

A Technical Newsletter for Analytical & Chromatography

The Reporter

Europe - Issue 23, October 2006, International

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HPLC/LC

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Dear Colleague,

Welcome to the new twenty-page "Reporter", a technical newsletter for the Analytical and Chromatography industry with worldwide contributions. Our goal when redesigning this magazine was to provide you technical information on new and innovative products and applications.

Each issue will include various articles covering areas such as liquid and gas chromatography, sample handling and preparation including solid phase extraction and microextraction, standards, and accessories that are written by industry leaders, special collaborators and our application specialists. If you have an application or article you would like to submit for future newsletters please let me know, as I would be glad to talk with you about the topic and application.

In this issue Sigma-Aldrich/Supelco is proud to announce the distribution of **radiello** new generation diffusive air monitoring products. Furthermore we are proud to announce the launch of the new Phenyl phase of Supelco Ascentis HPLC columns and new US EPA SOM01.1 deuterated monitoring standards. Other articles cover additional unique applications and products that we feel answer special concerns for certain industries. We spend many hours looking over new regulations and talking with our sales specialists trying to decide on the articles and areas of interest. If you discover something new or learn about a new technique, then we have accomplished our goal.

We hope you enjoy this newsletter and find the articles both technically stimulating and informative. If you have time, please provide me any feedback you may have on its content and appearance as our goal is to provide a newsletter you find useful and worth reading.

Thank you and I look forward to hearing from you in the future.

A handwritten signature in black ink that reads "J. Donald Hobbs".

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Introducing the New Ascentis Phenyl HPLC Column

Separation Mechanisms: π - π Interactions in HPLC Chromatography

William H. Campbell and Wendy Roe
wcampbell@sial.com

Introduction

Separation of small molecules in chromatography arises from the differential relative affinities of the analytes for the stationary phase in relation to the mobile phase. Selectivity differences between two different stationary phases, under otherwise identical conditions, arise from the mechanisms in which the

analytes interact with the respective phases. Understanding and ultimately predicting selectivity changes can be tricky. To this end, the study of simple model compounds is useful to elucidate contributions from interactive mechanism on different phases.

The alkyl chain is still short enough to allow for strong contributions from the phenyl ring and maximize selectivity

The Ascentis Phenyl is a butyl phenyl phase that can act as a strong π -base (electron donor). The four-carbon alkyl spacer on the phenyl ring helps stabilize the phase toward lower UV and MS bleed. At the same time, the alkyl chain is still short enough to allow for strong contributions from the phenyl ring and maximize selectivity. In this study we probed the potential effect of π - π interactions by comparing the Ascentis Phenyl with the Ascentis C18 phase on separations of methyl substituted and nitro substituted benzene analogues. Mono, di and tri-substituted methyl or nitro benzenes chromatographed on both phases. All Ascentis phases are bonded on the same high surface area 100 Å silica.

Figure 1. Hydrophobic Interactions: Separation of Methylbenzenes

column: Ascentis C18, 15 cm x 4.6 mm I.D., 5 μ m particles (581324-U)
Ascentis Phenyl, 15 cm x 4.6 mm I.D., 5 μ m particles (581616-U)
mobile phase: 60:40, acetonitrile:water
flow rate: 1.0 mL/min
temp.: 30 °C
det.: UV at 210 nm
injection: 5 μ L
sample: toluene, p-xylene, 1,2,4-trimethylbenzene

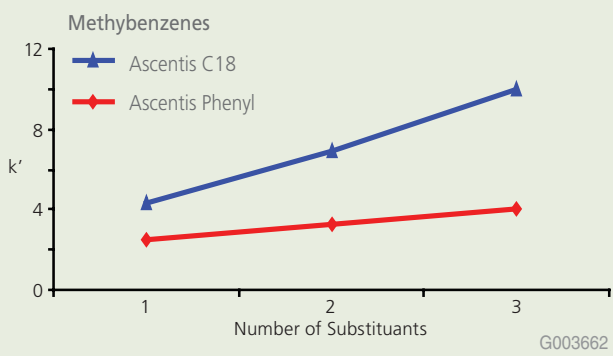
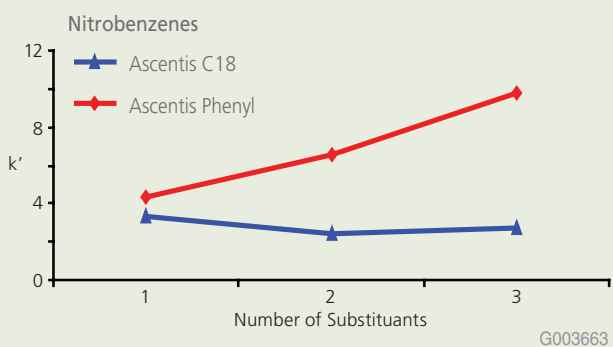


Figure 2. π - π -Interactions: Separation of Nitrobenzenes

column: Ascentis C18, 15 cm x 4.6 mm I.D., 5 μ m particles (581324-U)
Ascentis Phenyl, 15 cm x 4.6 mm I.D., 5 μ m particles (581616-U)
mobile phase: 40:60, acetonitrile:water
flow rate: 1.0 mL/min
temp.: 30 °C
det.: UV at 210 nm
injection: 5 μ L
sample: nitrobenzene, m-dinitrobenzene, 1,3,5-trinitrobenzene



Results and Discussion

The methylated aromatic analytes show increased hydrophobicity with the addition of each methyl group to the ring. So when a purely hydrophobic (dispersive) mechanism is active in separation, as with the C18, addition of each methyl should show an incremental increase in the k' of the analyte. In the absence of other effects, the degree of increase in k' is related to the hydrophobic power of the stationary phase. In Figure 1 the C18 phase shows a predictable increase in k' in going to higher methyl substitution. The phenyl shows the same trend, but to a lesser degree, indicating that the phenyl is a weaker phase for hydrophobic mechanisms.

Replacing the methyl groups with nitro groups increases the polarity of the analytes with each nitro. For a phase that functions through strictly hydrophobic interactions, the trend should be for k' values to decrease. This general trend holds for the C18 phase as shown in Figure 2.

The Ascentis phenyl does not follow the trend predicted for strictly hydrophobic phases, but in fact shows a large increase in retention with the addition of each nitro group. The addition of each nitro group increases the π -acidity (electron acceptor) of the analytes due to the strong electron withdrawing character of the nitro groups. The increase in retention indicates the formation of strong π - π complexes between the π -basic Phenyl phase and the π -acidic analytes.

The Ascentis phenyl does not follow the trend but in fact shows a large increase in retention with the addition of each nitro group

The power of π - π interactions is observed in the separation shown in Figure 3. Unique selectivity and enhanced separation and are found for the phenyl phase as opposed to the C18. The pharmaceutical analytes in this set each has aromatic moieties with electron withdrawing groups attached. The phenyl phase shows a profound difference in selectivity and a definite increase in retentivity over C18, which is most attributable to an increase in π - π interactions.

Conclusions

The new Ascentis Phenyl HPLC phase provides an alternative selectivity for π -acidic analytes relative to C18, C8 and RP-Amide phases. Additionally the Ascentis Phenyl phase is very stable, enabling both UV and LC detection.

Related Products

Description	Cat. No.
Ascentis Phenyl HPLC Columns	
5 cm x 2.1 mm, 3 μ m particles	581603-U
15 cm x 4.6 mm, 3 μ m particles	581610-U
5 cm x 2.1 mm, 5 μ m particles	581611-U
10 cm x 2.1 mm, 5 μ m particles	581612-U
15 cm x 4.6 mm, 5 μ m particles	581616-U
25 cm x 4.6 mm, 5 μ m particles	581617-U

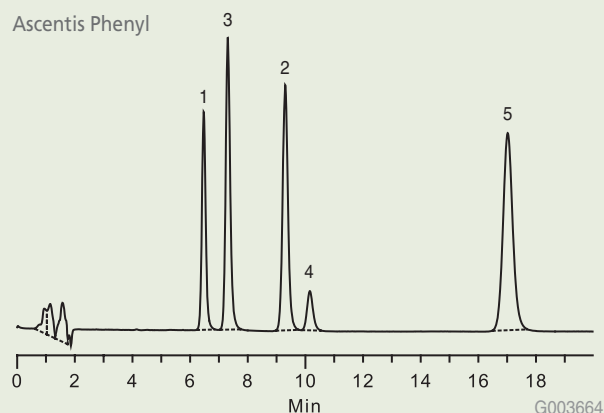
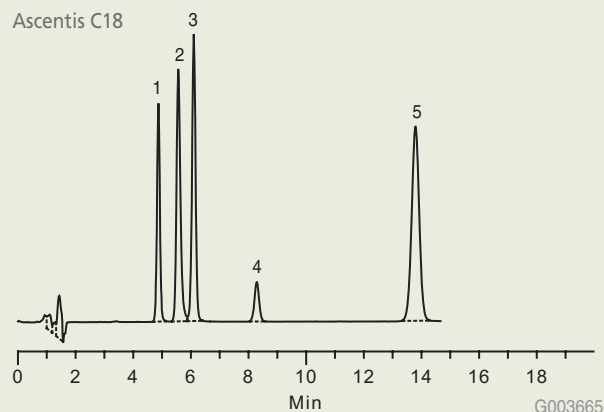
Related Information

For more information on the Ascentis HPLC Columns talk to your technical sales specialists or visit sigma-aldrich.com/ascentis

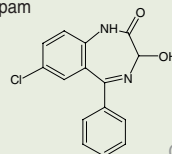
Figure 3. Separation of Anti-Anxiety Drugs

column: Ascentis C18, 15 cm x 4.6 mm I.D., 5 μ m particles (581324-U)
 Ascentis Phenyl, 15 cm x 4.6 mm I.D., 5 μ m particles (581616-U)
 mobile phase: 40:60, acetonitrile:water
 flow rate: 1.0 mL/min
 temp: 25 °C
 det: UV at 254 nm
 injection: 10 μ L

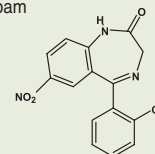
1. oxazepam
2. alprazolam
3. cloazepam
4. n-desmethyldiazepam
5. diazepam



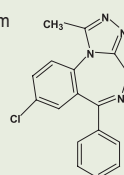
oxazepam



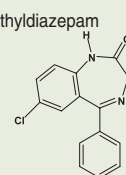
cloazepam



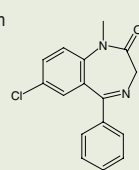
alprazolam



n-desmethyldiazepam



diazepam



Indole Alkaloid Separation Using the Discovery® HS F5

Chemotaxonomic Study of Two Closely Related Brown-Spored Mushrooms

Ilia Brondz¹, Klaus Høiland², David Bell³ and Amy R. Annino³

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² Department of Biology, University of Oslo, Norway

³ Supelco, Bellefonte, Pennsylvania USA, e-mail: aannino@sial.com

Abstract

Chromatographic analysis can be an extremely valuable tool in the taxonomic classification of microorganisms, plants and fungi. In this study, the Discovery HS F5 column was used to analyze the indole alkaloid content of two closely related species of brown-spored mushroom: *Cortinarius infractus* and *C. subtortus*. The comparative indole alkaloid content analysis, along with chromatographic fingerprinting evidence, allowed the researchers to classify *C. infractus* and *C. subtortus* into two different taxonomical sections.

Introduction

The pentafluorophenylpropyl phase of the Discovery HS F5 column offers excellent separation of alkaloid compounds under conditions suitable for LC-MS. The fluorinated stationary phase provides a sufficient substrate for ionic interactions with basic compounds so that ion pair reagents and phosphate buffers are not necessary. The aim of this investigation was to determine the section-level taxonomic classification of two brown-spored fungi (*C. infractus* and *C. subtortus*, in the order *Agaricales*, *Basidiomycota*, subgenus *Phlegmacium*). Some researchers classify them into one section, *Amarescens* Mos., based on certain shared physical characteristics and partial DNA sequence information (1), while others place them into two sections, *Infracti* and *Subtorti* (5), based on several physical differences and supposed dissimilar alkaloid composition using bitter taste as evidence for the latter.

C. infractus has an unpleasant, fishy odor and a strong bitter taste which may partly be caused by the previously demonstrated presence of the indole alkaloids infractine, 6-hydroxyinfractine and infractopicrine (2). There is no known published chemical analysis for *C. subtortus*; however, this pleasant-smelling species with only a weakly

bitter taste is not expected to contain indole alkaloids. As infractine-like alkaloids, which belong to the indole alkaloid group, may be exclusively found in *C. infractus*, these compounds may prove to have profound value as chemotaxonomic markers in the genus *Cortinarius*.

Experimental

The Discovery HS F5 HPLC column was conditioned specifically for Supercritical Fluid (SFC) by flushing with 100 % methanol followed by a gradient of methanol: hexane 2 % increase per minute and subsequently held at 100 % hexane for 10 min. Before use, the column was flushed with carbon dioxide in the supercritical state.

SFC-MS analyses were performed on a Berger SFC MiniGram equipped with an UV K-2501 detector and ProNTo software. The flow stream exiting the UV detector was diverted by a fixed splitter and used to feed a Micromass PLCZ 4190 mass spectrometer equipped with ESI running under MassLynx. The biological samples were prepared as follows: 1 g samples of *C. infractus* and *C. subtortus* were air dried and extracted in 10 mL ethanol.

Results and Discussion

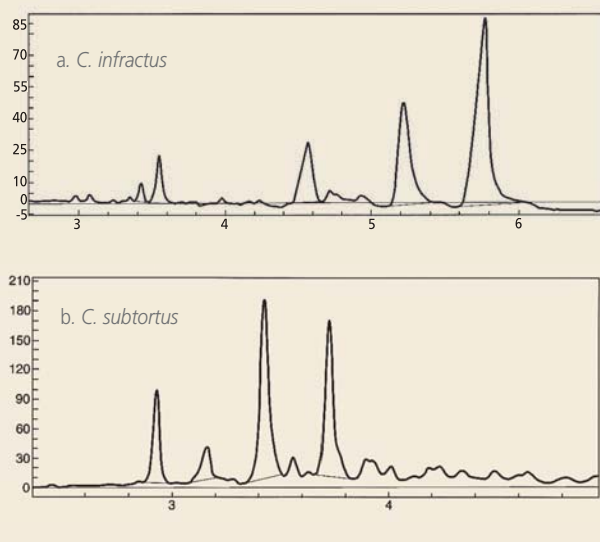
The total ion chromatograms (TIC) for the extracts of *C. infractus* and *C. subtortus* are shown in Figures 1a and 1b, respectively. There are five significant peaks in the chromatogram of *C. infractus*, and four in the chromatogram for *C. subtortus* (almost all of which retain longer than the peaks for *C. infractus*). A mixture of the two extracts yielded no common peaks.

The SFC-MS fragmentation patterns obtained offered vital information to the identification of two compounds in the *C. infractus* extract: that which eluted at 4.6 min and that which eluted at 5.3 min (Figure 1a). It was proposed that the compound eluting at 4.6 min was infractopicrine based on the fragmentation pattern (which indicates that the compound in question contains fused aromatic rings) and molecular mass M^+ detected was m/z 261. The structure of infractopicrine is shown in Figure 2a.

The peak that eluted at around R_t 5.3 min was putatively identified as pre-infractine. Though the poor

Figure 1. Chromatograms for *C. infractus* and *C. subtortus*

column: Discovery HS F5, 25 cm x 4.6 mm I.D., 5 µm particles (567517-U)
 mobile phase: A: supercritical carbon dioxide B: methanol
 gradient: 5 - 55 % B, 10 %/min.
 flow rate: 5 mL/min.
 temp.: 35 °C
 det.: UV at 268 nm
 MS conditions: cone voltage 60 V, ion energy 1.0 V, multiplier 400 V, vacuum 2.6 kPa, desolvation gas flow of 495 L/hr., function type: scan, mass range from 50 to 400.
 injection: 5 mL

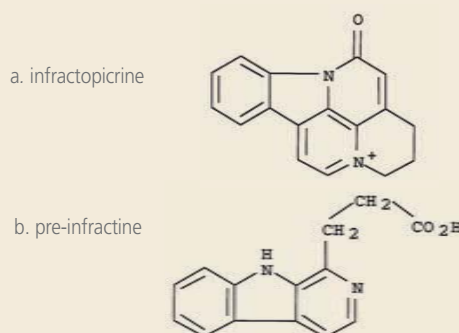


fragmentation of this peak at a 60 V cone voltage, the $[M+H]^+$ ion at m/z 241 was easily visible. The molecular weight of this compound should be 240, which is 14 units fewer than infractine. It was temporarily proposed that this compound is pre-infractine (β -carboline-1-propionic acid); the structure is shown in Figure 2, compound b.

Conclusions

SFC-MS equipped with the Discovery HS F5 column was proven to be a powerful tool in separating synthetic alkaloid-like substances (3, 4), and likewise it was a valuable tool in this separation of natural alkaloids. Analysis of the indole alkaloid content of *C. infractus* and *C. subtortus* showed that *C. infractus* contained pre-infractine (β -carboline-1-propionic acid) and infractopicrine. However, neither compound was found in *C. subtortus*. The chemotaxonomic evidence does not support a close relationship between *C. infractus* and *C. subtortus*. This research supports the separation of these two species into two different sections, *Infracti* and *Subtorti*, following the previous proposal by Brandrud *et al.* (5).

Figure 2. Structures of indole alkaloids



Taking into consideration the molecular analysis by Garnica *et al.* (1) and a SFC-MS analysis conducted during this study, it is accepted that these two sections belong to the same monophyletic clade.

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- Garnica, S., Wei, M., Oertel, B. & Oberwinkler, F. 2003. Phylogenetic relationships of European Phlegmacium species (Cortinariaceae, Agaricales). *Mycologia* 95: 1155-1170.
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- Brandrud, T.E., Lindström, H., Marklund, H., Melot, J. & Muskos, S. 1989-1998. Cortinarius Flora Photographica I-IV. Cortinarius HB, Matfors.

Related Products

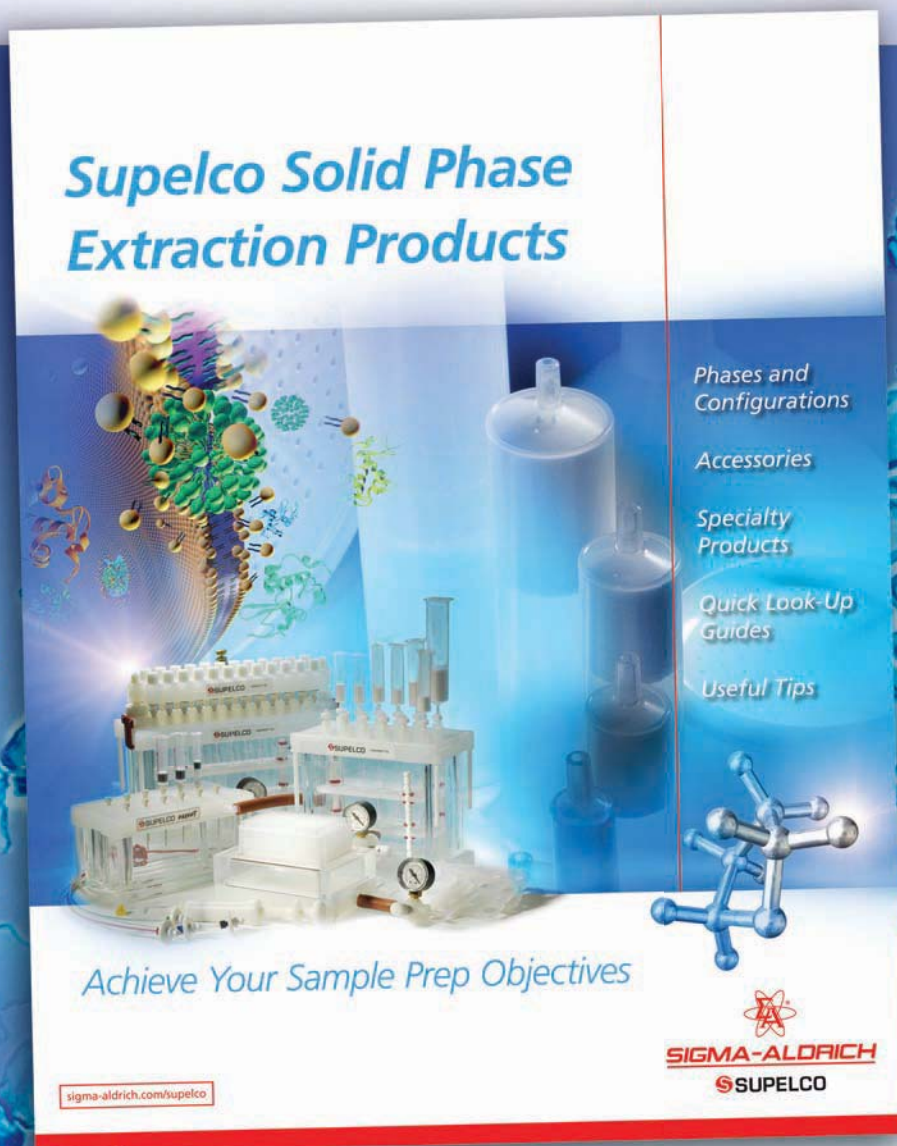
Description	Cat. No.
Discovery HS F5 HPLC Columns	
25 cm x 4.0 mm I.D., 5 µm particles	567536-U
15 cm x 4.6 mm I.D., 5 µm particles	567516-U
5 cm x 2.1 mm I.D., 5 µm particles	567508-U
5 cm x 2.1 mm I.D., 3 µm particles	567500-U

Did you know...?

Polar stationary phases often exhibit increased ion-exchange interactions over C18 columns. It is important to choose appropriate mobile phase modifiers to control ion-exchange mechanisms. Simply adjusting pH with formic acid, for example, may not be as favorable for such systems as using ammonium formate adjusted to the appropriate pH.

TRADEMARKS: Ascendis, Carboxen, Discovery, ENVI-Carb, OMI, SLB, StableFlex, Supelclean, Supelco - Sigma-Aldrich Co.; Florisil - U.S. Silica Company; Microseal - Merlin Instrument Company; Nanochem - Matheson Tri Gas; SCOTTY - Scott Specialty Gases, Inc.; Swagelok - Swagelok Co.; Viton - E.I. duPont de Nemours & Co., Inc.

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sigma-aldrich.com/supelco



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radiello[®] The new generation of diffusive air sampling: - Fast and reliable passive samplers now available from Supelco

The widely accepted **radiello** passive air samplers of the Fondazione Salvatore Maugeri/Padova (FSM), Italy are now available from Sigma-Aldrich / Supelco. The Fondazione and Sigma-Aldrich have entered into a global distribution agreement* on this unique line of diffusive sampling products. This cooperation brings expertise in air monitoring (FSM) and in adsorbent technology (Supelco) together to form a strong alliance for supporting the air monitoring needs of analysts now, and into the future.

Passive Diffusive Sampling

Passive diffusive sampling relies on the diffusion of analytes through a diffusive surface onto an adsorbent. After sampling, the analytes are chemically desorbed by solvent extraction or thermally desorbed and analysed. Passive sampling does not involve the use of heavy and encumbering pumping systems, is not impacted by power disruptions, does not require extensive supervision, is quiet, non-flammable, and does not represent an explosion hazard. It can be performed by anyone, anywhere, and at a very low cost. Moreover, it is not susceptible to sample breakthrough, a common problem associated active sampling performed with an air pump.

radiello overcomes problems associated with diffusive sampling

Because of the limits set by its geometry, the axial symmetry of traditional passive samplers results in poor sensitivity and irreproducibility. Uptake rate values are low and often highly variable depending on environmental conditions. The **radiello** sampler has overcome these limitations.

Development of the radiello design

In the mid 1990's, Dr. Vincenzo Cocheo, director of the Fondazione Salvatore Maugeri, Padova, Italy, in collaboration with the European Commission's Joint Research Center and other institutions, developed and patented a revolutionary diffusive/sampling design: Radial symmetry (Figure 1) now known by the registered trademark "**radiello**."

* Italy from March 2007



Figure 1. Radial Design

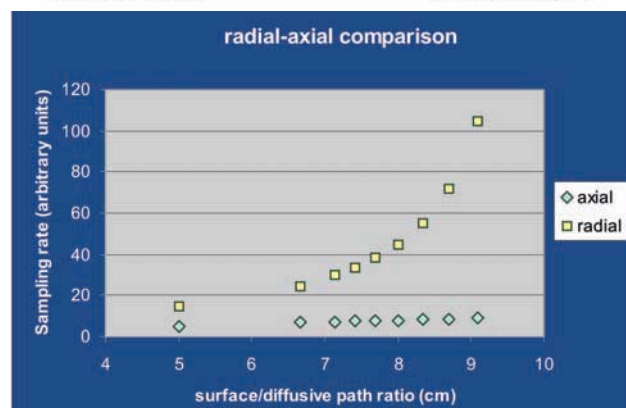
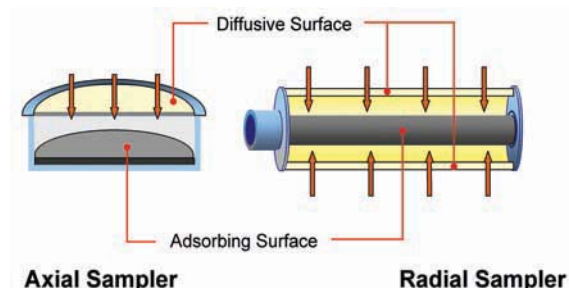


Figure 2. Comparison radial to axial sampler

By virtue of radial symmetry, uptake rate is:

- **High** - The **radiello** cartridge's uptake rate is at least three times higher than an axial diffusive sampler with the same dimensions.
- **Constant** - This is a result of the significantly increased adsorbing capacity of the adsorbing cartridge.
- **Reproducible** - The stiffness of the diffusive wall and cartridge, together with the close tolerances characterizing all of the **radiello** components, greatly reduces uptake rate variation.
- **Independent of air speed** - Because of the tortuous nature of the diffusive path inside the micro-porous diffusive membrane, uptake rate is not affected by wind or air currents.
- **Precise** - Uptake rate is not calculated, but is experimentally measured in a controlled atmosphere chamber over a wide range of concentration, temperature, relative humidity, air speed, and with and without interferences.
- **Adjustable and flexible** - By selecting a less porous diffusive body, the virtual diffusion length can be extended from 18 mm to 150 mm for longer-term sampling.

! Related Information

For more information on **radiello** please visit sigma-aldrich.com/radiello

Applications

Typical applications include Ambient/Outdoor Air Monitoring, Industrial & Indoor Air Quality (IAQ), and Industrial Hygiene / Personal Sampling (Figure 3).

radiello diffusive samplers have been designed for the following air-borne contaminants.

- Aldehydes
- VOCs and BTEX (with solvent desorption)
- VOCs and BTEX (with thermal desorption)
- Nitrogen and Sulfur dioxide (NO₂ and SO₂)
- Ozone (O₃)
- Hydrogen sulfide (H₂S)
- Ammonia (NH₃)
- Hydrogen chloride (HCl)
- Hydrogen fluoride (HF)
- Anesthetic Gases and Vapors
- Phenol, methylphenol, and dimethylphenol (with thermal desorption)

radiello Components and Sampling Procedure

A **radiello** diffusive sampler consists of 3 parts: the diffusive body, the adsorbent tube, and the triangular support plate (Figure 3 right). For personal air sampling, a vertical adapter can be installed. The adsorbent cartridge is removed from its glass or plastic storage tube and is



Figure 3. Outdoor (left) and personal (right) sampling. Outdoor sampling using the **radiello** shelter. The sampler consists of the diffusive body (white cylinder), the adsorbent tube (not shown, inside the diffusion body), and the blue triangular support plate.

inserted into the diffusive body. This is then threaded onto the triangular support plate in order to place the sampler in its sampling position. The date and time for the start and end of sampling period are noted on an adhesive barcode label that is supplied with the sampling cartridge. Following sampling, the adsorbent cartridge is returned to the storage tube and the barcode label is affixed to the tube. The tube containing the sampling cartridge is then ready for shipment to the lab for analysis. The diffusive body and the support

plate can be reused, making the **radiello** sampler system very economical. All components can be purchased separately.

Analyzing Collected Samples

Any skilled laboratory can perform the analysis. Procedures are provided in the **radiello** manual. Additionally, the Fondazione Salvatore Maugeri, Padua/Italy provides analysis services. Loaded adsorbent cartridges can be shipped directly to the FSM for determination. Details such as shipping procedures and analysis prices can be requested from the FSM or found at www.radiello.com.



radiello and Official Methods & Studies

The **radiello** sampler is cited in the European Method EN 14662-5 (Benzene in Ambient Air) as a type B sampler. ISO/FDIS 16200-2 (Workplace Air Quality) describes the **radiello** sampler as a type D sampler. In EN 14662-4 (Ambient Air Quality – Measurement of Benzene), EN ISO16017-2 (Indoor, Ambient and Workplace Air – Measurement of VOC's) and ASTM D6196-3 (Volatile Organic Compounds in Air) a passive sampler, followed by thermal desorption is required. **radiello** offers a thermal desorption option with its Carbograph 4 packed adsorbent cartridge (code 145) that can be placed in an empty thermal desorption tube for analysis.

The Artemide project for "High Temporal Resolution Monitoring of VOCs by Diffusive Sampling" employed **radiello** samplers and was recently recognized as one of 24 of the best LIFE projects 2004/2005 funded by the European Commission. Further studies and projects utilizing **radiello** samplers are listed in the **radiello** brochure.

The **radiello** product portfolio consists of ready to use samplers, replacement adsorbent tubes, standards for calibration and accessories such as a shelter for outdoor measurements.

Are you already using radiello?

If you are already using **radiello** samplers and wish to purchase them from Sigma-Aldrich, just use the **radiello** code and place "RAD" in front to convert to the Supelco catalog number, (e.g. **radiello** code 130 ► RAD130 or Code 126-1 ► RAD1261).

More Information

For more information on passive diffusive air sampling and the extensive line of **radiello** products, please request your free copy of the **radiello** overview brochure T406090 (IXV), the **radiello** CD (IXW) containing all information and instructions for these products, or visit our website at www.sigma-aldrich.com/radiello.

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SPME Fiber Selection (Part 1)

The Evolution of the SPME Fiber Assembly

Daniel Vitkuske

dvitkuske@sial.com

The expansion of the SPME product portfolio and the development of new and improved fiber coatings and fiber assembly technology have led to confusion for people using SPME and to new SPME users. Today SPME users must choose between manual and autosampler fiber assemblies, fused silica, StableFlex™ or metal alloy fiber cores, 24 versus 23 gauge needles, 1 cm versus 2 cm fibers, and multiple fiber coating chemistries. Altogether there are almost 50 different standard SPME fiber coatings and assembly options to choose from, each one offering a unique combination of physical and chemical interaction capabilities.

Manual versus Autosampler Fibers

The main difference between fibers for manual sampling versus autosampler fibers is the presence of a spring on the manual fibers to maintain the fiber in position during sample extraction and to assist in retracting the fiber prior to analysis. While autosampler fibers can be used in a manual assembly, manual fibers should not be used in an autosampler without removing the spring.

SPME Fiber Cores

The workhorse of the SPME fiber assembly is the coated fiber, the first SPME fiber assemblies were coated fused silica rods. While the silica core also provides some adsorption capability, these coated fibers could be brittle

and can break more easily than other fiber core types. StableFlex SPME fibers were later developed by applying a thin, flexible polymeric coating over the fused silica prior to applying the adsorbent/absorbent fiber coating. This “pre-coating” increased the durability of the fiber core and made it less susceptible to breakage. More recently, the durability of the fiber assembly was improved by using a super-elastic metal alloy for the fiber core, further increasing fiber assembly life up to 10 fold. The metal alloy fibers are especially suited to cope with the potential mechanical stress during autosampler use.

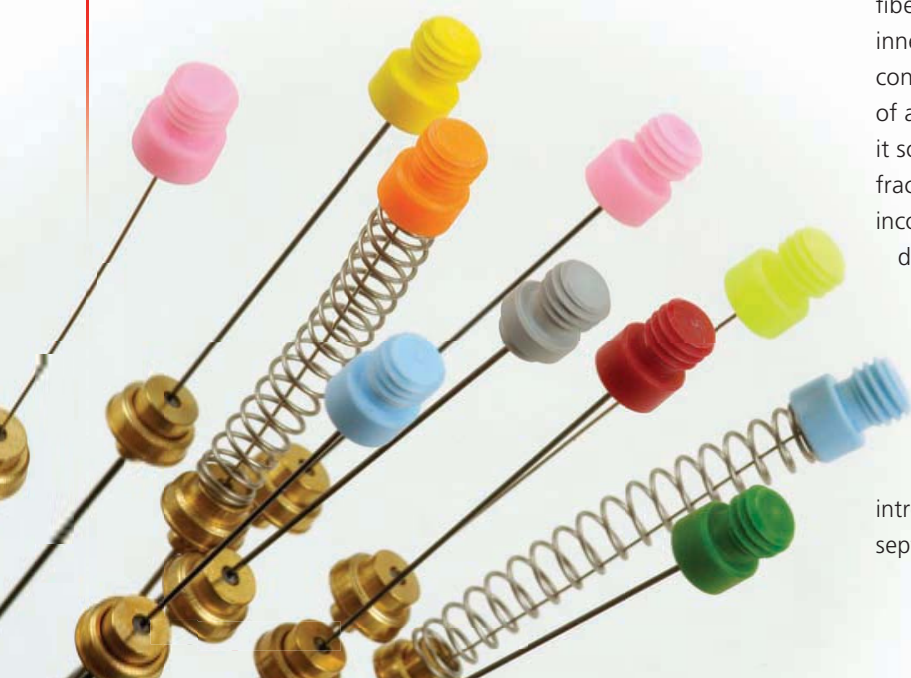
However, there’s more to the SPME fiber cores than the physical characteristics of the material from which they are made and the resulting fiber durability. The super-elastic metal alloy also lends itself to a continuous fiber coating process that results in a much more consistent and uniform coating thickness resulting in a significant improvement in fiber-to-fiber reproducibility.

This improvement results more from the limitations of the fused silica which cannot be wound on a core for continuous processing and must be handled in relatively short “rods” in a “batch treatment” process which can lead to inconsistent coating thickness and concentricity problems.

SPME Needle Gauge, Material and Tip Design

Proper and lasting functioning of the SPME assembly requires that the SPME fiber is always retracted into the needle when piercing a septa for injecting into a sample or the GC port. The initial SPME fiber assemblies used 24 gauge stainless steel needles to protect the coated SPME fiber during septa puncture and GC injection. While the inner diameter of the 24 gauge needle is sufficient to contain/house the coated SPME fiber, the combination of a narrow inner diameter and a thin needle wall makes it somewhat fragile and susceptible to bending and fracture. The 23 gauge stainless steel needles were later incorporated into the fiber assemblies with a larger inner diameter and thicker needle wall resulting in increased resilience to the challenges of septa and injection port penetration. The 23 gauge needles are also compatible with the Merlin Microseal™ or similar septum-less injection systems, eliminating septa coring problems sometimes encountered with SPME.

More recently, 23 gauge metal alloy needles were introduced with a tapered tip as an additional aid to make septa penetration less stressful on the fiber assembly.



Reproducibility and Durability of Different Fiber Assembly Types

Core Type	Needle & Tip Type	Durability	Reproducibility
Fused Silica	24 gauge SS, blunt tip	Good	Good
StableFlex	23 gauge SS, blunt tip	Better	Good
Metal Alloy	23 gauge metal alloy, tapered tip	Best	Best

Comparison of SPME Fiber Needle Tips



Standard versus 2 cm Fibers

Although a limited number of fiber coatings are available as 2 cm options, the fiber capacity of a 2 cm fiber is approximately double, relative to the 1 cm version and therefore can be advantageous for trace analysis.

SPME Fiber Coatings

Today there are 5 different standard SPME fiber chemistries ranging from adsorbent to absorbent coatings, polar to non-polar in a wide range of coating thicknesses. Characteristics such as the analyte's molecular weight, polarity and volatility help to determine the best fiber coating for specific analytes. Due to space constraints, we will not give a comprehensive overview of SPME fiber coating selection in this issue. This will be covered in a future Reporter article, Part 2 of SPME Fiber Selection. Please refer to the Supelco Catalog or sigma-aldrich.com/supelco-spme. There is a previous publication on SPME fiber coating selection, *Optimization of Extraction Conditions* (1).

Custom SPME Fibers

Despite the wide range of SPME fiber assemblies and coatings, researchers routinely contact us interested in obtaining non-standard SPME fiber assemblies. For more information on custom SPME capabilities, please contact Technical Service (EurTechServ@europe.sial.com).

Reference

1. Shirey, R., J. of Chrom. Science, Vol. 38, July 2000.

+ Featured Products

Description	Cat. No.
SPME Metal Fiber Assemblies	
Polydimethylsiloxane (PDMS)	
100 µm	57928-U
30 µm	57922-U
7 µm	57919-U
Polydimethylsiloxane/Divinylbenzene (PDMS/DVB)	
65 µm	57902-U
Carboxen™/Polydimethylsiloxane (CAR/PDMS)	
85 µm	57906-U
Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS)	
50/30 µm	57912-U
50/30 µm - 2 cm length fiber	57914-U

+ Related Products

Description	Color	Qty.	Cat. No.
Screw Neck Vials			
10 mL round bottom	Clear	100	SU860099
10 mL round bottom	Amber	100	SU860100
20 mL round bottom	Clear	100	SU860097
20 mL round bottom	Amber	100	SU860098
Magnetic Screw Caps, 8 mm Hole			
with 1.3 mm thick silicone blue/PTFE white septa		100	SU860101
with 1.5 mm thick silicone blue/PTFE white septa		100	SU860103
with 1.6 mm thick butyl red/PTFE gray septa		100	SU860102
SPME Flat Neck Vials			
20 mL round bottom	Clear	100	SU860104
Magnetic Crimp Caps, Gold, 8 mm Hole			
with 1.5 mm thick silicone white/PTFE blue septa		100	SU860053
with 1.0 mm Viton™ black septa		100	SU860106
20 mm x 1.5 mm thick silicone/PTFE septa		100	27541
20 mm x 0.75 mm thick Viton septa		100	27247

! Related Information

For a complete list of available products, visit us online at sigma-aldrich.com/supelco-spme or request the SPME Applications CD (T199925-CJQ) for a comprehensive collection of the available literature and references to SPME.

Did you know...?

The 5th Edition SPME Applications CD includes over 1500 references for articles on the use of SPME in a wide range of applications, as well as comprehensive literature on the SPME theory and optimization. The 6th Edition SPME CD will soon be released with over 2200 application references as well as short video demonstrations of various aspects of SPME, including *Getting Started with SPME*, *SPME Use with An Autosampler*, as well as many other topics.

Extraction and Analysis of Agricultural Pesticides from Oranges Using the “QuEChERS” Method

Katherine Stenerson, Robbie Wolford, and Olga Shimelis

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Introduction

The toxicological effects of the chemicals which humans and animals are exposed to daily are of ever-increasing concern. In the last few years, emphasis has been placed on a group of chemicals loosely referred to as endocrine disruptors; mostly man-made compounds suspected of interfering with the body’s hormone system (1) by blocking or mimicking normal function. One of the avenues for human exposure to these compounds is through the consumption of agricultural products that have been treated with pesticides. These pesticides may have been used as insecticides, fungicides, or herbicides during growth, transportation and storage stages.

A number of methods currently exist for the extraction and analyses of multi-residue pesticides from a variety of food matrices (2,3). A new method, known as the “QuEChERS” (Quick, Easy, Cheap, Effective, Rugged, and Safe) method, has recently been introduced (4) and subsequently improved (5,6). This method employs dispersive solid phase extraction (SPE) and gas chromatography-mass spectrometry (GC-MS) techniques.

“Dispersive SPE” Procedure

With typical SPE methods, sample is passed through a tube that contains sorbent, and retained analytes are eluted with solvent. In dispersive SPE, organic solvent is mixed with a sample, and gram levels of salts are added to drive partitioning of the analytes between the aqueous residues and the solvent. An aliquot of the organic solvent is then removed and mixed with additional salts and sorbent as an additional cleanup step. This procedure requires less time than traditional SPE, and simultaneously removes residual water and matrix interferences. After a simple vortex and centrifugation step, the supernatant is ready for analysis.

The improved QuEChERS method published by Lehotay (6) was used for the extraction of 29 different agricultural pesticides from oranges. The oranges used for the extractions were obtained from a local grocery store and were not labeled as “organic.” Four extracts were prepared from orange skins according to the procedure

Table 1. Extraction Procedure

1. Weigh 15 g of ground-up orange.
2. Add 75 µL of the internal standard stock solution (ethoprophos at 20 ppm in methanol) to all samples.
3. Add 75 µL of the pesticide stock solution (29 pesticides, each at 20 ppm in methanol) to the “spike” samples.
4. Add 15 mL of 1 % acetic acid in acetonitrile.
5. Add 6 g anhydrous magnesium sulfate (MgSO ₄) and 1.5 g anhydrous sodium acetate.
6. Shake by hand for 1 minute.
7. Centrifuge for 2 minutes at 3300 rpm.
8. Take a 2 mL aliquot of extract.
9. Add 100 mg primary-secondary amine (PSA) and 300 mg anhydrous magnesium sulfate (MgSO ₄).
10. Centrifuge for 2 minutes at 3300 rpm.
11. Take a 1 mL aliquot of extract and evaporate to 0.1 mL.
12. Reconstitute in toluene to 1.0 mL using a volumetric flask.
13. Proceed to GC analysis.

summarized in Table 1. An extract spiked only with internal standard at 100 ppb served as a control. Three replicate extracts were spiked with each pesticide plus the internal standard (each at 100 ppb) and used to determine the accuracy and precision of the method. The final extracts were solvent exchanged from acetonitrile to toluene to increase the sensitivity of the GC-MS analysis. Vials containing pre-weighed salts and sorbent were used to perform the extraction and cleanup procedures. These vials were produced in-house, and are currently available as custom items (7).

GC-MS Analyses

GC-MS analysis of the extracts described in the previous paragraph was performed on a single quadrupole GC-MS system using selective ion monitoring (SIM). Monitoring ions were chosen based on the spectra of the pesticides taken from a full mass range analysis of a high level standard. An SLB™-5ms capillary column was chosen for the analysis due to its low bleed and high inertness characteristics, resulting in its ability to detect the pesticides at a low level (8,9). Complete GC-MS conditions are listed in Figure 1. A five-point calibration using matrix-matched standards was performed prior to analyses of the extracts.

Figure 1. Extract of Spiked Orange Sample

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m (28471-U)
 oven: 100 °C (1 min.), 10 °C/min. to 300 °C (5 min.)
 inj.: 250 °C

MSD interface: 300 °C

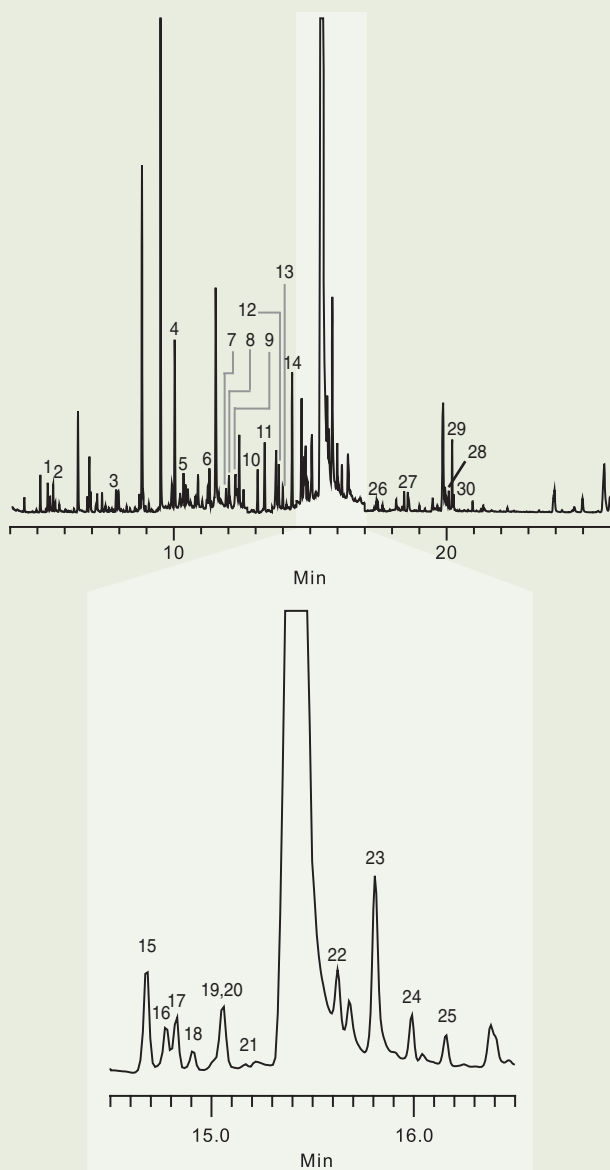
scan range: selected ion monitoring (SIM), 7 monitoring groups used

carrier gas: helium, 1 mL/min constant

injection: 1 μ L, pulsed (20 psi until 0.20 min.), splitless (1.0 min.)

liner: 4 mm I.D., single taper

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



G003590

Results

Chromatograms of the spiked orange samples are presented in Figure 1. Several background peaks eluting prior to nine minutes are due to impurities in the toluene. Despite extract cleanup, matrix peaks are also present in the chromatograms. Further sample cleanup may be possible by increasing SPE sorbent weight. Nevertheless, all pesticides were detected. Calibration, recovery, and precision data are presented in Table 2. A first order fit was used for calibration. Linearity for the five-point calibration curves was excellent, with 28 of the 29 pesticides having r^2 values >0.995 at a range of 50-500 ppb. Proper calibration of imazalil was not possible due to its presence in the orange blanks.

Several pesticides were tentatively detected in the orange blanks. The identity of imazalil was confirmed spectrally by re-analyzing the sample in the full scan mode. The peak was beyond calibration range, and was therefore, not quantified. Imazalil is a post-harvest fungi-

Table 2. Calibration and Recovery Results

Analyte	r^2 Value	Average Recovery (%)	% RSD n=3
Methamidiphos	0.999	86	5
Dichlorvos	0.999	96	13
Acephate	0.998	94	4
Propoxur	0.999	98	6
Hexachlorobenzene	0.998	98	20
γ -BHC	0.998	105	14
Diazinon	0.999	105	19
Chlorothalonil	0.997	90	10
Methyl chlorpyrifos	0.999	108	13
Carbaryl	0.999	113	12
Dichlofluanid	0.999	102	8
Chlorpyrifos	0.999	109	11
p-Dichlorobenzophenone	0.999	103	7
Cyprodinil	0.999	103	12
Pencanazole	0.999	109	7
Tolyfluanid	0.998	91	13
Heptachlor epoxide	0.998	102	18
Captan	0.997	91	42
Thiabendazole	0.999	76	34
Folpet	0.999	111	13
cis-Chlordane	0.998	102	22
Imazalil	0.969	335	26
4,4'-DDE	0.998	100	17
Dieldrin	0.998	104	11
Endosulfan sulfate	0.998	102	12
Dicofol	0.995	151	27
cis-Permethrin	0.999	118	6
trans-Permethrin	0.999	112	5
Coumaphos	0.999	114	7

cide that is commonly used on citrus, so it is not unreasonable for it to be present. Peaks corresponding to the retention times of dicofol and captan were detected in the orange blank extracts but their low levels did not allow mass spectral confirmation in a subsequent full scan mode analysis. Because of their possible presence in the oranges prior to spiking, the recovery values for imazalil and dicofol were much higher than expected (335% and 151%, respectively).

Overall, recovery and precision were generally good averaging at $101.6 \pm 13.4\%$ for 27 of the 29 pesticides tested.

Conclusion

The QuEChERs method is an emerging extraction approach within area of food quality/safety analysis, and proved to be fairly simple and easy to perform. Table 3 lists the available sorbents and salts commonly used in dispersive SPE. The use of vials containing pre-weighed salts and SPE sorbent eliminated the need for a chemist to spend time performing this task. Sorbent weighing could be a time consuming bottleneck for food safety laboratories that need to perform hundreds of these extractions. For the GC-MS analysis, the SLB-5ms column provided adequate inertness and low bleed, allowing for low level detection of these pesticides.

Table 3. Available SPE Sorbents and Salts Commonly Used in Dispersive SPE¹

Florisil® (57209)	C18 SPE (52600-U)
NH ₂ SPE (57212-U)	PSA SPE (52738-U)
SAX SPE (57214-U)	ENVI-Carb™ (graphitized carbon black) (57210-U)
Sodium acetate (24,124-5)	Magnesium sulfate (23,039-1)
Sodium sulfate (23,859-7)	Sodium chloride (S 9888)

¹ Catalog numbers in parentheses are for bulk quantities of 100 g or greater.

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1. Communication From the Commission to the Council and the European Parliament on the Implementation of the Community Strategy for Endocrine Disrupters – A Range of Substances Suspected of Interfering With the Hormone Systems of Humans and Wildlife, Commission of the European Communities, June 14, 2001.
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3. O. Shimelis, A. Trinh, K. Stenerson, Recovery and Sample Cleanup of Pesticides in Spinach Using Supelclean ENVI-Carb II/PSA SPE, *Supelco Reporter*, Jun 2005; Vol. 23.3: 3-4.
4. M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenck, Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and “Dispersive Solid-Phase Extraction” for the Determination of Pesticide Residues in Produce, *J AOAC Int.*, Mar-Apr 2003; 86(2): 412-431.
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+ Featured Products

Description	Qty.	Cat. No.
SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm	1	28471-U

+ Related Products

Description	Qty.	Cat. No.
Supelclean™ ENVI-Carb II/PSA SPE Tubes		
300 mg/600 mg/6 mL	30	54058-U
500 mg/300 mg/6 mL	30	55119-U
500 mg/500 mg/6 mL	30	54067-U
500 mg/500 mg/20 mL	20	54217-U
Supelclean ENVI-Carb II/SAX/PSA SPE Tubes		
500 mg/500 mg/500 mg/12 mL	20	52574-U
Supelclean SAX/PSA SPE Tubes		
250 mg/250 mg/6 mL	30	52576-U
500 mg/500 mg/6 mL	30	52577-U
Supelclean PSA SPE Tubes		
200 mg/3 mL	54	52578-U
500 mg/6 mL	30	52579-U
SLB-5ms Capillary Columns		
10 m x 0.10 mm I.D., 0.10 µm	1	28465-U
15 m x 0.10 mm I.D., 0.10 µm	1	28466-U
20 m x 0.18 mm I.D., 0.18 µm	1	28564-U
12 m x 0.18 mm I.D., 0.30 µm	1	28566-U
30 m x 0.18 mm I.D., 0.30 µm	1	28575-U
20 m x 0.18 mm I.D., 0.36 µm	1	28576-U
30 m x 0.20 mm I.D., 0.20 µm	1	28513-U
30 m x 0.25 mm I.D., 0.10 µm	1	28467-U
15 m x 0.25 mm I.D., 0.25 µm	1	28469-U
60 m x 0.25 mm I.D., 0.25 µm	1	28472-U
15 m x 0.25 mm I.D., 0.50 µm	1	28577-U
30 m x 0.25 mm I.D., 0.50 µm	1	28473-U
60 m x 0.25 mm I.D., 0.50 µm	1	28474-U
30 m x 0.25 mm I.D., 1.0 µm	1	28476-U
15 m x 0.32 mm I.D., 0.25 µm	1	28557-U
30 m x 0.32 mm I.D., 0.25 µm	1	28482-U
30 m x 0.32 mm I.D., 0.32 µm	1	28532-U
15 m x 0.32 mm I.D., 0.50 µm	1	28597-U
30 m x 0.32 mm I.D., 0.50 µm	1	28484-U
30 m x 0.32 mm I.D., 1.0 µm	1	28487-U

! Related Information

For more information on Supelco Low Bleed SLB-5ms capillary columns, request T405130 (IKA) or visit sigma-aldrich.com/slb

For more information on SPE products from Supelco, see page 7.

Did you know...?

Supelco can provide vials containing pre-weighed amounts of the salts and SPE sorbent(s) mentioned in this article. For example, two sets of vials can be prepared to support the method described in this article. Each vial in the first set would contain 6 g anhydrous magnesium sulfate plus 1.5 g anhydrous sodium acetate. Each vial in the second set would contain 50 mg primary-secondary amine plus 150 mg anhydrous magnesium sulfate. Simply contact your local Sigma-Aldrich sales office to request a quotation for these, or any other custom dispersive SPE products.

Exactly Why Is Durability Important for Capillary Columns?

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Introduction

In two previous Reporter articles, the importance of selecting capillary columns with low bleed (1) and inert (2) characteristics were discussed. In this issue's article, the importance of using a durable column is investigated. In particular, how using a durable capillary column, such as Supelco SLB™-5ms, can save the user time and money.

Durability = Higher Maximum Temperature = Shorter Analysis Times

The type of capillary column that most readers may be familiar with consists of a thin film of liquid stationary phase coated on the inner wall of fused silica tubing. This type of column is termed fused silica open tubular (FSOT). The separation process for FSOT columns takes place through gas-liquid chromatography (GLC); the separation of analytes due to

the differences in their partitioning rates between a gas phase (the carrier gas) and a liquid phase (the stationary phase). In GLC, the gas chromatograph (GC) oven temperature can be used to affect partitioning, and hence retention. At lower oven temperatures, partitioning is toward the stationary phase. At higher oven temperatures, partitioning is toward the gas phase. Analyte boiling points must also be considered. Analytes with higher boiling points will tend to require more time to elute from the column than analytes with lower boiling points.

One of the many benefits of GC is the sheer number of compounds that can be separated in a single analysis. However, analysis of analytes with a wide range of boiling points can lead to long analysis times. One of the factors influencing analysis time is the maximum allowable operating temperature (MAOT) of the column. When compared to columns with lower MAOTs, a column with a higher MAOT offers more flexibility with regards to the temperature program and final temperature used to elute the analytes.

Figure 1. The Benefit of Higher Maximum Temperature

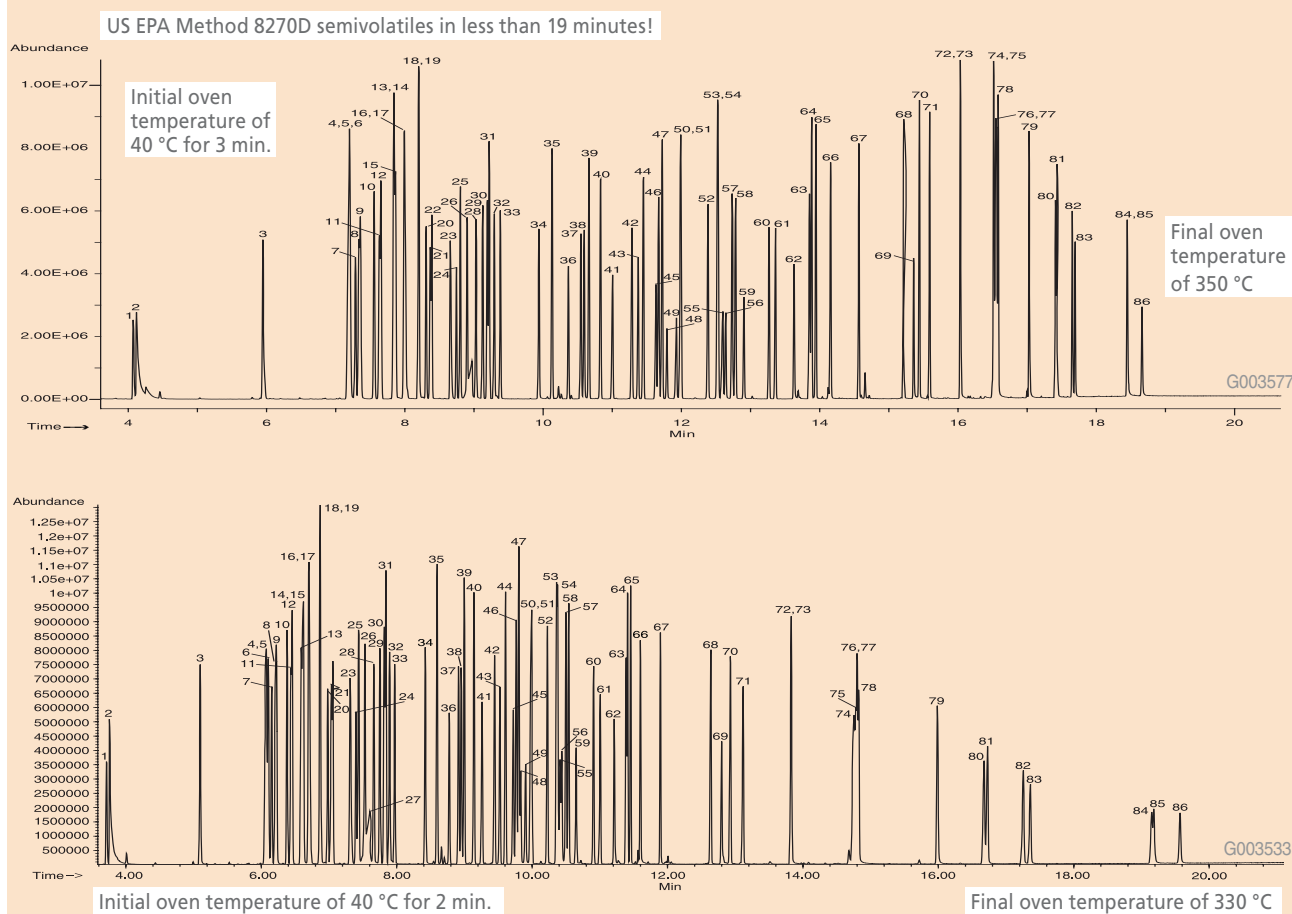
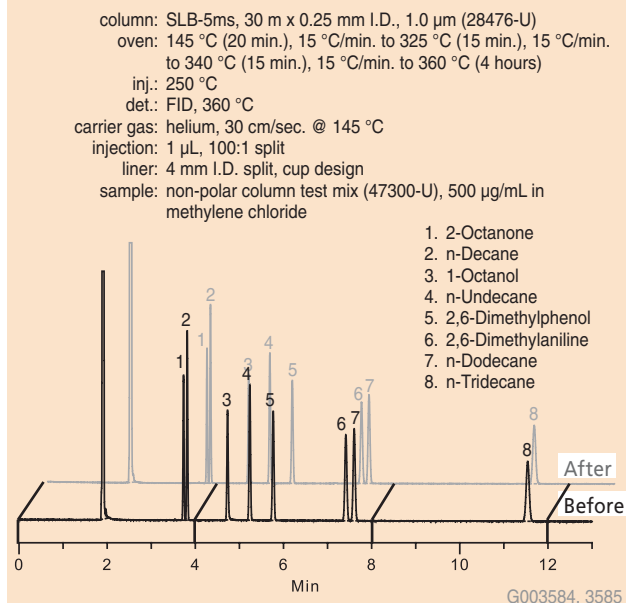


Figure 2. Column Evaluation Test Mix, Before and After



The two chromatograms shown in Figure 1 can be used to illustrate this point. The last eluting peak, benzo(g,h,i)perylene with a 500 °C boiling point, requires a high final oven temperature to elute with good peak shape and in a reasonable amount of time. By increasing the final oven temperature from 330 °C to 350 °C, partitioning of the analyte is driven towards the gas phase, resulting in less retention and a shorter analysis time. This is observed by a total analysis time of less than 19 minutes. The benefit to the analyst is that more billable samples can be analyzed in a given period of time. In addition, the shorter retention of later eluting peaks will result in less band broadening.

The more durable the column, the longer its usable life.

Durability = Less Phase Loss = Longer Column Life

As carrier gas passes through any capillary column at elevated temperature, phase is continuously being degraded, creating column bleed. Elevated temperatures hasten this degradation, seen as a baseline rise when using oven temperature programs. At some point, enough phase will have degraded so that resolution and retention are no longer acceptable. It is at this time that the column must be replaced.



Low bleed, inert, durable and consistent capillary GC columns for trace analyses.

To test column life, we cycled an SLB-5ms column through a rigorous oven temperature program that ended with a four-hour hold at 360 °C. This is 20 degrees above the published maximum isothermal temperature limit. While it is never recommended to operate above a column's MAOT, 360 °C was selected in an attempt to expedite degradation in column performance to prove our point. A 30 m x 0.25 mm I.D. x 1.0 μ m dimension was selected over a 30 m x 0.25 mm I.D. x 0.25 μ m dimension for this test because a column with a higher film thickness is more susceptible to phase damage from elevated temperature than a column with a lower film thickness. The column was evaluated for key performance parameters at the beginning of each cycle.

Figure 2 shows the isothermal portions of the chromatograms from the analyses of a column evaluation test mix from the first and the 20th cycle. The darker bottom chromatogram was generated prior to the start of the test, and the lighter top chromatogram after the 20th cycle (after the column had been exposed to 360 °C for a total of 76 hours). As expected, there was slight decrease in retention, but no change in peak shape, response, or resolution. Additionally, column bleed remained at a low level, indicating good phase stability.

Conclusion

Durability improvements incorporated into the SLB-5ms column result in shorter analysis times and longer column life. These improvements directly impact the time needs of analysts in addition to the financial needs of lab managers.

References

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! Related Information

For the complete US EPA Method 8270D instrument conditions and peak identifications, request T006391 (330 °C chromatogram) and/or T006398 (350 °C chromatogram).

For additional information on Supelco Low Bleed SLB-5ms capillary columns, request T406067 (IVH) or visit sigma-aldrich.com/slb

These publications are available in electronic form only. Be sure to include your email address on the request form.

Supelco OMI™ Indicating Purifiers: An Essential Part of Gas Delivery

Robert F. Wallace

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Introduction

Most chromatographers realize the importance of using gas purifiers as part of a gas delivery system for the supply of high purity carrier gas for gas chromatography (GC) instruments. Unfortunately, the replacement of these purifiers as they become spent may be overlooked, potentially leading to chromatographic problems. The dilemma facing chromatographers is determining the proper time to change-out purifiers. Changing too early does not allow for the maximum return of the purifier cost. Changing too late runs the risk of contaminants entering the GC. The solution is to install an indicating purifier downstream from all other purifiers.

Why Use an Indicating Purifier?

Capillary columns can degrade rapidly when exposed to oxygen or water vapor in the carrier gas. Even the highest purity carrier gas may contain trace amounts of these contaminants. Installing an indicating purifier as a final safeguard before the carrier gas enters the GC is recommended as part of any carrier gas delivery system. An indicating purifier is designed to give a visual indication of carrier gas contamination by color change. Contaminants may originate from the gas source (cylinder or generator) or from leaks in the system. By placing an indicating purifier downstream of all other gas purifiers, the user can determine if the purifiers installed upstream have expired and need replaced. Note that it is important to mount the indicating purifier in a prominent location so that it can be routinely inspected visually.

The Supelco OMI Purifier

The Supelco oxygen moisture indicating (OMI) purifier simultaneously and irreversibly reduces oxygen and water to less than 10 ppb in a carrier gas stream. Oxygen and water breakthrough are minimized because the OMI purifier is



P000245a

capable of handling oxygen surges of several hundred parts per million, or carrier gas delivery rates up to 1000 cc/minute (~150 psig). The OMI purifier can also remove carbon dioxide, carbon monoxide, most sulfur compounds, most halogen compounds, alcohols, phenols, and other trace impurities. Because of its capacity to remove a number of impurities, it provides point-of-use gas polishing in addition to a final visual assurance of the gas quality before it enters the GC. The main reason for the OMI purifier's effectiveness is the Nanochem® resin, a material developed for the demanding gas purity needs of the semiconductor manufacturing industry. Contaminants, even at concentrations below 1 ppm, progressively change the resin's color from black to light brown. Because Nanochem's color change can be seen at a glance through the OMI purifier's clear glass body and plastic safety guard, remaining purification capacity can easily be monitored. When the color change reaches the replacement mark, simply change the tube.

Manufacture/Installation of the OMI Purifier

The Supelco OMI purifier is packaged in an atmospherically controlled environment. Foil seals on each end of the purifier prevent contamination of the resin from oxygen and water. Installation into the beveled thorns inside the holder will produce a closed system for the carrier gas flow to assure the resin is not exposed to ambient contaminants. These thorns also allow the OMI purifier to maintain a leak free connection. The inert glass body prevents the diffusion of oxygen and water into the carrier gas stream, which helps eliminate unacceptable background noise that can affect highly sensitive detectors.

OMI Purifier Versus Other Purifiers

The OMI-2 purifier removes up to 0.16 grams of oxygen or 0.2 grams of moisture. Figure 1 shows that these capacities equal or exceed those of many non-indicating ambient temperature purifiers. Unlike many other purifiers, OMI purifiers are effective at the removal of contaminants other than just oxygen and moisture. In fact, an OMI purifier will remove all critical carrier gas contaminants, providing a carrier gas purity that is better than most certified gases.

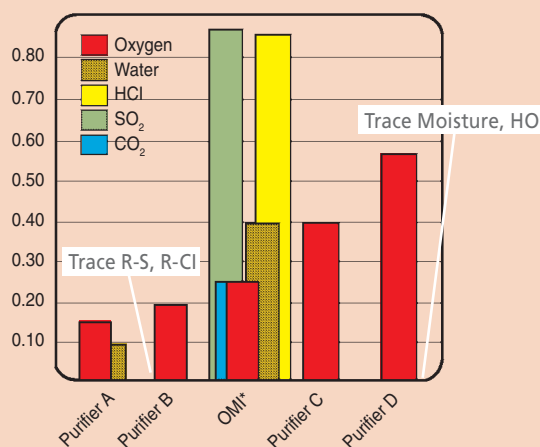
The OMI purifier should not be confused with the green oxygen indicating tubes that contain manganese oxide. These tubes will only indicate oxygen. Additionally, concentrated oxygen surges above 10 ppm will swamp the manganese oxide, allowing oxygen to breakthrough to the chromatographic system. Figure 2 shows comparisons of oxygen capacity. Given the low capacities of the green tube, they only signal an already existing problem. These

green tubes offer no other carrier gas protection than for oxygen and will not ward off impending problems you may encounter.

Conclusion

The use of an indicating purifier mounted in a visible location close to the GC allows the user to easily determine when purifier change-out should occur. This will help insure a constant supply of clean carrier gas. The Supelco OMI purifier not only outperforms other indicating purifiers, it also outperforms many non-indicating purifiers. Connecting the OMI purifier downstream from other Supelco gas purifiers will give a purification system designed to economically give maximum gas purity, providing quality gas for excellent detector response.

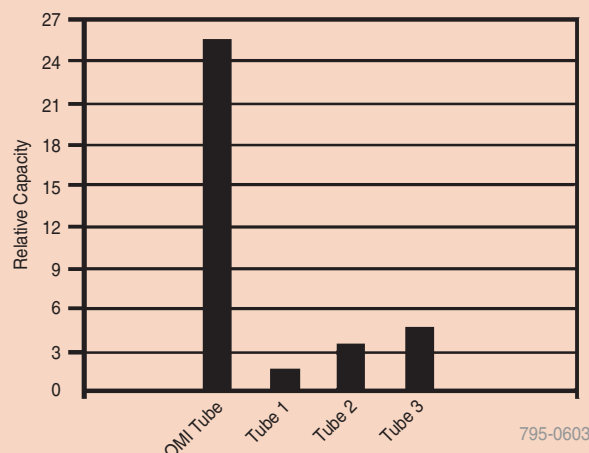
Figure 1. Comparison of Purifier Capacities of Ambient Temperature Purifiers



*Also purifies CO, R-S, R-X, C=O, ROH. Capacities not additive.

795-0602

Figure 2. An OMI Purifier Traps More Oxygen Compared to Green Indicating Purifiers



795-0603

Featured Products

Description	Cat. No.
OMI-2 Purifier, 15 cc tube only ¹	23906
OMI-2 Holder, 1/8 in. fittings	23917
OMI-4 Purifier, 90 cc tube only ¹	23909
OMI-4 Holder, 1/8 in. fittings	23926

¹ First time users must order the corresponding holder. Holders are reusable.

Related Products

Description	Cat. No.
Swagelok® Reducer 1/4 in. to 1/8 in., stainless steel	21517
Precleaned Copper Tubing 1/8 in. O.D. x 0.065 in. I.D., 50 ft. 1/4 in. O.D. x 0.190 in. I.D., 50 ft.	20488 20489
Stainless Steel Tubing 1/8 in. x 0.085 in. I.D., 50 ft. 1/4 in. x 0.209 in. I.D., 50 ft.	20526-U 20527
Heavy Duty Tubing Cutter	20425-U

Related Information

For more information on gas purifiers, request Bulletin 918 (BIT), see the Supelco catalog, or visit sigma-aldrich.com/supelco.

Performance Tip

Change Gas Cylinders Early to Extend Purifier and Column Life

Ron Shawley
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The tank pressure at which you decide to change your gas cylinders may affect the quality of your carrier gas. The lower the pressure, the greater the risk of causing problems with your chromatography system and your column due to higher levels of tank contaminants.

When the pressure in your gas cylinder drops below 500 psig, you risk drawing higher levels of contaminants from liquids that may be present in the bottom of the cylinder. You increase the chance of adding oxygen, moisture, and other contaminants to your carrier gas. These contaminants will shorten the lifetime of your purifiers and column.

Additionally, two-stage pressure regulators do not operate effectively at pressures less than 300psig. Below 300psig, your two-stage regulator begins to function more like a single-stage regulator, resulting in variable pressure. Without constant pressure, retention times will vary and irreproducible chromatography will be the result.

Supelco recommends that you change your gas cylinders when they drop to a pressure between 500 and 300psig. Avoid the risk of using impure carrier gas and causing more costly problems.

For more information on gas management, request Bulletin 898 (AYW).

New Standards!

US EPA SOM01.1 Deuterated Monitoring Compounds

Supelco now provides Deuterated Monitoring Compound (DMC) standards for use by all commercial testing laboratories involved in the U.S. Environmental Protection Agency Contact Laboratory Program and following methodology outlined under SOM01.1 (5/26/05). This initial offering includes volatile and semi-volatile compounds in two different concentrations.

These high purity analytical standards have been carefully formulated to ensure product stability, in particular ketone analytes. All deuterated raw materials and solvents are screened for identity and purity. The mixtures are gravimetrically prepared and then quantitated using GC-MS. Each DMC standard is supplied with a certificate of analysis. Free data packets are available upon request.

US EPA SOM01.1 Deuterated Monitoring Compounds

Description	Concentration	Solvent	Pkg size	Cat. No.
SOM01.1 Deuterated Monitoring CPD (DMC) Mix <i>Fluoranthene-d₁₀</i> <i>2-Methylnaphthalene-d₁₀</i>	2000 µg/mL	Methylene chloride	1 x 1 mL	47196-U
SOM01.1 HC Deuterated Monitoring CPD (DMC) Mix <i>Fluoranthene-d₁₀</i> <i>2-Methylnaphthalene-d₁₀</i>	4000 µg/mL	Methylene chloride	1 x 1 mL	47197-U
SOM01.1 Volatiles Deuterated Monitoring CPD (DMC) Mix	2000 µg/mL 2000 µg/mL	Methanol-d ₄ :D ₂ O Methanol-d ₄ :D ₂ O	10 x 1 mL 1 x 1 mL	47193-U 47254-U
<i>Benzene-d₆</i> <i>2-Butanone-d₅</i> <i>cis & trans-1,3-Dichloropropene-d₄</i> <i>Chloroethane-d₅</i> <i>Chloroform-d₁</i> <i>1,2-Dichlorobenzene-d₄</i>	<i>1,1-Dichloroethene-d₃</i> <i>1,2-Dichloroethane-d₄</i> <i>1,2-Dichloropropane-d₆</i> <i>1,4-Dioxane-d₃</i> <i>2-Hexanone-d₅</i>	<i>1,1,2,2-Tetrachloroethane-d₂</i> <i>Toluene-d₈</i> <i>Vinyl chloride-d₃</i>		
SOM01.1 Semivolatiles Deuterated Monitoring CPD (DMC) Mix	2000 µg/mL 4000 µg/mL	Methylene chloride Methylene chloride	1 x 1 mL 1 x 1 mL	47194-U 47195-U
<i>Acenaphthylene-d₈</i> <i>Anthracene-d₁₀</i> <i>Benzo(a)pyrene-d₁₂</i> <i>Bis-(2-chloroethyl)ether-d₈</i> <i>4-Chloroaniline-d₄</i> <i>2-Chlorophenol-d₄</i>	<i>2,4-Dichlorophenol-d₃</i> <i>Dimethyl Phthalate-d₆</i> <i>4,6-Dinitro-2-methylphenol-d₂</i> <i>Fluorene-d₁₀</i> <i>4-Methylphenol-d₈</i> <i>Nitrobenzene-d₅</i>	<i>2-Nitrophenol-d₄</i> <i>4-Nitrophenol-d₄</i> <i>Phenol-d₅</i> <i>Pyrene-d₁₀</i>		

New!

Sigma-Aldrich Analytical Standards Web Site

Our new Analytical Standards web site makes it faster and easier to locate certified reference materials and chemical standards for your specific applications. The web site features the Standards Explorer, a standards-only search engine, where you can search by product number, product name, chemical name, CAS#, molecular weight,

or agency/method, (e.g EPA 8270, ASTM 2887). We've also included an electronic custom standards quote request form, links to government agencies and their methodologies, as well as standards accessories we believe will make your job easier. Visit us at sigma-aldrich.com/standards to see for yourself.



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
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