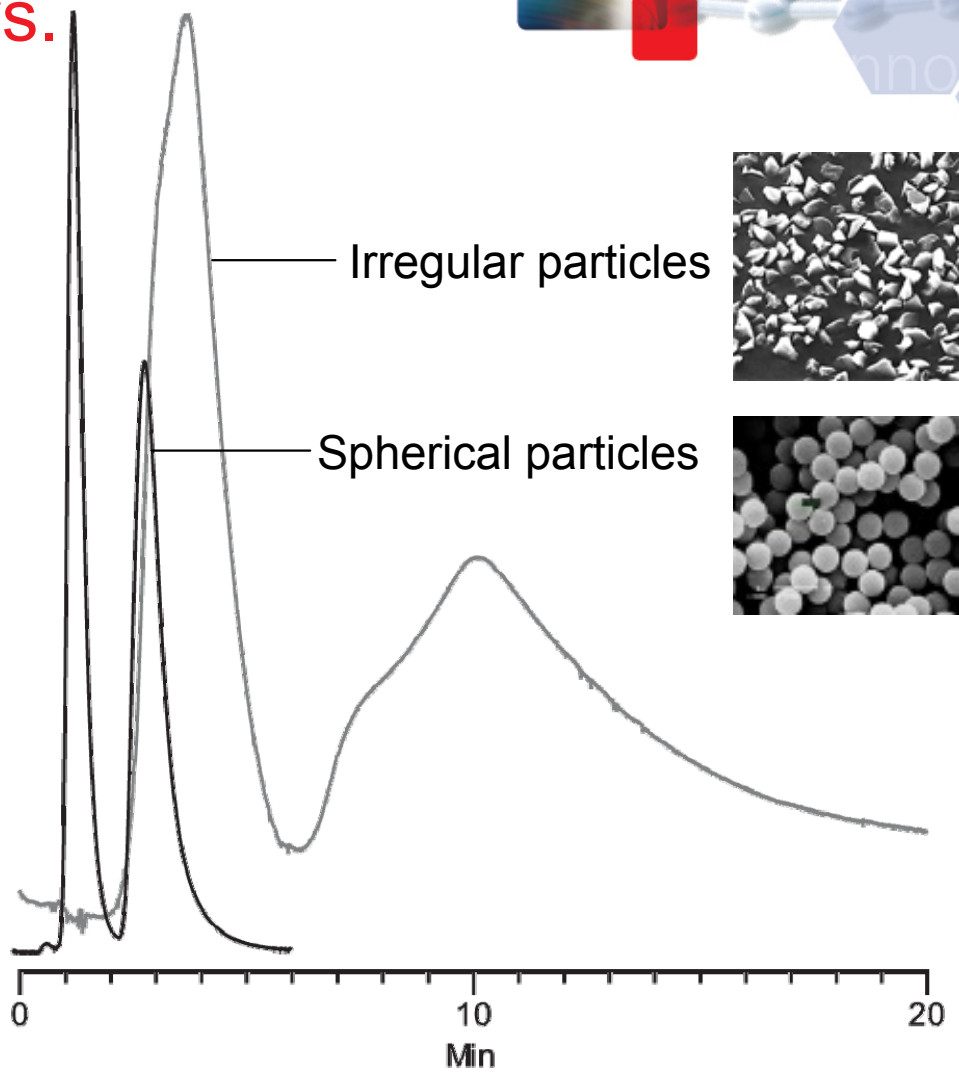
Higher efficiency of spherical particles translates to narrower bands for more concentrated fractions and faster isolations.

samples: 5-hydroxy-DL-tryptophan and DL-tryptophan

cartridges: 53 mm x 23 mm I.D.

mobile phase: methanol:water (90:10)
detection: UV 254 nm

flow rate: 20 mL/min.



Particle Type	Total Volume	Fraction 1 Volume	Fraction 2 Volume
Spherical silica	120 mL	25 mL	40 mL
Irregular silica	400 mL	80 mL	260 mL

VersaFlash Support Literature & Products

Silica & C18 Cartridges

- Particle size options
- Cartridge size options
- Cartridges can be coupled
- Reversible
- Compatible with other systems

All system components

Summary

Solid Phase Microextraction (SPME) http://www.sigma-aldrich.com/spme

High specificity SPE (SupelMIPs) http://www.sigma-aldrich.com/supelmip

Dispersive SPE for pesticide extraction http://www.sigma-aldrich.com/spe

Silver Ion SPE for FAMEs (Discovery Ag-Ion SPE)
http://tinyurl.com/agionspe

Carbon adsorbents for polar compounds (ENVI-Carbs)
http://tinyurl.com/carbonspe

Flash chromatography http://www.sigma-aldrich.com/versaflash

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Prof. Janusz Pawliszyn, U. Waterloo, Canada
Ines de Lannoy, NoAb Biodiscovery (*in vivo* applications)
Joseph Kennedy, Prosolia (DESI)

Supelco and Fluka R&D Teams

For more information on the subjects presented here, please contact techservice@sial.com or your regional sales team.

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