

A Technical Newsletter for Analytical & Chromatography

The Reporter

Issue 27, July 2007, International

SUPELCO

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Innovation!

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Sigma-Aldrich: INNOVATION!

Dear Colleague:

Innovation and Customer-Centricity

Innovative companies capture attention by providing unique solutions or by creating new markets. Customer-centric companies put into action the knowledge that their success is inextricably linked to satisfying customer needs. Within Sigma-Aldrich's Analytical and Chromatography initiative, we have placed heavy emphasis on increasing our portfolio of innovative new products not only by being, but because of being customer-centric.



Wayne K. Way
HPLC Market Segment Manager

A Robust Spirit of Innovation

In the past few years, we have taken on many exciting new and unique products and technologies. I'd like to take a moment here to describe a few.

Ascentis® Express HPLC Columns

Imagine an HPLC column with the efficiency of sub-2 µm particles but at half the pressure or, even more intriguing, twice the efficiency at comparable pressures. Based on patented Fused-Core™ particle technology, Ascentis Express provides rapid analysis, high resolution and high sensitivity on all HPLC, UHPLC and LC-MS instruments.

Supelco Astec Chiral Chromatography

In 2006 we acquired Astec and its innovative line of CHIROBIOTIC™, CYCLOBOND™ and CHIRALDEX™ columns. By acquiring Astec, we've acquired not only their existing line, but the pipeline of exciting new products from this innovative leader in chiral chromatography.

SupelMIP™ SPE Products

Analysts using SPE no longer have to put up with long method development time, low recoveries and high background interferences that reduce sensitivity, even when dealing with ultra-complex matrixes. Innovative, new SupelMIP SPE products, based on unique molecularly imprinted polymer (MIP) technology, have specific affinity for single compounds or classes of commonly-analyzed compounds.

radiello® Passive-Diffusive Air Monitors

Designed for monitoring both workplace and outside environments, Radiello samplers are based on an innovative, patented radial design that provides significantly faster sampling rates than classical diffusive samplers.

While these products represent significant innovative advances, our journey is by no means over. We will continue to develop innovative solutions that keep pace with your continually evolving needs and increasing expectations.

Sincerely,

Wayne K. Way
HPLC Market Segment Manager

Improving HPLC Sample Throughput using Ascentis Express Fused-Core Technology Columns

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Introduction

Today, increasing pressure to process more samples per unit time has placed emphasis on developing faster separations. However, any improvement in throughput cannot come with sacrifices in resolution, sensitivity or ruggedness. Supelco has answered this challenge by introducing Ascentis® Express HPLC columns, which deliver speed and resolution on conventional as well as ultra-high pressure liquid chromatography (UHPLC) systems.

Ascentis Express: breakthrough HPLC technology

The radically new Fused-Core™ HPLC particle technology behind Ascentis Express permits dramatic reductions in analysis time compared to conventional HPLC columns, without sacrificing resolution or column ruggedness.

Dramatic improvements in speed and throughput

A unique combination of high column efficiency and high column permeability gives Ascentis Express columns a dramatic improvement in speed and sample throughput:

- **High Column Efficiency**

Twice the efficiency of 3 µm particles and thrice the efficiency of 5 µm particles allow for shorter columns
Comparable to sub-2 µm particles, but at half the back pressure

- **High Column Permeability**

Half of the back pressure generated by sub-2 µm particles permits faster flow rates and longer columns for even more efficiency

High column efficiency and high throughput via the kinetic advantage

Highly efficient columns deliver more plates per meter, which means shorter columns and subsequently higher flow rates can be used. Compared to conventional 3 and 5 µm totally porous particles, the 2.7 µm Ascentis Express gives two- and three-times the efficiency, respectively. Therefore, shorter Ascentis Express columns can be run at higher flow rates for fast separations. Compared to conventional sub-2 µm particles, Ascentis Express gives equivalent efficiency, but it can be run at much higher flow rates because of its lower backpressure.

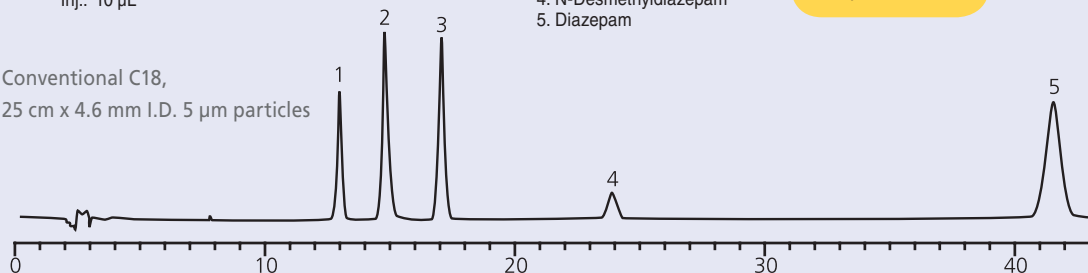
Figure 1. Increase sample throughput by using Ascentis Express

Mobile phase: 65:35 water:acetonitrile
Flow rate: 1 or 1.5 mL/min. (as indicated)
Temp. ambient
Det.: UV at 254 nm
Inj.: 10 µL

Analytes:
1. Oxazepam
2. Alprazolam
3. Cloazepam
4. N-Desmethyldiazepam
5. Diazepam

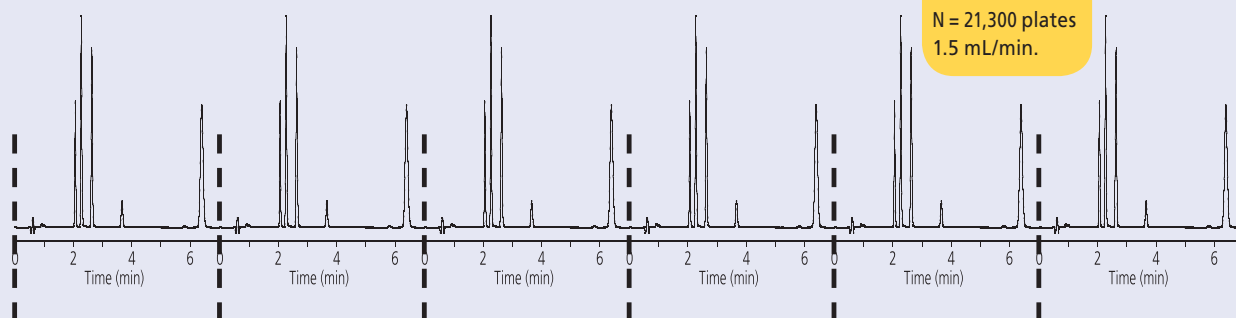
Run time = 43 min.
N = 22,150 plates
1 mL/min.

Conventional C18,
25 cm x 4.6 mm I.D. 5 µm particles



Ascentis Express C18, 10 cm x 4.6 mm I.D., 2.7µm particles.

Run time = 4.3 min.
N = 21,300 plates
1.5 mL/min.



Up to 6-fold increase in throughput with Ascentis Express

Comparable plates on 10 cm Ascentis Express as on a 25 cm 5µm column

The improvement in throughput on Ascentis Express is demonstrated in Figure 1. Although the conventional, 25 cm, 5 µm column and the 10 cm Ascentis Express column have approximately the same total plates per column (~22,000), separation on the shorter Ascentis Express column is much faster. A dramatic six-fold improvement in sample throughput was realized on the Ascentis Express column using a conventional HPLC system. Further increases in throughput on the Ascentis Express column can be achieved by running the column at higher flow rates on higher pressure instruments (up to 9,000 psi).

The improved kinetics from the physical structure of the Ascentis Express particle leads to efficiencies that are higher than predicted by particle size alone. Called "Fused-Core™," the 2.7 µm Ascentis Express particle comprises a solid 1.7 µm silica core surrounded by a 0.5 µm porous silica layer. For a visual depiction, please see page 5. The solid core prevents solutes from diffusing as deeply into the Ascentis Express particle as they can in a totally porous particle, thus reducing band broadening.

Another possible contribution to the high efficiency is the fact that Ascentis Express particles have a very narrow size distribution which leads to very uniform and densely packed columns. This also has economic implications: Because of the uniform, 2.7 µm particle size, 2 µm pore size frits are used in Ascentis Express columns. These larger pore size frits do not foul as easily as do the 0.5 µm frits that are necessary to retain the sub-2 µm and conventional 3 µm particles.

High column permeability permits higher flow rates

Ascentis Express particles generate significantly lower back pressure and can be operated at higher flow rates than conventional sub-2 µm particles. This is a result of the relationship between pressure and particle diameter:

$$P \propto 1/d_p^2$$

Compared to 1.8 µm particles, the 2.7 µm Ascentis Express particles give $(1.8/2.7)^2$ -fold less or roughly half the back pressure when packed into the same column dimensions and run under the same conditions. This means that Ascentis Express delivers the same efficiency as sub-2 µm particles (as discussed above), but at half the pressure. This in turn has three important implications:

- First, with Ascentis Express, UHPLC-like performance, in terms of both efficiency and speed, is achievable on conventional HPLC systems.
- Second, you can run Ascentis Express columns at twice the flow rate as sub-2 µm columns. Pressure and efficiency will be the same, but analysis time will be cut in half.

- Third, because Ascentis Express delivers twice the efficiency at the same pressure, you can use longer columns and actually exceed by two-fold or more the efficiency possible from sub-2 µm columns.

Using Ascentis Express to improve the speed of a current analysis

- Obtain the same or more separating power as a 25 cm column packed with 5 µm particles, but on a shorter, faster 10 cm Ascentis Express column. Realize an immediate four-fold time savings.
- Obtain the same or more separating power as a 15 cm, 3 µm column, but on a shorter, faster column 7.5 cm Ascentis Express column. Realize an immediate two-fold time savings.
- Obtain additional time savings and throughput by increasing the flow rate.

Conclusion

Ascentis Express is the ideal choice for HPLC analysts interested in increasing sample throughput while maintaining or even improving resolution. By reducing solute dispersion, the unique Fused-Core technology gives Ascentis Express a kinetic advantage over conventional sub-2, 3 and 5 µm particles. Its higher column permeability compared to sub-2 µm particles means that Ascentis Express can achieve UHPLC-like performance on conventional HPLC systems. Under UHPLC conditions, Ascentis Express can exceed the efficiency possible on sub-2 µm columns because longer columns can be used.

For more information, please visit our Web site:
www.sigma-aldrich.com/express

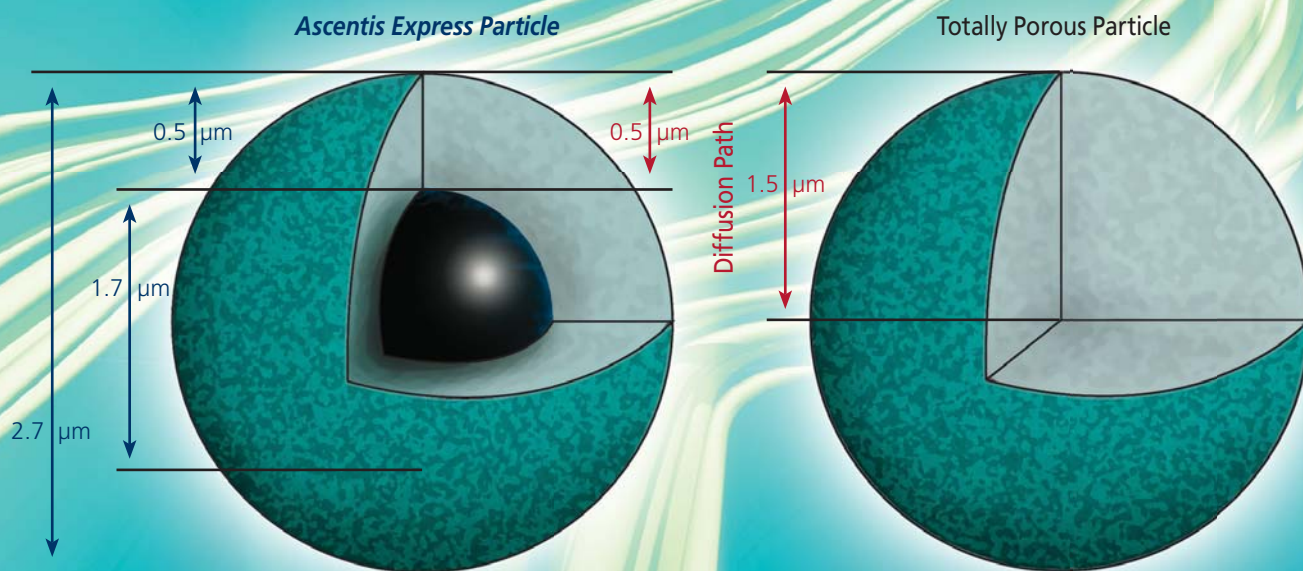
ID (mm)	Length (cm)	Ascentis Express C18	Ascentis Express C8
2.1	3	53802-U	53839-U
2.1	5	53822-U	53831-U
2.1	7.5	53804-U	53843-U
2.1	10	53823-U	53832-U
2.1	15	53825-U	53834-U
3	3	53805-U	53844-U
3	5	53811-U	53848-U
3	7.5	53812-U	53849-U
3	10	53814-U	53852-U
3	15	53816-U	53853-U
4.6	3	53818-U	53857-U
4.6	5	53826-U	53836-U
4.6	7.5	53819-U	53858-U
4.6	10	53827-U	53837-U
4.6	15	53829-U	53838-U

Fused-Core is a trademark of Advanced Materials Technology, Inc.
Ascentis is a trademark of Sigma-Aldrich Corp.

Ascentis[®] Express

Extreme Performance on **Any** LC System

Fused-Core Structure of Ascentis Express Compared to Totally Porous Particles



Ascentis[®] Express
HPLC Columns

"Based on innovative Fused-Core[™] particle technology, Ascentis Express provides the high speed and high efficiency of sub-2 μm particles, but at approximately half the backpressure for the same column length."

Interested in Boosting your HPLC Sample Throughput?

Try a risk-free 30 day trial offer today!

Contact our technical service at EurTechServ@sial.com and quote U37. Offer valid through August 31st 2007.

Improved Analysis of Simple Sugars Using apHera NH₂ HPLC Columns

Hugh M. Cramer and Ric Cone
ric.cone@sial.com

Chromatographic analysis of simple sugars is important to the food and beverage, pharmaceutical and biotech industries. Effective separations of sugars can be challenging because these compounds are highly polar, uncharged and lack a chromophore.

The preferred mode of HPLC separation for simple sugars is hydrophilic interaction chromatography (HILIC), an aqueous-organic variation of normal-phase chromatography where water is used as the strong mobile phase (*i.e.* increasing water percentage decreases retention). HILIC provides the advantage of retaining highly polar analytes that are difficult to separate on reversed-phase columns such as C18 (1-3). Simple sugars are well suited for this type of chromatography due to their polar nature.

Silica bonded with an aminopropyl silane is a popular phase for HILIC separation of sugars. Aminopropyl groups bonded to silica, however, tend to be susceptible to hydrolysis and are thus less stable than other silica-based

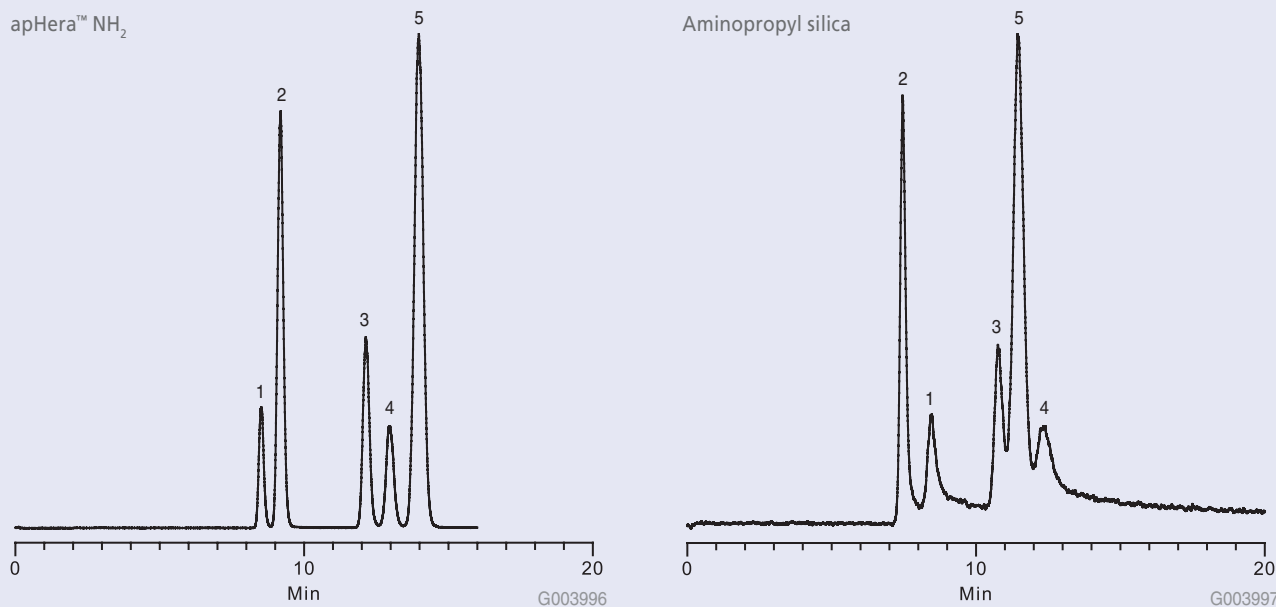
phases. The propensity of the aminopropyl ligand toward ionization also limits suitability of the phase for mass spectrometric (MS) detection.

Polymeric columns bonded with aminopropyl groups offer improved stability and MS compatibility over silica-based amino phases. The apHera™ NH₂ column, now available from Supelco/Sigma-Aldrich, is based on covalently bonded polyamine to a copolymer that offers stability from pH 2-12, mechanical and chemical strength and high column efficiency. Figure 1 compares the separation of several simple sugars on a polymeric polyamine (apHera NH₂) column, versus a silica-based aminopropyl column. The apHera NH₂ column shows significantly higher efficiencies as exhibited by the sharper peaks it produces. In addition, the two phases exhibit different selectivity indicating that the surface plays a significant role in the chromatographic separation mechanism. In this case it is assumed that the selectivity difference is due to strong secondary interactions between the sugar analytes and surface silanol groups on the aminopropyl column.

Figure 1. Comparison of five simple, underivatized sugars separated on apHera NH₂ vs. aminopropyl silica columns

Columns: apHera NH₂, 15 cm x 4.6 mm I.D., 5 μm particles (56401AST)
aminopropyl silica column, 15 cm x 4.6 mm I.D., 5 μm particles
mobile phase: 20:80, water:acetonitrile
flow rate: 1.0 mL/min.
temp.: 25 °C
det.: ELSD, 45 °C, 3.5 psi nitrogen
injection: 10 μL
sample: 500 μg/mL in 30:70, water:acetonitrile

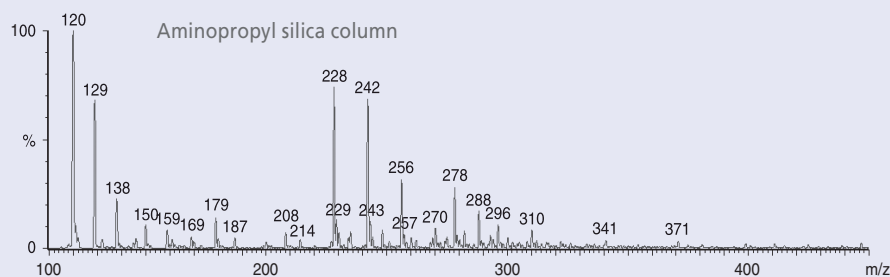
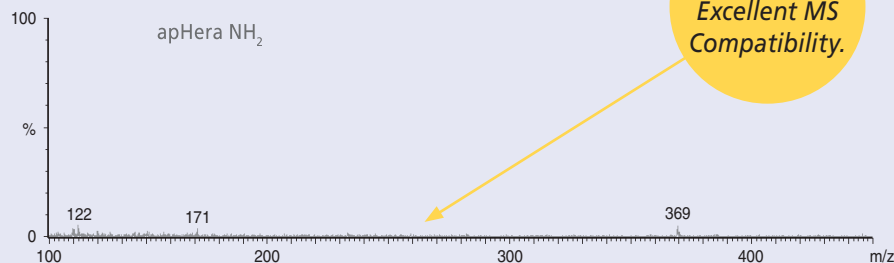
1. D-(-)-arabinose
2. D-(+)-xylose
3. D-(+)-mannose
4. D-(+)-galactose
5. D-(+)-glucose



apHera™ HPLC columns are products from Advanced Separation Technologies, Inc. (Astec), now a member of the Sigma-Aldrich Group.

Figure 2. Analysis of column effluent by MS

columns: 25 cm x 4.6 mm I.D., 5 µm particles
 mobile phase: 20:80, 25 nM sodium chloride in water:acetonitrile
 flow rate: 1.0 mL/min.
 temp.: 25 °C
 det.: ESI (+), scan range 110 – 450 m/z



MS bleed profiles for both the aminopropyl silica and apHera NH₂ columns are shown in Figure 2. Depicted are accumulated background scans over a one-minute period with each of the respective columns in-line. It is apparent that the aminopropyl silica-based phase exhibits high levels of background ions that may interfere with spectral analysis, make trace components difficult to find and ultimately cause source fouling/instrument down time. The background observed for the apHera NH₂ polymeric phase is very low, demonstrating that the phase is much more compatible with MS detection.

In summary, the apHera NH₂ polymeric column is an excellent choice for the separation of closely-related polar analytes such as simple sugars using HILIC chromatography. These columns demonstrate higher efficiencies than their silica counterparts for this application, and the extended pH range and low bleed offer a much wider range of applications and improvements for use in LC-MS. Other potential applications include derivatized sugars, complex carbohydrates, oligonucleotides, glycopeptides, amino

acids, peptides, and polar organic acids and bases (3). For example, the apHera NH₂ column was recently used in an LC-MS assay for taurine and methionine in a carbohydrate-rich energy drink, with detection limits as low as 20 µg/L for taurine, and 50 µg/L for methionine (4).

References

1. A. Alpert, J. Chromatogr., 499, 177 (1990).
2. S. Churms, J. Chromatogr. A 720, 75 (1996).
3. U.D. Neue ed. HPLC Columns, Wiley-VCH, New York, 1997, 217-223.
4. M. de Person et. al., J. Chromatogr. A. 1081,174 (2005).

Featured Products

Length (cm)	I.D. (mm)	Cat. No.
apHera NH₂		
4.6	15	56401AST
4.6	25	56403AST
2.0	15	56400AST

Related Products

Length (cm)	I.D. (mm)	Cat. No.
apHera C18		
4.6	15	56102AST
4.6	25	56103AST
2.0	15	56100AST
apHera C8		
4.6	15	56202AST
4.6	25	56203AST
apHera C4		
4.6	15	56302AST
4.6	25	56303AST

Related Information

For more information please request the Astec Chromatography Product Guide (JCI). For further discussion regarding HPLC columns for carbohydrate analysis, request T195887 (AIU).

Avoid LC-MS Source Contamination by Using High Quality CHROMASOLV Solvents and Blends

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Analytical sensitivity and specificity are two fundamental requirements that have led to the adoption of LC-MS as a preferred analytical tool for chromatographic separation and detection. To achieve this high level of performance, the use of purified and well-specified solvents is required. A major source of contamination in LC-MS comes from lower purity standard HPLC grade solvents as they are constantly being introduced into the instrument source. The use of contaminant free solvents is crucial in keeping the mass spec source clean. Should the MS source become fouled by the use of impure solvents, problems such as poor detection, complex spectral analysis and instrument downtime may result. The use of purified solvents can reduce or eliminate these problems.

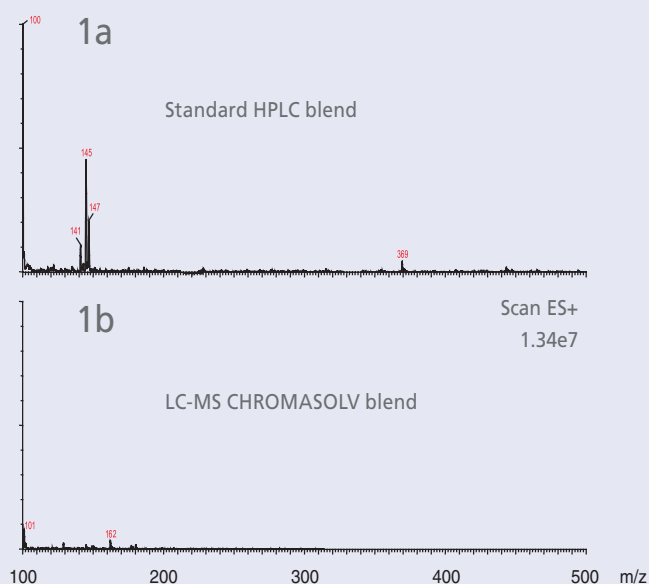
A study was recently conducted to demonstrate solvent purity in an MS source. Starting with a clean MS source, a high purity LC-MS CHROMASOLV® grade mix of 50:50, water: acetonitrile was run through the MS and a three minute scan from m/z 100-500 was collected. Next, a standard HPLC

grade (lower purity brand) of 50:50, water:acetonitrile was run overnight. The system was then re-equilibrated with the previously used high purity LC-MS CHROMASOLV grade mix of 50:50, water:acetonitrile and a repeat three minute scan from m/z 100-500 was collected.

Comparison of the before and after scans showed source contamination from running the standard HPLC grade brand solvent. The results of this experiment are shown in Figure 1. Figure 1a shows the mass spectrum obtained of the high purity LC-MS CHROMASOLV grade mix of 50:50, water:acetonitrile after running overnight with the standard HPLC grade brand of 50:50, water: acetonitrile. Figure 1b is the mass spectrum obtained after running overnight with LC-MS CHROMASOLV grade mix of 50:50, water:acetonitrile. Note that both spectra are on the same scale and MS conditions were generic starting conditions. The m/z ratio range was 100-500. Note some contamination in Figure 1a after running the lower purity solvent. To examine the spectra more closely, Figures 2-4 show the same scans as Figures 1a and 1b, only the m/z ratio ranges have changed to 150-500 (Figure 2), 200-500 (Figure 3) and 300-500 (Figure 4). Note the

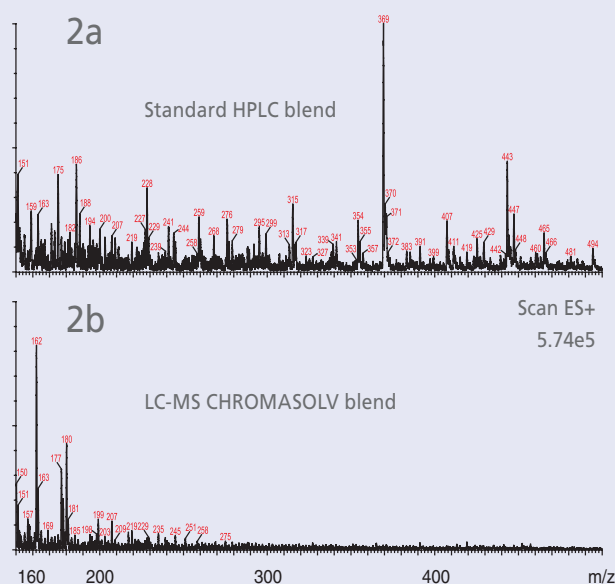
“a” Figures. Spectrum after overnight run with standard HPLC grade blend (lower purity brand).

Figure 1. Mass spectrum of 50:50, water:acetonitrile blend, m/z range 100-500 (same scale for 1a and 1b)



“b” Spectrum after overnight run with high purity LC-MS CHROMASOLV blend.

Figure 2. m/z range 150-500



contamination in the top mass spectrum of each when the spectra was obtained after running the lower purity standard HPLC grade brand of 50:50, water:acetonitrile.

This study demonstrates that lower quality solvents contaminate the source. Higher quality LC-MS CHROMASOLV® solvents do not foul the source and minimize chance for lower detection, complex spectral analysis and instrument downtime.

The LC-MS CHROMASOLV solvents and blends offered by Sigma-Aldrich are prepared with unsurpassed attention to quality for meeting the purity standards of your analysis and the LC-MS system. To see the complete line of CHROMASOLV solvents, additives and blends for LC-MS and other sensitive analytical applications, visit our Web site: www.sigma-aldrich.com/lc-ms-solvents

Figure 3. m/z range 200-500

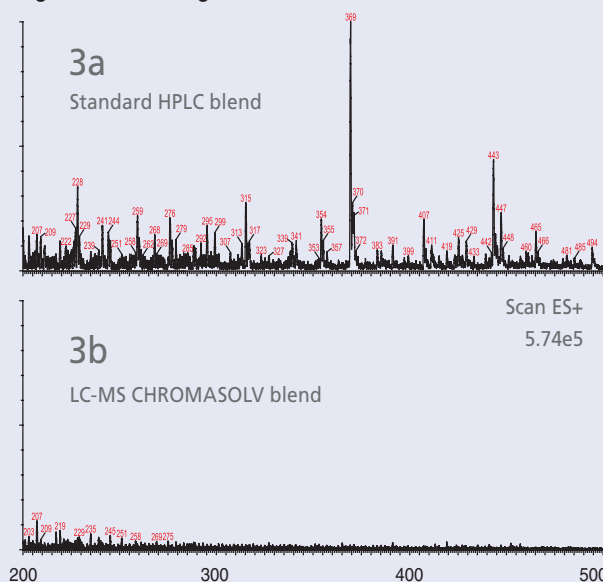
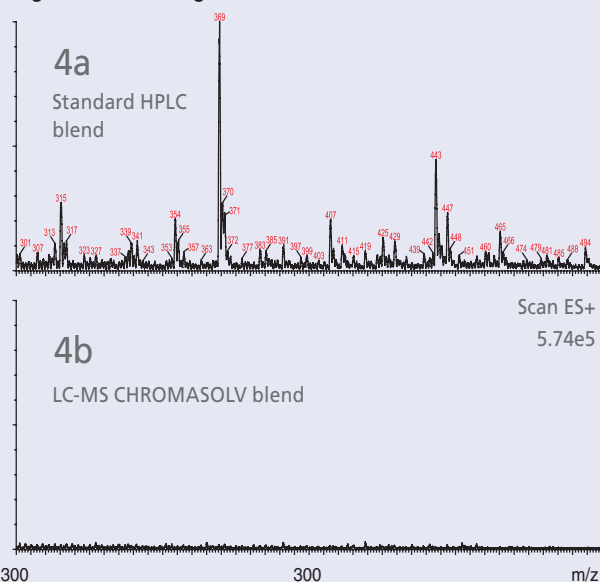


Figure 4. m/z range 300-500



NEW LC-MS Post Column Flow Splitters

Supelco now offers QuickSplit™ flow splitters that are elegant in design and easy-to-use. The QuickSplit flow splitters come in a variety of fixed and adjustable flow split ratios. Split ratios are created by two or more fluid resistors, which can be purchased separately and are interchangeable.

The primary benefits of QuickSplit™ flow splitters are:

- Eliminates tedious adjustments to capillary tubing for split ratio optimization
- Split ratios are stable and reproducible and are not affected by changes in viscosity or pressure
- Ultra low dead volume design
- Easy-to-use interchangeable fluid resistors
- Rugged stainless steel construction



Description	Cat. No.
HPLC Post Column Flow Splitters - Fixed	
Split Ratio = 20:1	56624-U
Split Ratio = 10:1	56625-U
Split Ratio = 5:1	56626-U
Split Ratio = 3:1	56627-U
Mounting Bracket for HPLC Post Column Flow Splitters - Fixed	56630-U
HPLC Post Column Flow Splitter - Adjustable	
Split Ratio = 1:1 to 20:1	56629-U
HPLC Post Column Resistor Sets - Binary	
Split Ratio = 20:1	56631-U
Split Ratio = 10:1	56632-U
Split Ratio = 5:1	56633-U
Split Ratio = 3:1	56634-U

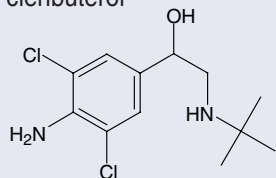
Molecularly Imprinted Polymer SPE Increases Sensitivity for the Extraction and Analysis of Clenbuterol from Urine

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Introduction

Clenbuterol (Figure 1) is a beta-agonist known for its growth-promoting properties whereby use of the drug induces significant weight gain by increasing the proportion of muscle mass to fat. Although the US Food and Drug Administration, US Department of Agricultural and European Union (1) have banned the use of clenbuterol for humans and livestock, illegal use of the

Figure 1. Structure of clenbuterol



drug still frequently occurs. For example, clenbuterol is widely used among body builders and athletes due to its anabolic effects. Two Olympic athletes were banned from the 1992 Barcelona Olympic Games due to the

drug, and two swimmers tested positive for the drug in the 2002 World Championships in Perth, Australia. Clenbuterol is readily used by farmers to give show animals a competitive advantage as well. In 1995, the US Department of Agriculture issued a press release stating that both they and the FDA will be taking enforcement actions against the use of clenbuterol (2). In the 1990s, numerous outbreaks in clenbuterol food poisoning arose throughout Europe (3). As recently as September 2006, over 300 cases of food poisoning occurred in Shanghai from the consumption of clenbuterol-contaminated pork (4).

Due to the potential health risks and competitive advantage associated with clenbuterol's use in livestocking and human performance enhancement, residue screening programs for it are conducted worldwide. It is therefore critical to develop a highly selective and sensitive analytical assay to monitor clenbuterol residues in difficult biological matrixes such as urine, retina, tissues, etc.

In this report, we discuss the use of a molecularly imprinted polymer technology (SupelMIP™) specifically designed for the selective extraction of trace levels of clenbuterol from urine for subsequent LC-MS-MS analysis. The technique was compared against a published method using a conventional hydrophilic polymer SPE phase (5).

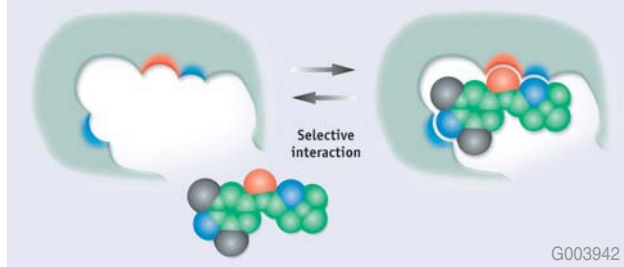
Molecularly Imprinted Polymers

Molecularly imprinted polymers (MIPs) are a class of highly cross-linked polymer-based molecular recognition elements engineered to bind one target compound or a class of structurally related compounds with high selectivity. Selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guides the formation of specific cavities that are sterically and chemically complementary to the target analyte(s).

As a result, multiple interactions (e.g., hydrogen bonding, ionic, van der Waals, hydrophobic) can take place between the MIP cavity and analyte functional groups (Figure 2). The strong retention between a MIP phase and its target analyte(s) allows for the use of exhaustive wash procedures during solid phase extraction that results in superior sample cleanup prior to analysis.

Extraction of Clenbuterol from Urine

Figure 2. Visual depiction of MIP binding site



In this study, an extraction method using SupelMIP SPE – Clenbuterol was compared against a published method using conventional hydrophilic polymer SPE phase (5). Table 1 describes the two extraction procedures.

SupelMIP SPE Offers Reduced Background Resulting in Greater Sensitivity

Clenbuterol was extracted from urine using both molecularly imprinted polymer SPE and conventional hydrophilic polymer SPE via the procedures described in Table 1. Extracts were further analyzed via LC-MS-MS. Inspection of the LC-MS-MS chromatograms (MRM) depicted in Figure 3, shows that blank urine samples extracted with the SupelMIP protocol gave low background. In contrast, the conventional hydrophilic polymer SPE procedure co-extracted matrix interferences

Table 1. Comparison of SupelMIP SPE – Clenbuterol method and conventional hydrophilic polymer SPE method

SupelMIP SPE – Clenbuterol Method

Sample Pre-Treatment:

Human urine samples were spiked with 0.0 (blank), 0.04, 0.1, 0.5, and 1.0 ng/mL clenbuterol. Spiked and blank urine samples centrifuged at 3000 rpm for 10 min., and the resultant supernatant was diluted 1:1 (v/v) with 25 mM ammonium acetate buffer, pH 6.5

SPE Procedure:

SupelMIP SPE – Clenbuterol, 25 mg/10 mL (LRC) (Cat. No. 53201-U)

1. Condition and equilibrate MIP phase with 1 mL methanol, 1 mL DI water, and 1 mL 25 mM ammonium acetate, pH 6.5
2. Load 1 mL pre-treated sample on to the cartridge.
3. Wash (elute interferences) using the following wash scheme: 1 mL DI water followed by 2 min. vacuum; 1 mL 2% acetic acid in acetonitrile; 1 mL 0.5 M ammonium acetate, pH 5; 1 mL 70% acetonitrile followed by 2 min. vacuum
4. Elute clenbuterol with 2 x 1 mL 1% TFA in methanol. Apply gentle vacuum between each fraction.
5. Evaporate under nitrogen and reconstitute with 1 mL LC mobile phase prior to LC-MS-MS analysis

Published Clenbuterol Method Using Conventional Hydrophilic Polymer SPE Phase (5)

Sample Pre-Treatment:

Human urine samples were spiked with 0.0 (blank), 0.2, 1.0, and 2.0 ng/mL clenbuterol. Spiked and blank urine diluted 1:1 (v/v) with 2% ammonium hydroxide

SPE Procedure:

Conventional Hydrophilic Polymer SPE Phase, 30 mg/1 mL

1. Condition and equilibrate SPE phase with 1 mL methanol and 1 mL DI water
2. Load 1 mL pre-treated sample on to the cartridge.
3. Wash (elute interferences) with 0.5 mL 2% ammonia and 0.5 mL 30% methanol
4. Elute clenbuterol with 2 mL methanol
5. Evaporate under nitrogen and reconstitute with 1 mL LC mobile phase prior to LC-MS-MS analysis

resulting in a high background response within LC elution area of clenbuterol (1-2 min.). This can potentially lead to lower assay reproducibility, accuracy and sensitivity thereby elevating lower limits of quantitation (LLOQ). Table 2 lists recovery values for clenbuterol using both sample prep procedures. Recovery for clenbuterol using the SupelMIP phase was 75% at the spike levels tested (0.1-1.0 ng/mL). 99% recovery was observed at the 0.1 ng/mL spike level. In contrast, reduced response levels were observed across the spike range tested using the

hydrophilic polymer SPE protocol with a recovery of 8% at the lowest spike level (0.1 ng/mL). Note that both extraction procedures were repeated using buffer as a sample matrix, and recovery values were comparable for all three spike levels (data not shown); therefore, the lower response values observed for the polymer phase were caused primarily by matrix effects. To further demonstrate the selectivity and sensitivity differences between the two procedures, Figure 4 compares the linear relation of known spike concentrations vs.

Figure 3. Clenbuterol-spiked urine samples extracted with SupelMIP SPE vs. conventional hydrophilic polymer SPE

column: Ascentis Express C18, 5 cm x 2.1 mm I.D., 2.7 μ m particles (53822-U)
 instrument: Applied Biosystems 3200 Q-TRAP
 mobile phase: 10% acetonitrile in 10 mM ammonium acetate (pH unadjusted):acetonitrile (80:10)
 temp.: 35 °C
 flow rate: 0.2 mL/min.
 detection: MS/MS, MRM transitions (277.2/203.1 and 277.3/168.2 m/z)
 ion mode: Positive
 ion source: Turbospray
 ion spray voltage: 3200 V
 source temperature: 425 °C
 collision gas: 45 psi
 injection: 5 μ L

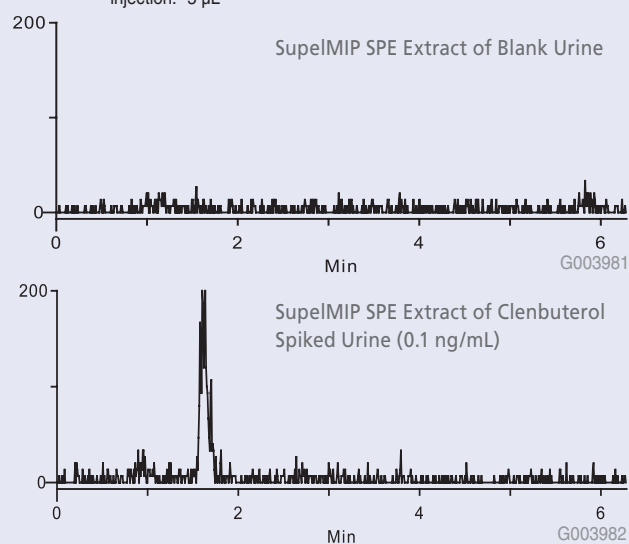


Table 2. Recovery comparison for clenbuterol from urine using SupelMIP SPE and conventional hydrophilic polymer SPE

Spike Level (ng/mL)	% Recovery from Urine	
	SupelMIP SPE - Clenbuterol	Hydrophilic Polymer SPE
0.1	99%	8%
0.5	75%	66%
1.0	75%	69%

calculated concentrations determined from the signal responses obtained from blank urine extracts spiked post-extraction. Post-extraction spiked samples were compared for both the SupelMIP and polymer SPE protocols described. Increased levels of ion-suppression were observed for the polymer SPE protocol relative to the SupelMIP procedure.

Conclusion

In this report, we demonstrated the extraction of SupelMIP SPE – Clenbuterol method against a published hydrophilic polymer SPE method for the trace extraction of clenbuterol from urine for subsequent LC-MS-MS analysis. Because selectivity is introduced during the development of the MIP itself, it creates a binding site that is sterically and chemical complementary to the target analyte(s). The multiple interactions that take place between the imprint binding site and analyte(s) of interest offer strong interactions enabling the use of exhaustive wash conditions during the SPE process to provide cleaner extracts prior to analysis. This was demonstrated for clenbuterol in which the SupelMIP SPE approach offered greater selectivity resulting in lower limits of quantitation

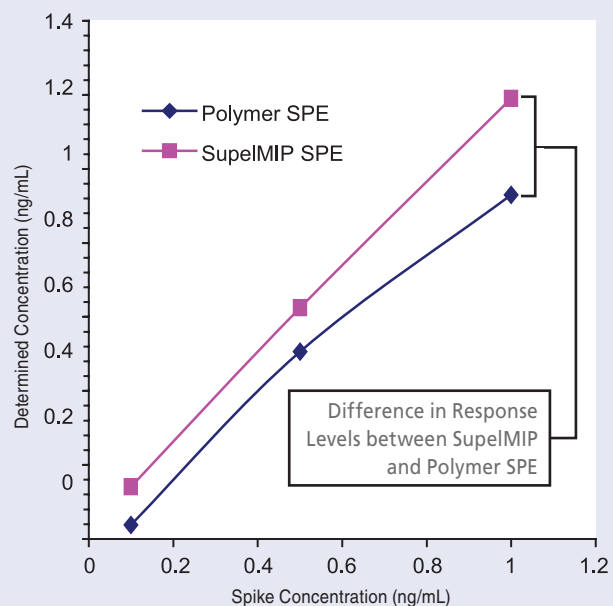
Featured Products

SupelMIP SPE Cartridges	Bed Weight (mg)	Cartridge Volume (mL)	Cartridges per Box	Cat. No.
Clenbuterol	25	10	50	53201-U
Beta-agonists (class selective)	25	10	50	53202-U
Beta-agonists (class selective)	25	3	50	53225-U
Beta-blockers (class selective)	25	10	50	53218-U
Beta-blockers (class selective)	25	3	50	53213-U
Full Beta Receptor (Beta-agonists and Beta-blockers)	25	10	50	53223-U
Full Beta Receptor (Beta-agonists and Beta-blockers)	25	3	50	53224-U
Chloramphenicol	25	10	50	53210-U
Chloramphenicol	25	3	50	53209-U
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	25	10	50	53206-U
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	25	3	50	53203-U
TSNAs (4 different Tobacco specific Nitrosamines: NNK, NNN, NAB, NAT)	50	10	50	53221-U
TSNAs (4 different Tobacco specific Nitrosamines: NNK, NNN, NAB, NAT)	50	3	50	53222-U
Riboflavin (vitamin B2)	25	10	50	53207-U
Triazines (class selective)	25	10	50	53208-U

To learn more about the SupelMIP SPE products, request the SupelMip brochure (JOZ) or visit us on our Web Site: www.sigma-aldrich.com/supelmip.



Figure 4. Known spike concentration vs. determined concentration for SupelMIP SPE and polymer SPE extracts of clenbuterol from urine (post-extraction spike)



which is critical for the analysis of this banned drug. Although the conventional hydrophilic polymer SPE method offers less procedural steps, sample prep selectivity was inadequate, giving higher background and ultimately resulting in a less sensitive assay.

References

1. Council Directive 86/469/EEC, European Union, Brussels, 1988.
2. Clenbuterol, FSIS Backgrounders, July 1995, <<http://www.fsis.usda.gov/OA/background/clenbute.htm>>.
3. GA Mitchell and G Dunnavan, J. Anim. Sci., 1998, 76:208-211.
4. D Patton, New Food Poisoning Case Hits China, Sep 2006, <<http://www.foodproductiondaily.com/news/ng.asp?id=70659>>.
5. M Josefsson et al., J Chromat. A, 2006, 1120:1-12.

radiello® – Fast and Efficient Diffusive Sampling for Thermal Desorption

Klaus Buckendahl

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For most diffusive/passive air sampling systems, the collected samples are desorbed via chemical desorption. A typical example of a desorption solvent is carbon disulfide (CS₂) for the desorption of volatile organic compounds (VOC) / BTEX from activated charcoal. Due to its health implications and pungent odor, many users would prefer not to work with CS₂ if possible.

In addition to cartridges for solvent desorption, the Radiello diffusive sampling line offers two cartridges for thermal desorption of VOC/BTEX and phenolic compounds (see Table 1 for compound listing). After sampling, the adsorbent cartridge is transferred into an empty thermal desorption tube with ID >4.8 mm (see

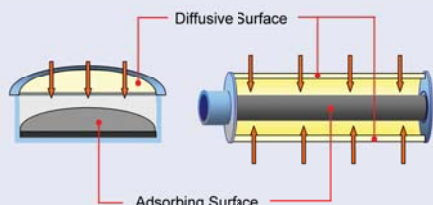


Figure 3. Placing cartridge into TD tube

Figure 3) that is then heated in a thermal desorber. As a result, all of the retained compounds are thermally desorbed and directly transferred into a GC instrument. Unlike solvent desorption, no dilution is involved.

Because of their radial design (see Figure 1), the Radiello samplers provide significant increases in sampling rates relative to other diffusive samplers that are commercially available. Such devices for thermal desorption often have diffusion caps installed, and typical sampling rates are in the range of 1-3 mL/min, making them more suitable for longer term measurements. In contrast, sampling rates for Radiello samplers are considerably higher (often by an order of magnitude, see Table 1). Such sampling rates are more on par with sampling rates observed with active sampling followed by thermal desorption (e.g. NOISH 2549, VOC Screening, recommends 10-50 mL/min) Faster sampling rates allow for shorter sampling times. Also, unlike active sampling, passive sampling requires no expensive pumps that need to be calibrated. This also permits simpler handling in hazardous environments.

Figure 1. Axial and radial sampler design



VOC/BTEX

The material used in the adsorbing cartridge (RAD145 for analyzing VOC/ BTEX by thermo desorption/GC) is graphitized carbon (Carbograph 4). Because of graphitised carbon's limitation in capacity, there is a greater risk of back diffusion. To circumvent this issue, a modified yellow diffusive body was developed to house the cartridge during sampling. The yellow diffusive body, due to its smaller pore size and thicker diffusive wall, provides a diffusive path of 150 mm while the standard white diffusive body (used with chemical desorption cartridge RAD130) has a path length of 18 mm. By this extension of the diffusive path length, the sampling rates and therefore the amount of analyte reaching the adsorbent cartridge are reduced, allowing for longer exposure times while greatly reducing the risk of back diffusion. Unlike chemical desorption, there is no dilution effect in thermal desorption. The complete amount of collected analyte is transferred onto the GC system, thereby increasing sensitivity.

Figure 2. Yellow diffusive body (RAD 1202)



Phenols Sampler for TD

Tenax TA is used for the adsorption and thermal desorption of phenolic compounds (RAD147). The Tenax TA cartridge requires a white diffusive body.

Please note: The diffusive bodies are not interchangeable between cartridges. The presented sampling rates are only valid for the listed adsorbing cartridge and diffusive body combination.

Please refer to table 1 for a complete listing of sampling rates for VOCs and Phenolic compounds.

The European Commissions LIFE project "ARTEMIDE (High Temporal Resolution Monitoring of VOCs by Diffusive Sampling)" which focused on benzene, MTBE and 1,3-butadiene, employed *radiello*® samplers for thermal desorption. This project and was recently recognized as one of the 24 best LIFE projects in 2004/2005 funded by the European Commission.

The *radiello*® product portfolio consists of adsorbing cartridges, diffusive bodies, ready-to-use samplers, standards for calibration and accessories, such as a shelter for outdoor measurements.

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

Table 1 Compounds and sampling rates at 25°C (Q_{298}) for Radiello samplers suitable for thermal desorption

	Q_{298} mL·min ⁻¹		Q_{298} mL·min ⁻¹
VOC/BTEX (RAD145&RAD1202)		VOC/BTEX (cont.)	
acetone	77	isobutanol	77
benzene	27.8	_-pinene	6.4
benzene	26.8	styrene	27.1
butylacetate	24.5	tetrachloroethylene	25.4
2-butoxyethanol	19.4	toluene	30
cyclohexane	27.6	1,1,1-trichloroethane	20
n-decane	22.3	trichloroethylene	27.1
1,4-dichlorobenzene	22	1,2,4-trimethylbenzene	21.9
dimethyldisulfide	23.7	n-undecane	12
n-heptane	25.3	m-xylene	26.6
n-hexane	25.5	o-xylene	24.6
ethylbenzene	25.7	p-xylene	26.6
2-ethoxyethylacetate 14.3		Phenols (RAD147 & RAD120) 65	
2-ethoxyethanol 26		phenol 38	
2-ethoxyethylacetate 20.9		o-cresol 45	
isopropylacetate 25.8		m-cresol 48	
limonene 12.8		p-cresol 48	
2-methoxyethanol 4		2,3-dimethylphenol 53	
2-methoxyethylacetate 21		2,5-dimethylphenol 51	
1-methoxy-2-propanol 26.6		2,6-dimethylphenol 46	
n-nonane 21		3,4-dimethylphenol 60	
n-octane 24.1		3,5-dimethylphenol 61	



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Length (m)	d_f (μ m)	Beta	Cat. No.
0.10 mm ID Fused Silica			
10	0.10	250	28465-U
15	0.10	250	28466-U
0.18 mm ID Fused Silica			
20	0.18	250	28564-U
12	0.30	150	28566-U
30	0.30	150	28575-U
20	0.36	125	28576-U
0.20 mm ID Fused Silica			
30	0.20	250	28513-U
0.25 mm ID Fused Silica			
30	0.10	625	28467-U
15	0.25	250	28469-U
30	0.25	250	28471-U
60	0.25	250	28472-U
15	0.50	125	28577-U
30	0.50	125	28473-U
60	0.50	125	28474-U
30	1.0	63	28476-U
0.32 mm ID Fused Silica			
15	0.25	320	28577-U
30	0.25	320	28482-U
30	0.32	250	28532-U
15	0.50	160	28597-U
30	0.50	160	28484-U
30	1.0	80	28487-U
0.53 mm ID Fused Silica			
15	0.50	265	28542-U
30	0.50	265	28541-U
30	1.0	132	28559-U

Phase: bonded and highly crosslinked silphenylene polymer virtually equivalent in polarity to 5% phenyl polymethylsiloxane

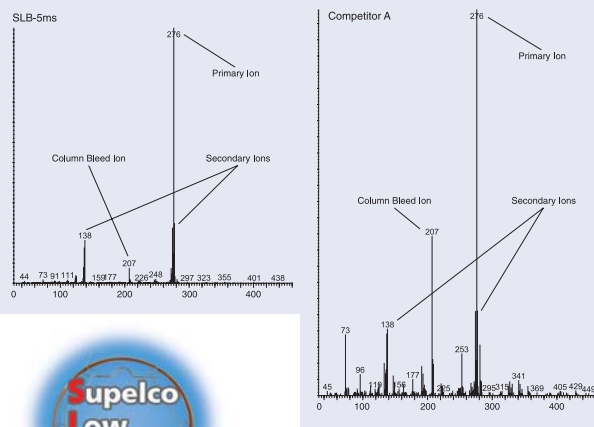
Temp. Limits:

0.10 to 0.32 mm I.D.: -60 °C to 340 °C (isothermal),

-60 °C to 360 °C (programmable)

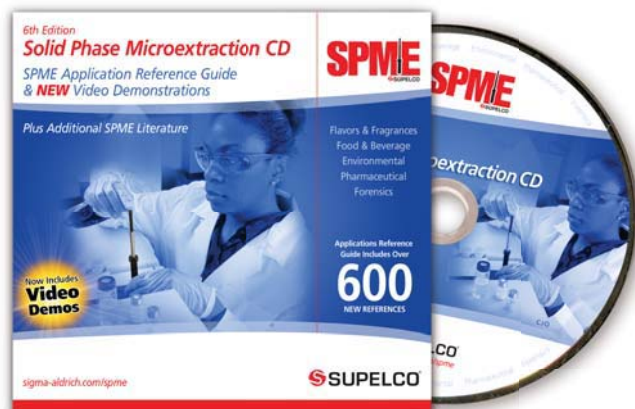
0.53 mm I.D.: -60 °C to 330 °C (isothermal),

60 °C to 340 °C (programmable)



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New 6th Edition SPME Applications CD Demonstrates the Versatility of SPME



Daniel Vitkuske
daniel.vitkuske@sial.com

While attending Pittcon® this year it was refreshing to see 29 different oral or poster presentations and multiple short courses on solid phase microextraction (SPME). What was even more impressive was to see them in areas so diverse, from US homeland security to biopharmaceuticals. Despite its introduction less than 15 years ago as an analytical sample prep technique, SPME rivals solid phase extraction (SPE) when it comes to its popularity among researchers. Compare the almost 30 sessions at Pittcon on SPME to 48 for SPE and significantly less for other common sample prep techniques (Table 1).

Table 1. Pittcon® 2007 Oral Presentations, Posters and Short Courses

Solid Phase Extraction (SPE).....	48
Solid Phase Microextraction (SPME).....	29
Automated Solvent Extraction (ASE).....	12
Thermal Desorption (TD).....	10
Purge & Trap.....	7

So it is quite appropriate to release the sixth edition of the SPME Applications CD approximately ten years after the first CD, with an additional 600 new application references since the last edition. Roughly 30-40 new articles are published each month referring to SPME use in analytical sample preparation.

SPME was invented by Dr. Janusz Pawliszyn at the University of Waterloo in 1989. The versatility of SPME is best demonstrated by the broad range of applications for which it has been used, including food & beverage, environmental, forensics and homeland security.

SPME Video Demonstrations

Most researchers unfamiliar with SPME are curious to see how SPME works. The newest enhancement to the

SPME CD is the inclusion of video demonstrations that show various aspects of the technique. The short video clips show manual SPME sampling, used with an autosampler as well as headspace and direct immersion sampling. The videos are intended to show how simple and easy SPME is to use and yet how versatile it is.

Applications Reference Guide

For many researchers the most useful part of the SPME CD is the applications reference guide, which now includes over 2200 SPME literature references. Table 2 shows that SPME applications are split between the major market segments. The applications reference guide can be searched by keyword, such as a specific analyte or sample matrix, to find previous research on SPME related to your specific areas of interest.

Table 2. SPME applications by area

Food, Flavors and Fragrances.....	550
Environmental.....	720
Forensics/Toxicology.....	243
Pharmaceutical/Biologicals.....	150

Technical Literature and Reference Information

The CD also includes a broad selection of technical information available on SPME, including application notes or technical bulletins on:

- Field Sampling for Pesticides
- Nitrogen Herbicides
- Organophosphate Insecticides
- Odors in Drinking Water
- Semivolatiles by EPA 625
- VOCs by EPA 524.2
- BTEX Compounds
- PAHs in Water
- Food Antioxidants and Preservatives
- Flavor Compounds
- Forensic Analysis
- Fire Debris
- Explosives
- Drugs in Biological Fluids
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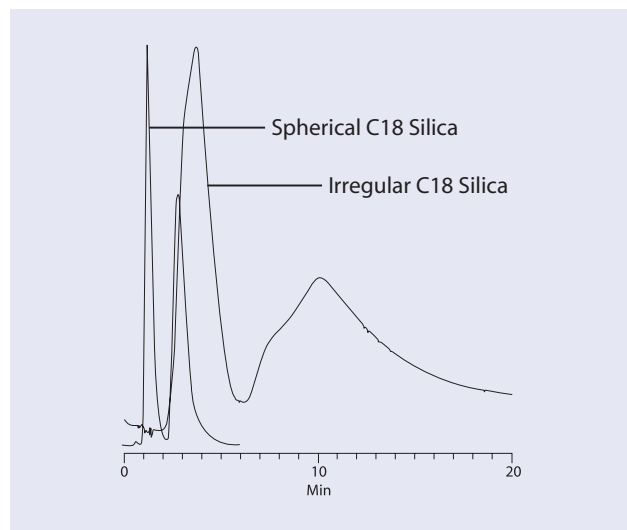


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- **Various Sample Loading Options**

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For your free copy of the Supelco VersaFlash Demonstration CD, call your local Sigma-Aldrich office and request literature code IZI.



Chiral GC – Using Cyclodextrin Derivatization to Create Ultimate Selectivity

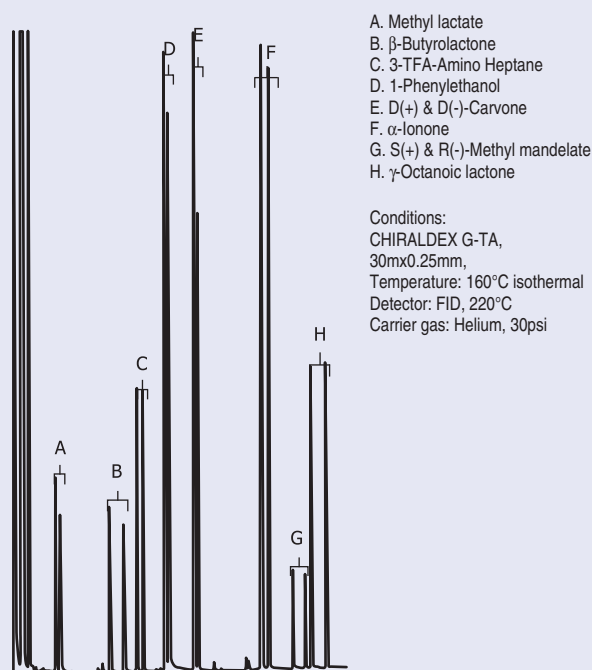
Denise Wallworth

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As a fast, high efficiency and high sensitivity analytical technique, chiral GC has probably the most to offer out of all chromatographic possibilities. Although the choice of separation technique can, of course, be solely driven by the solute properties – and for GC, compounds need to be volatile and thermally stable – chiral GC has wide appeal, especially for complex matrixes in environmental, biological, agricultural, food, and essential oil applications. The high efficiencies in GC have the additional benefit of providing lower limits of detection for many applications.

Cyclodextrins have developed a dominant role in chiral GC since 1985 as a result of the ability to derivatise the three cyclodextrins (CD), alpha, beta and gamma, altering the mechanism and ultimate selectivity towards chiral molecules. Hydroxyl groups in the 2- and 3-position around the rim, and at the 6-position at the base are used to create derivatised CDs and a series of GC phases that offer an extremely broad range of chiral separations. Enantioselectivity occurs by either an inclusion mechanism or a surface interaction (such as dipole-dipole) – or by a combination of both – and is therefore determined by both the position and type of the derivative.

Figure 1: Application: Range of polar chiral compounds separated on ChiralDEX G-TA



The first generation of chiral GC phases have all CD hydroxyls methylated and are solubilised (typically 10-20%) in a polysiloxane carrier. There is a large number of such permethylated phases available today – the Supelco DEX-110 and 120 (α , β , and γ) and the ChiralDEX B-PM being key phases in this area. They are highly selective and versatile in a wide range of applications, especially for aliphatic compounds, with some acids and bases requiring derivatisation. A second generation of non-polar permethylated phases changed the chemistry of the 6-position of the CD ring from methyl to tertiary butyl silyl, a group hydrophobic enough to block residual surface activity of the capillary and, more importantly, to increase the solubility of the CD in the carrier, resulting in higher selectivity at lower retention times. Aromatics and cyclics in general now show greater separation than on the traditional permethylated. The two important phases here are the Supelco DEX 325 and ChiralDEX B-DM and these differ in the type of carrier used and the concentration of CD (25 and 50% respectively). The effect of this is that the latter column gives higher efficiency for those separations that occur at lower temperatures.

Polar Derivatives

Once you start to substitute polar functional groups around the CD rim, versatility increases even further and selectivity broadens as you change the type and position of the derivative. Dipole-dipole interactions are created and the mechanism becomes a very efficient surface interaction. This allows the efficient use of gamma CD with a larger surface to cover a broader range of molecular analogs.

The ChiralDEX range provides a selection of such phases, two of which are the subject of global patents. The ChiralDEX G-TA has probably one of the broadest range of applications in this series from diols and lactones to alcohols and amines (Figure 1 gives just one example), while the B-DP separates aromatic and aliphatic amines well. Changing the derivative to hydroxypropyl (B-PH) extends the range of separation further to include sugars, bicyclics and haloalkanes. Additionally, the Supelco DEX225 phase uses a novel 2- and 3-acetyl derivative that provides unique selectivity. Together, this group offers some of the most efficient separations available. The complete range is summarised in Table 1.

Coated or Bonded?

The majority of chiral GC columns in use today are coated CD capillaries that provide the widest scope of chiral applications. A further increased degree of inertness can be introduced by bonding the CD. A new bonding chemistry recently developed for the Chiraldex permethylated phase has resulted in a very low bleed that is ideal for MS applications. This new column, the Chiraldex Bonded B-PM, is effective in separating complex mixtures including underivatized volatile chiral acids, alcohols, lactones and diols. Figure 2 shows one application for the separation of a mixture of polar and non-polar molecules, including an underivatized alcohol.

Method Development Techniques

In chiral GC method development, the temperature window of separation is first found for the solute by running a temperature gradient to 150°C at 1-5°C/minute (or to the maximum allowable operating temperature (MAOT) of the column if the volatility of the sample requires it – using 5-10°C/minute). A sample solvent is chosen that volatilises at least 40°C below the elution temperature – often methylene chloride is a good choice – and a 30:1 or 40:1 split ratio used. Unlike achiral GC, temperature is not subsequently used as an optimising parameter. Instead, velocity is used to increase peak efficiency and reduce retention times. Most enantioselective separations are optimal at much lower temperatures than the MAOT of the column.

Derivatization in chiral GC is far more common than in LC. Where a compound is insufficiently volatile, or is

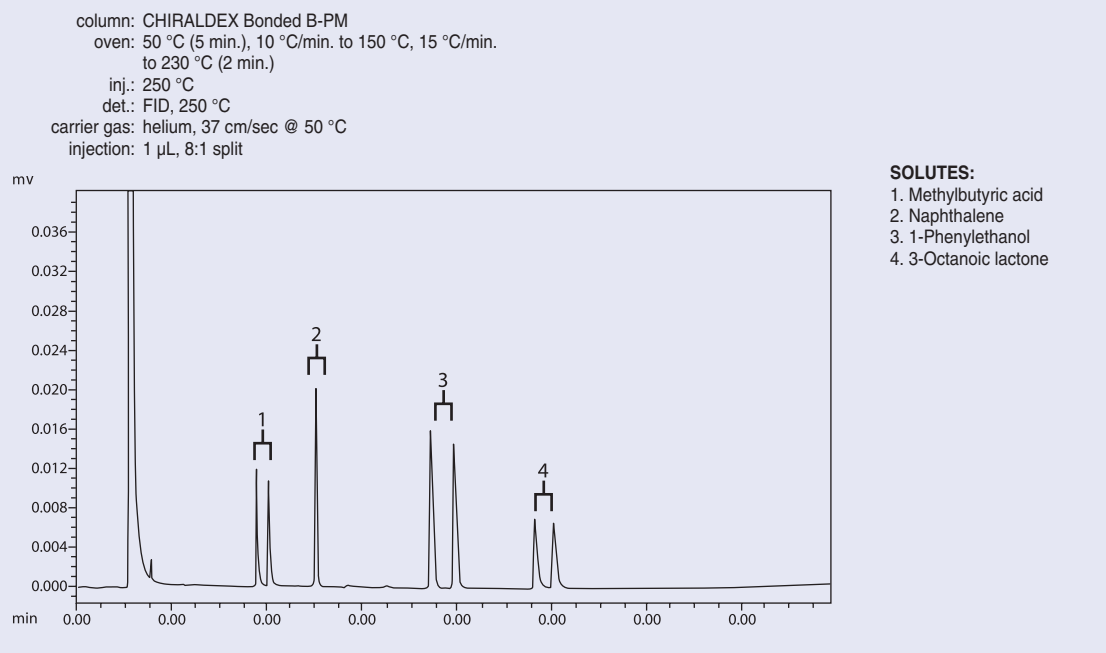
temperature labile at higher temperatures, derivatization with a suitable achiral reagent is used. Creating a derivative can also introduce different chiral interactions, resulting in a faster analysis times and more efficient separations, and can even effect a separation where one was not possible before. Generally, it is very polar molecules, such as alcohols, amines, acids, amino alcohols that are derivatised prior to chiral separation. The Chiraldex Handbook gives a very useful guide to the techniques used. All derivatization reagents used are available from Sigma-Aldrich.

Reversal of Elution Order

In trace analysis, elution order is critical for optimal quantification. Derivatization becomes extremely useful in this case, and simply changing the type of derivative made can reverse elution order. In summary, elution order reversal can occur by:

- Changing from one type of cyclodextrin to another (e.g. G-TA to B-TA)
- Changing from one phase type to another (e.g. B-PH to B-DA)
- Changing from one %CD to another (e.g. β -DEX 110 to 120)
- Changing of derivative (e.g. trifluoroacetyl to acetyl)
- Operating below ambient temperatures

Figure 2: Separation of non-polar and underivatized polar solutes using the new CHIRALDEX Bonded B-PM



Applications

The list of application areas for each column type is too long to present here. For a comprehensive guide, see the table in the Astec Product Guide, or consult the ChiralDEX GC Handbook. Table 1 shows the complete range of chiral GC capillary columns available; a special development kit is available that includes three ChiralDEX columns for the broadest range of applications: – the ChiralDEX G-TA, B-DM and B-DA.

ChiralDEX column kit

Cat. No.	Size
CHIRALDEX GC Kit: one each of G-TA, B-DM & B-DA	
71010AST	10m x 0.25mm
71020AST	20m x 0.25mm
71030AST	30m x 0.25mm
71040AST	40m x 0.25mm
71050AST	50m x 0.25mm

Table 1: Supelco ChiralDEX polar and non-polar substituted chiral GC phases from-Sigma Aldrich.

Column	Substitution Chemistry	Most Useful
Non-polar:		
CHIRALDEX B-PM	Permethyl (2,3,6-tri-O-methyl)	B-PM
α, β, γ -DEX 110/120	Permethyl (2,3,6-tri-O-methyl) (10 or 20% in polysiloxane)	β -DEX
CHIRALDEX B-, G-DM	Dimethyl (2,3-di-O-methyl-6-t-butyl dimethyl-silyl) in tri-methyl polysiloxane	B-DM
α, β, γ -DEX 325	Dimethyl (2,3-di-O-methyl-6-t-butyl dimethyl-silyl) in phenyl di-methyl polysiloxane	β -DEX
CHIRALDEX A-, B-, G-DA	Dialkyl (2,6-di-O-pentyl-3-methoxy)	B-DA
Polar:		
CHIRALDEX A-, B-, G-TA	Trifluoroacetyl (2,6-di-O-pentyl-3-trifluoroacetyl)	G-TA
α, β, γ -DEX 225	Diacetyl (2,3-di-O-acetyl-6-t-butyl dimethyl-silyl)	β -DEX
CHIRALDEX B-, G-DP	Dipropionyl (2,3-di-O-propionyl-6-t-butyl silyl)	B-DP
CHIRALDEX G-PN	Propionyl (2,6-di-O-pentyl-3-propionyl)	B-PN
CHIRALDEX G-BP	Butyryl (2,6-di-O-pentyl-3-butyl)	B-BP
CHIRALDEX A-, B-PH	S-Hydroxypropyl ((S)-2-hydroxypropyl methyl ether)	B-PH
Bonded:		
CHIRALDEX Bonded B-PM	Permethylated	B-PM

For more information request your literature today!

! Information

- HPLC Column Selection Guide/Astec (IEY)
- Chromatography Product Guide/Astec (JCI)
- ChiralDEX GC Columns (Handbook)/Astec (JCH)

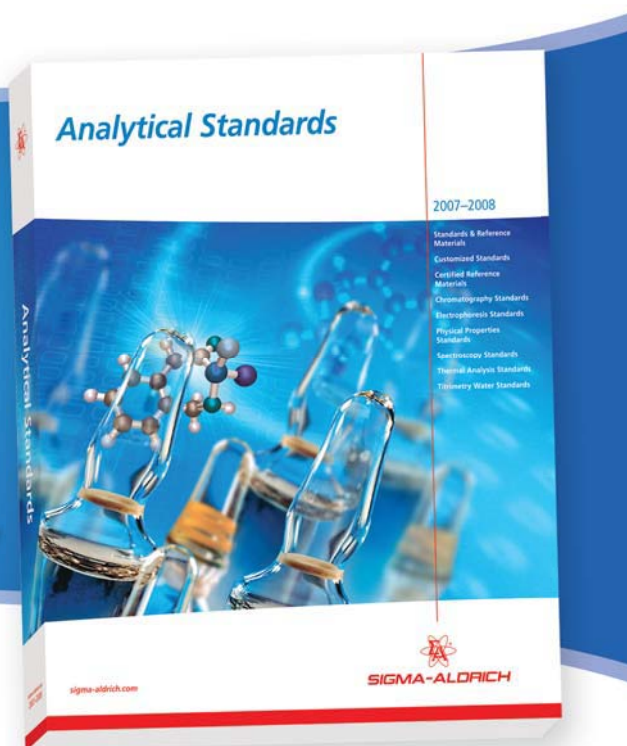


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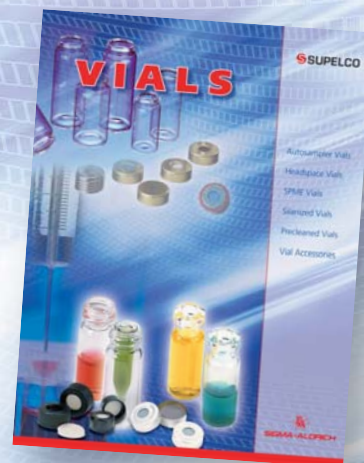
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Flame Retardant Standards

Polybrominated diphenyl ethers (PBDEs) have been used effectively as flame-retardants for more than thirty years. They have been instrumental in reducing the spread of fire in fabrics, foams and plastics. Unfortunately, PBDEs are now considered to be as toxic, if not more so, than PCBs. They are known to leach into the environment and bioaccumulate in the fatty tissues of living organisms. Research studies conducted on mice have shown that high PBDE bioaccumulations impair learning and development, causing great concern for developing human fetuses, infants and children.

To reduce these health hazards, the European Union has banned the use of the most toxic PBDEs. Some U.S. manufacturers have voluntarily stopped using these same chemicals. Ongoing environmental and health impact

studies are needed to understand the long-term effects of human and animal exposure to PBDEs.

To aid in this investigation, Sigma-Aldrich has launched a new line of flame retardant analytical standards through its Riedel-de Haën brand. The initial offering, listed below, consists of ten standards of analytical quality, suitable for GC and GC-MS analysis.

Description	Cat. No.
BDE No. 209, 50 µg/mL in isooctane:toluene (9:1)	34120
BDE No. 119, 50 µg/mL in isooctane	34121
BDE No. 119, 50 µg/mL in isooctane	34114
BDE No. 77, 50 µg/mL in isooctane	34115
BDE No. 75, 50 µg/mL in isooctane	34116
BDE No. 37, 50 µg/mL in isooctane	34123
BDE No. 71, 50 µg/mL in isooctane	34118
BDE No. 66, 50 µg/mL in isooctane	34119
BDE No. 138, 50 µg/mL in isooctane	34122
BDE No. 207, 10 µg/mL in nonane	34113

20% OFF Flame Retardants:

Please quote promotion code U15 when ordering.
Offer expires on 30.09.2007.

Natural Terpene Compounds for Flavor & Fragrance Analysis

Natural terpenes are a large and varied class of hydrocarbons whose molecular formula is based on multiples of $(C_5H_8)_n$. These materials are produced mainly by plants and, to a lesser degree, insects. When terpenes are chemically modified by oxidation or rearrangement of their carbon skeleton they are referred to as terpenoids.

Terpenes and terpenoids are the chief constituents of essential oils found in plants and flowers, and the reason for the oil's fragrant essence. Essential oils are used for aromatherapy, flavoring candies and foods, creating perfumes, and in the manufacturing of soaps, shampoos, and household cleaning products. This makes them valuable commodities to the flavor and fragrance industry. Analysts evaluating the quality of natural essential oils and/or examining the oils for possible adulteration by synthetic versions require high purity analytical standards.

Sigma-Aldrich, through its Fluka brand, has addressed this need by offering an extensive line of natural terpene standards for essential oil analysis. All compounds have been tested for purity and identity.

To view the complete list of natural terpene compounds, please visit sigma-aldrich.com/standards

Description	Cat. No.
Bergamottin, purum, 98+% (HPLC)	01338
(+)-β-Citronellene,* ≥98.5% (GC, sum of enantiomers)	27475
(-)-β-Citronellol,* ≥98.5% (GC, sum of enantiomers)	27483
Eucalyptol,* 99.7+% (GC)	29210
(±)-Lavandulol acetate,* ≥98.5% (GC)	61736
(R)-(+)-Limonene,* 99+% (GC, sum of enantiomers)	62118
(S)-(-)-Limonene,* 99+% (GC, sum of enantiomers)	62128
(+)-Menthol,* 99+% (GC sum of enantiomers)	63658
(+)-Menthone,* ≥98.5% (GC, sum of enantiomers)	63675
(-)-Menthone,* 99.0% (GC, sum of enantiomers)	63677
Myrcene, purum, ≥95.0% (GC)	64643
(+)-α-Pinene,* ≥99.5% (GC, sum of enantiomers)	80605
(-)-α-Pinene,* ≥99.0% (GC, sum of enantiomers)	80599
Rosmarinic acid, purum, ≥95.0% (HPLC)	44699
trans-Terpin,* ≥99.0% (GC)	09828
γ-Terpinene,* ≥98.5% (GC)	86476

*purriss p.a., terpene standard for GC



Headspace Vials

Ron Shawley

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Sigma-Aldrich offers a wide range of headspace vials for a variety of autosamplers that include Agilent®, CTC, PerkinElmer®, Shimadzu®, Varian® and others. We supply headspace vials with a variety of top and bottom finishes, and vials for SPME applications. We inventory a large range of the most popular vials and are able to ship to you within forty-eight hours.

Our headspace vials are manufactured in many sizes (5-27 mL), in clear and amber glass, and in both crimp style and screw thread versions. These vials are available with beveled top or flat top finishes. The beveled top is required for some autosampler systems, including those manufactured by PerkinElmer. Using this style of vial reduces the amount of sealing surface between the crimp seal and the glass surface. Therefore, we recommend using bevel-topped vials only when specified by the autosampler manufacturer.

Vials for headspace applications are available with flat bottom or rounded bottoms. Vials with a rounded bottom are sturdier and more resistant to pressure than the flat bottom vial. The rounded bottom version was created to

Figure 1. Characteristics of headspace vials



allow the vial, when transported by a magnet, to drop more easily into the heating block.

Solid phase microextraction (SPME) requires the use of thin vial septa to minimize septum coring. Thin seal vials used for SPME provide a thicker sealing lip to compensate for the thinner septa.

For more information or to inquire about products, please email techservice@sial.com



Related Information

For a free copy of our vial brochure, please request IXH or visit us on the Web at sigma-aldrich.com/vials

Table 1. Vials

Description	Compatibility	Cat. No.
5 mL, Flat Top, Rounded Bottom		
Clear glass, 10-425 screw thread, 12 x 96 mm	Shimadzu TOC	27319
Clear glass, crimp top, 12 x 96 mm	Shimadzu TOC	27324
6 mL, Beveled Top, Rounded Bottom		
Clear glass, crimp top, 22 x 38.2 mm	PerkinElmer	SU860065
Clear glass, crimp top, 22 x 38 mm	PerkinElmer	508403
6 mL, Beveled Top, Flat Bottom		
Clear glass, crimp top, 22 x 38 mm	General Use	27292
Clear glass, crimp top, 23 x 38 mm	General Use	27197
10 mL, Flat Top, Rounded Bottom		
Clear glass, crimp top, 22.5 x 46 mm	CarloErba, CTC, Fisons, Varian (CP)	854180-U
10 mL, Flat Top, Flat Bottom		
Clear glass, (long neck), crimp top, 22.5 x 46 mm	CarloErba, Dani, Fisons, Agilent	SU860029
Clear glass, crimp top, 20 x 54.5 mm	Varian	854151
10 mL, Beveled Top, Rounded Bottom		
Clear glass, crimp top, 22.6 x 46 mm	General Use	27294
10 mL, Beveled Top, Flat Bottom		
Clear glass, crimp top, 23 x 46 mm	General Use	508438
10 mL, Screw Thread, Flat Top, Rounded Bottom		
Clear glass, 22.5 x 46 mm	CTC PAL (Varian, GERSTEL, Atas, Shimadzu, Agilent)	SU860099
Amber glass, 22.5 x 46 mm	CTC PAL (Varian, GERSTEL, Atas, Shimadzu, Agilent)	SU860100
10 mL SPME Vial, Flat Top, Round Bottom		
Clear glass (Thin Seal), crimp top, 24.5 x 80 mm	SPME vial for CTC PAL	27386
12 mL, Beveled Top, Round Bottom		
Clear glass, crimp top, 18 x 65 mm	Tekmar, Varian	508446
20 mL, Beveled Top, Round Bottom		
Clear glass, crimp top, 23 x 75.5 mm	PerkinElmer, Tekmar	SU860049
Clear glass, crimp top, 22.6 x 75 mm	PerkinElmer, Tekmar	27296
Clear glass, crimp top, 22 x 75 mm	Agilent, CTC, Dani, Fison HS 850, PerkinElmer	508454

Description	Compatibility	Cat. No.
20 mL, Beveled Top, Flat Bottom		
Clear glass, crimp top, 23 x 75 mm	General Use	27199
20 mL, Flat Top, Round Bottom		
Clear glass (long neck), crimp top, 22.5 x 75.5 mm	CTC PAL (Varian, GERSTEL, Atas, Shimadzu, TriPlus HS)	SU860030
Clear glass (thin seal), crimp top, 22.5 x 75.5 mm	SPME Vial for CTC PAL	854181-U
20 mL, Screw Thread, Flat Top, Round Bottom		
Clear glass, 23 x 75.5 mm	PerkinElmer	SU860051
Clear glass, 22.5 x 75.5 mm	CTC PAL (Varian, GERSTEL, Atas, Shimadzu, Agilent)	SU860097
Amber glass, 22.5 x 75.5 mm	CTC PAL (Varian, GERSTEL, Atas, Shimadzu, Agilent)	SU860098
27 mL, Beveled Top, Flat Bottom		
Clear glass, crimp top, 30 x 60 mm	Shimadzu	27298

Table 2. Caps and Seals

Description	Septa	Thickness	Pk. Size	Cat. No.
Crimp Seals for Headspace Vials				
Aluminum seal, 10 mm center hole	Pharma-Fix	3.0 mm	100	SU860011
Magnetic seal, 5 mm center hole	Pharma Fix	3.0 mm	100	854178-U
Magnetic seal, 8 mm center hole	Pharma Fix	3.0 mm	100	SU860014
Aluminum seal, 10 mm center hole	PTFE/silicone	3.0 mm	100	SU860010
Aluminum seal, 10 mm center hole	PTFE/silicone	3.25 mm	100	854996
Magnetic seal, 5 mm center hole	PTFE/silicone	3.0 mm	100	854179-U
Magnetic seal, 8 mm center hole	PTFE/silicone	3.0 mm	100	SU860015
Magnetic seal for SPME, 8 mm center hole	PTFE/silicone	1.5 mm	100	SU860053
Magnetic seal for SPME, 8 mm center hole	Viton	1.0 mm	100	SU860106
Aluminum seal, open center	Viton	1.0 mm	100	27245
BiMetal seal (tin/aluminum), silver	PTFE/silicone	3.0 mm	100	29179-U
BiMetal seal (tin/aluminum), red	PTFE/silicone	3.0 mm	100	29169-U
BiMetal seal (tin/aluminum), blue	PTFE/silicone	3.0 mm	100	29176-U
Pressure release seal, open center	PTFE/rubber	3.0 mm	100	27454-U
Pressure release seal, open center	PTFE/silicone	3.0 mm	100	27455-U
Magnetic Screw Caps for Headspace Vials				
Magnetic Screw Cap, 8 mm center hole	White PTFE/blue silicone	1.3 mm	100	SU860101
Magnetic Screw Cap, 8 mm center hole	PTFE/red butyl	1.5 mm	100	SU860102
Magnetic Screw Cap, 8 mm center hole	Blue PTFE/silicone	1.6 mm	100	SU860103

Multipaks of SGE Syringes

Ron Shawley

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- Eliminate downtime by having a ready supply on hand
- Reduce lab expenses by ordering in multipacks
- Save time by ordering less often

Multipaks are the most convenient and economical way to purchase multiple units of the same SGE syringe. Each syringe multipak costs less than the same number of syringes separately, and is packaged in a convenient

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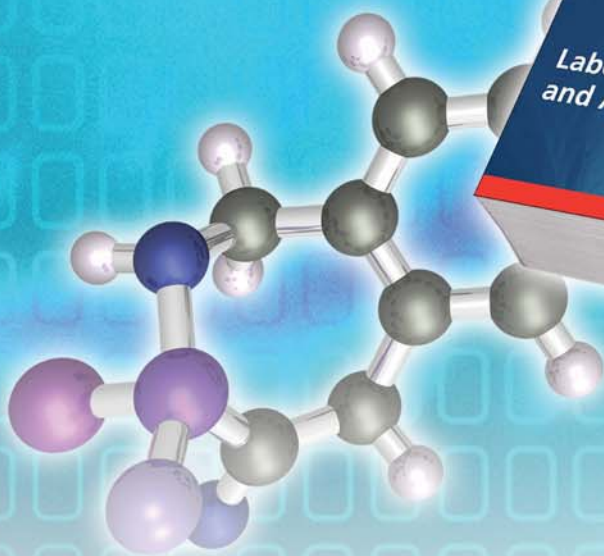
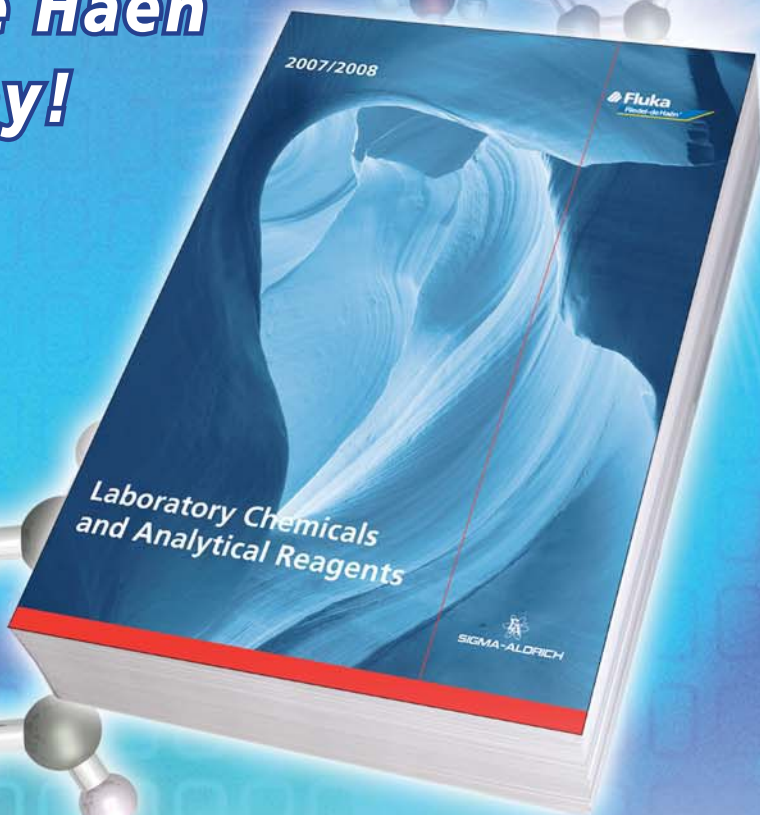
storage box. This special packaging requires much less drawer space to store than individually boxed syringes, while still protecting your syringes from being damaged or broken before use.

Volume	Length (mm)	Gauge	OD (mm)	Tip Style	Pack Size	SGE Cat. No.	Supelco Cat. No.
Standard Plunger Syringe							
10 µL, fixed needle	50	26	0.47	bevel	6	002030	21934-U
10 µL, fixed needle	50	26	0.47	bevel	10	002033	26239
SuperFlex Flexible Plunger							
10 µL, fixed needle	50	26	0.47	bevel	6	002130	23966
10 µL, removable needle	50	26	0.47	bevel	6	002180	23967
GC Autosampler Fixed Needle Syringes for Agilent (HP)							
5 µL	42	26	0.47	cone	6	001804	21910
5 µL	42	23	0.63	cone	6	001814	21911
5 µL Dual Gauge	42	23-26s	0.63/0.47	cone	6	001822	26887-U
10 µL	42	26	0.47	cone	6	002804	21912
10 µL	42	23	0.63	cone	6	002814	21544
10 µL Dual Gauge	42	23-26s	0.63/0.47	cone	6	002822	26889-U
10 µL Dual Gauge, Gas Tight	42	23-26s	0.63/0.47	cone	6	002827	26891-U

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