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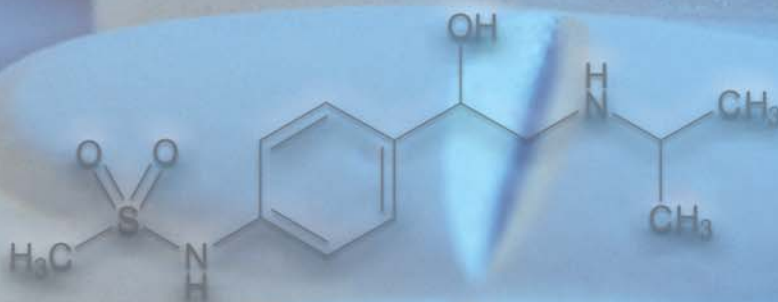
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**Dear Colleague,**

I'd like to bring to your attention a recent event that has particular importance to us and to our customers: Sigma-Aldrich's acquisition of Advanced Separation Technologies, Inc. (Astec), a leader in chiral chromatography.

Customer Service is also important to Astec, another reason it is a good fit within the Sigma-Aldrich organization. Besides the above-mentioned products, Astec also offers a method development service that helps customers choose the right chiral stationary phase (CSP) for both analytical and preparative separations. This service saves customers valuable time and reduces costs associated with buying and testing multiple CSPs. Astec also offers screening kits for both LC and GC, technical handbooks and method development wall charts to aid in the efficient development of methods.

Like Sigma-Aldrich, innovation is at the heart of Astec's corporate culture. Many of their products were conceived through collaboration with Dr. Dan Armstrong, a collaboration that will continue in the new organization. Several Astec products have been patented and one, the CHIROBIOTIC™ V HPLC column, was chosen by R&D Magazine to be among the 100 most technologically significant new products of 1995. Since that time, CHIROBIOTIC has grown into a family of columns that now represents the flagship of the Astec line. Because they permit the use of MS-compatible mobile phases in both anhydrous methanol/volatile salt and reversed-phase type eluants, CHIROBIOTIC columns have become the market leader for LC/MS of enantiomers.

Some of the well-respected Astec brand columns that will now be offered through Sigma-Aldrich include:

Astec HPLC and GC columns for chiral separations

- CHIROBIOTIC™ including CHIROBIOTIC V, V2, T, T2, R and TAG macrocyclic glycopeptide bonded HPLC columns (patented)
- CYCLOBOND™ bonded cyclodextrin HPLC columns
- ASTEC CLC copper ligand exchange HPLC columns
- P-CAP™ HPLC columns (patented)
- CHIRAL-AGP (α^1 -acid glycoprotein), CHIRAL-CBH (cellobiohydrolase), CHIRAL-HSA (human serum albumin) HPLC columns
- CHIRALDEX™ derivatized cyclodextrin capillary GC columns

Astec pH-stable HPLC columns

- apHera™ polymer-based HPLC columns in C_8 , C_{18} and NH_2 chemistries

This quote by Supelco's President Russel Gant summarizes the new relationship: "In addition to a wide range of proprietary chiral chromatography products, the acquisition of Astec brings the specialized knowledge of chiral separations embodied in its people. This enables our Analytical business to continue to expand the value we deliver to the scientific community."

If you are currently an Astec customer, welcome to the Sigma-Aldrich family! Here you will find the same high level of service and product quality to which you have become accustomed, now augmented by the strengths of the Sigma-Aldrich organization. If you're not familiar with Astec products, we look forward to the opportunity to share the benefits of Astec chiral column technology with you

Kind regards,

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Chiral Method Development Strategies for HPLC

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Abstract

Generic screening to provide fast solutions for chiral method development, whether needed for routine analytical use, trace analysis, LC/MS or preparative LC, has become increasingly important. In this article, simple screening tools are used with chiral stationary phases based on macrocyclic glycopeptides to generate robust methods for a broad range of applications.

Chiral HPLC

A change in discovery strategy in the pharmaceutical industry has resulted in the earlier investigation of the generally different biological activities for the enantiomers chiral drug molecules. The number of chiral molecules coming through the discovery process has increased enormously over the past few years; they have also become more and more diverse so the approach to chiral method development has needed to incorporate just those chiral stationary phases (CSPs) that offer the broadest possible range of applications for fast and efficient screening.

Over the past 25 years or more, several hundred CSPs have been developed, with around 110 commercially available. By bonding or coating a chiral selector – either a small chiral molecule or a larger polymer such as a protein – onto silica, different interaction strengths for the two enantiomers can be induced. The cellulosic and amylosic phases were some of the earliest and broadest ranging CSPs to be developed and are based on natural chiral polymers, coated (or bonded, as in 2 phases) onto silica; one of Advanced Separation Technologies' (Astec, now part of Sigma-Aldrich/Supelco) new products, the P-CAPT[™] column also utilises polymer technology to provide chiral selectivity in a number of mobile phase types.

In the mid-90's, a new CSP range based on the chemical bonding of macrocyclic glycopeptides was developed by Astec [1] and since they have extremely broad applicability for different compound classes, including polar ones, the pharma industry has widely accepted them into their chiral method development schemes. Applications cover a wide range from aliphatic to aromatic molecules, small heterocycles to large fused ring aromatics, acids, neutrals and bases, plus amino acids and peptides.

CHIROBIOTIC columns are produced by covalently bonding a highly selective glycopeptide to high purity silica in several positions, so that the columns are extremely robust and can be used over a wide flow rate range and varied mobile phase conditions. There are four CSPs in the series, based on vancomycin, teicoplanin, teicoplanin aglycone and ristocetin. There are no restrictions in solvent choice (even chlorinated solvents can be used), although some will give a higher chance of chiral selectivity – the only restriction is in reversed phase when a pH range of around 3 – 7 is applicable (slightly different for each CSP).

For chiral screening, it is essential that a number of mobile phase possibilities are available, both to provide a variety of enantioselective interactions between solute and CSP and to cover a range of sample solubilities. The strength and availability of these enantioselective mechanisms will vary according to the mobile phase – π - π interactions, for instance, are only strong in normal phase solvents, while ionic interactions are only possible in reversed phase or in polar (methanol-based) mobile phase types. An effective screening system will include all of these.

CHIROBIOTIC CSPs are used routinely in four distinct mobile phase types – polar ionic mode, polar organic mode, reversed phase and normal phase. The polar ionic mode ('PIM') is unique to the CHIROBIOTIC phases for ionisable molecules. It uses methanol with small amounts of acid and base added to it, or methanol with a small amount (typically 0.1g per 100mL MeOH) of a solid volatile ammonium salt directly added to the MeOH (this can be acetate, trifluoroacetate or formate). Figure 1 gives a typical example in this mode, popular for its speed and enhanced MS detection. The polar organic mode is

Figure 1. Separation of 2-Bromo-3-methylbutyric acid on CHIROBIOTIC R

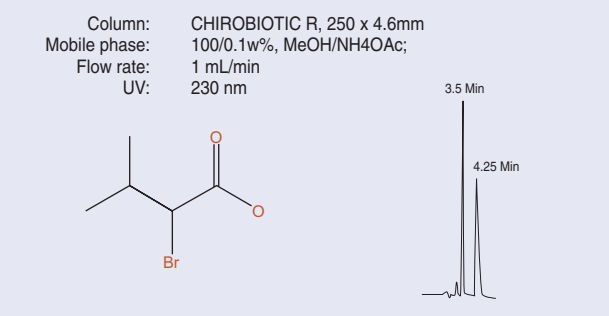
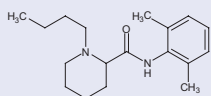
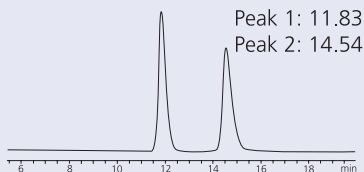


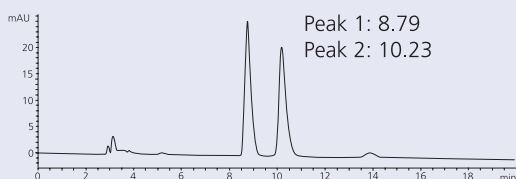
Figure 2. Separation of Bupivacaine



CHIROBIOTIC V2, 250x4.6mm
Mobile Phase: 90/10, MeOH/10mM NH₄OAc, pH 4.1
Flow Rate: 1 mL/min
 $\alpha = 1.30$



CHIROBIOTIC V2, 250x4.6mm
Mobile Phase: 80/20, MeOH/20mM NH₄OAc, pH 4.1
Flow Rate: 1 mL/min
 $\alpha = 1.24$



another non-aqueous mobile phase system that uses single or combinations of solvents and is effective on these CSPs for neutral molecules. Reversed phase is very similar to non-chiral type RP applications, where increasing the water content increases retention (except for some amino acid applications, when it is the reverse). It is also ideal for LC/MS methods. Finally, normal phase, representing about 15% of all separations, uses typical hexane or heptane-alcohol mixtures and has provided some unique separations. The CHIROBIOTIC Handbook [2] provides examples of applications in all mobile phase types.

By going from one mobile phase type to another - on the same column - the separation mechanism changes, giving you another opportunity for separation; there are no memory effects from the previous mobile phase that can affect results. Our methods development laboratory has over the years developed and refined the screening composition for each mobile phase type, so these are available to you to determine very quickly whether a given column type and mobile phase type are going to give you the desired selectivity. There are then simple optimization techniques for developing the method the way that you need it (for example, for MS detection, biocatalysis, dissolution analysis or preparative applications).

Table 1. Generic chiral method development screens

Mobile phase type	Screening mobile phase	Interaction*
Polar ionic mode	100/0.1/0.1 (v/v/v), MeOH/AcOH/TEA	Ionic , hydrogen bonding, steric, π - π
Reversed phase	30/70, MeOH/20mM NH ₄ Ac, pH 5	Ionic, hydrogen bonding, steric, inclusion complexing
Polar organic mode	100% EtOH	Hydrogen bonding, steric, π - π , dipole stacking , hydrophobic inclusion
Normal phase	20/80, EtOH/Heptane	Hydrogen bonding, steric, π - π , dipole stacking

* Strongest interaction in bold type

CONDITIONS: Generic chiral method development screens

Column size	250 x 4.6mm, 5mm	150 x 4.6mm, 5mm
Flow rate	1.0 mL/min	1.0 mL/min
Equilibration time	25 minutes	15 minutes
Run time	25 minutes	15 minutes

Results

The aim of this screening system is that it gives a rapid positive or negative answer; if a single, symmetrical peak is seen within the run time – provided the retention time is above 10 minutes – then this column or this mobile phase does not have the correct selectivity. If the retention time is below 10 minutes, it is important to re-run, diluting the mobile phase a little; the method can always be speeded up later as part of the optimization process. If there is peak splitting or even a slight shoulder, then you can progress to the optimization steps. For the complete screen, you proceed through the four screening mobile phases for each column to determine the best selectivity. Remember, there are no memory effects with the CHIROBIOTIC range, so each column can be used for each mobile phase type in sequence.

Optimization

There are simple optimization protocols available for these columns. The polar ionic mode is especially easy to manipulate, using acid/base or volatile salt concentration changes to increase selectivity and/or change retention. Normal phase and the polar organic mode use a standard selection of modifier choices – different alcohols, for instance – to optimize the method; the retention is simply controlled by the polarity of the solvent. Reversed phase uses the same variables as in most RP methods – buffer type and concentration, organic solvent choice – to provide the best separation (see Figure 2). In addition, temperature can be used; lower than ambient temperatures have always been used in chiral LC to

significantly increase selectivity, but for CHIROBIOTIC CSPs, higher temperatures are also often used to increase peak efficiency, reduce retention times or reverse elution order [3]. These columns can be used up to 50°C without any loss in performance.

References

1. D W Armstrong, Y Tang, S Chen, Y Zhou, C Bagwell, J-R Chen ; Macrocylic Antibiotics as a New Class of Chiral Selectors for Liquid Chromatography. Anal Chem, 1994, 66.9, 1473-1484.
2. CHIROBIOTIC Handbook, 5th Edition, Advanced Separation Technologies, now part of Sigma-Aldrich/Supelco
3. A Bertold, B L He, T E Beesley; Temperature and Enantioselectivity by Macrocylic Glycopeptide Chiral Stationary Phases. J Chrom A, 2004, 1060, 205-214.

+ Related Products

Description	Cat. No.
Chiral Screening Kits: Chirobiotic LC Chiral Selectivity Screening Kit - 100 Kit: 5mm CHIROBIOTIC V2, T, R, TAG, 100x4.6mm	10300
Chiral Selectivity Screening Kit - 250 Kit: 5mm CHIROBIOTIC V2, T, R, TAG, 250x4.6mm	10305

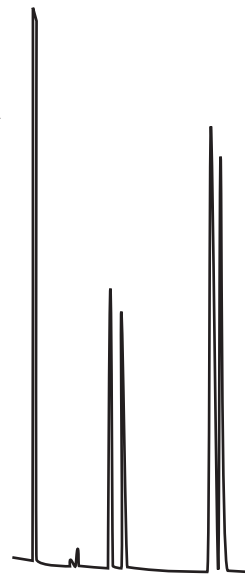
Individual columns in a wide range of sizes are also available.

Astec's CHIRALDEX Chiral GC Columns now Supplementing Sigma-Aldrich/ Supelco's GC Column Range



As of October 2006 Astec became part of Sigma-Aldrich/Supelco.

Separation of the enantiomers of 3 pheromones, using a CHIRALDEX B-TA column



Alternative Selectivity of Amide-based Embedded Polar Group HPLC Stationary Phases – Role of Hydrogen Bonding

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Abstract

High-performance liquid chromatographic (HPLC) method development often commences using alkyl (C8, C18) stationary phases. The method development scientist will commonly adjust the mobile phase organic modifier percentage and type as well as the solvent pH in an attempt to obtain the desired retention and resolution for a given analysis. When these common practices fail to yield the desired result, utilization of alternative stationary phase chemistries may be considered. What stationary phase, however, is most suitable for a given set of analytes?

Introduction

Among the more common commercially-available chemistries, embedded polar group (EPG) phases are becoming a preferred alternative stationary phase. EPG stationary phases generally consist of an alkyl chain with polar groups such as an amide, urea, sulfonamide or carbamate embedded in the bonded ligand. EPG phases have been observed to provide improved peak shape for basic compounds, differential selectivity, decreased retention of hydrophobic analytes and suitability for 100% aqueous applications. The types of analytes that show differential selectivity and the retention mechanisms that govern such differences, however, remain unclear.

In these investigations, the fundamental interactions that contribute to alternative retention on EPG phases as compared to traditional C18 chemistries is briefly reported. The studies include a comparison between Ascentis C18 and Ascentis RP-Amide using a column classification protocol as reported by Euerby et al.(1,2), and a comprehensive linear solvation energy relationship (LSER) investigation (3-5). Contributions from hydrogen bonding interactions are determined to be significant in governing alternative selectivity using the EPG phases relative to alkyl stationary phases. Representative chromatograms for the separation of resorcinols and catechols on both a C18 and EPG phase demonstrate alternative selectivity.

Column Classification Study

The column classification study was conducted according to the protocol utilized by Euerby, et. al.(2). Ascentis C18 and Ascentis RP-Amide (amide-based EPG phase) columns (15 cm x 4.6 mm, 5 μ m) along with a Discovery C18 (15 cm x 4.6 mm, 5 μ m, as a control) were employed.

Linear Solvation Energy Relationship (LSER) Study

Retention data for 25 neutral analytes with known molecular descriptors were acquired in triplicate according to the following conditions:

column: Ascentis RP-Amide (565323-U) or Ascentis C18 (581323-U),
5 cm x 4.6 mm I.D., 5 μ m particles
mobile phase: 50:50 (v/v), water:methanol
flow rate: 1 mL/min.
temp.: 35 °C
det.: UV at 220 nm
injection: 10 μ L

Multiple linear regression analysis on the resultant data was performed using Minitab ver.13 (State College, PA USA)

Separation of Catechols and Resorcinols

column: Ascentis RP-Amide (565324-U) or Ascentis C18 (581324-U),
15 cm x 4.6 mm I.D., 5 μ m particles
mobile phase: 75:25, 20 mM phosphoric acid (pH 2.0 unadjusted):acetonitrile
temp.: 30 °C
flow rate: 1.5 mL/min.
det.: UV, at 270 nm
injection: 25 μ L
sample: 50 μ g/mL in 20 mM phosphoric acid, pH 2.0

Results and Discussion

The goal of a column classification protocol is to provide performance comparisons between the myriad of stationary phases that are commercially available. The HPLC practitioner can use this information to determine the brands of columns likely to provide similar performance for an existing method or what columns might provide different performance for method development purposes. The studies attempt to break down the important interactions that contribute to retention and selectivity using test probes that attempt to ascertain interaction presence and dominance on a given stationary phase. The column classification protocol reported by Euerby includes tests for hydrophobic retentivity (k' pentylbenzene, k' PB), hydrophobic selectivity (k'/k' amylbenzene/butylbenzene, α CH₂), shape selectivity (k' triphenylene/ k' o-terphenyl, α T/O), hydrogen bond selectivity (k' caffeine/ k' phenol, α C/P) and ion-exchange capacity at pH 2.7 and pH 7.6 (k' benzylamine/ k' phenol, α B/P, 2.7 and α B/P, 7.6, respectively). By comparing the values obtained for different stationary phases, specific interactions can be identified as having relatively greater or lesser contributions to overall retention.

A C18 stationary phase, Ascentis C18 and an EPG phase, Ascentis RP-Amide were compared using the Euerby protocol and the results are presented in Table 1. In terms of selectivity differences between the phases, the hydrogen bonding capacity is observed to be dominant. Caffeine is a hydrogen bond acceptor whereas phenol is a hydrogen bond donor.

The lower value obtained on the EPG phase signifies that the phase preferentially retains the hydrogen bond donating phenol (denominator) compound. Based on these data, it is concluded that the amide stationary phase acts as a hydrogen bond acceptor.

Table 1. Column Classification Results

Column	k'PB	α CH ₂	α T/O	α C/P	α B/P, 2.7	α B/P, 7.6
Discovery C18 (control)	2.92	1.42	1.52	0.37	0.08	0.28
Ascentis C18	7.35	1.50	1.59	0.37	0.08	0.31
Ascentis RP-Amide	5.23	1.48	1.61	0.24	0.05	0.25

Linear solvation energy relationship studies attempt to relate fundamental molecular solute descriptors such as molecular volume (V), polarizability (S), electron lone pair interactions (E) and hydrogen bonding interactions (donating/acidity, A and accepting/basicity, B) to the free energy related to a phase transfer process (differential interaction with the mobile phase and stationary phase) through equations such as Eq. 1.

$$\text{Eq 1: } \log k' = c + eE + sS + aA + bB + vV$$

Where k' is the capacity factor for a given probe analyte, c is a constant and e , s , a , b and v are stationary phase characteristics that complement the analyte interaction descriptors. Retention data for a set of probe analytes with known interactions properties may be used to elucidate the dominant interactions contributing to retention on a given stationary phase using multiple linear regression analyses (for further information on LSER studies see references 3-5).

In the present study, Ascentis C18 and Ascentis RP-Amide were once again compared using the LSER approach. The data presented in Table 2 indicate that the polarization and hydrogen bonding terms are statistically different between the two stationary phase chemistries and thus contribute to the differences in selectivity often observed when employing the phases. Figure 1 shows the preferential retention of phenolic and aniline compounds used in the study on the EPG phase. Again, the preferential retention of compounds capable of donating toward a hydrogen bond is observed. The LSER study also implicates polar interactions (such as dipole-dipole) as a potentially differentiating interaction between EPG and C18 phases.

With hydrogen bonding being implicated as a dominant contributor to differential retention and selectivity on EPG phases, the separation of a group of catechols and resorcinols was compared on the amide and C18 phases. Figure 2

Figure 1. Preferential Retention of Hydrogen Bond Donors on EPG Phases

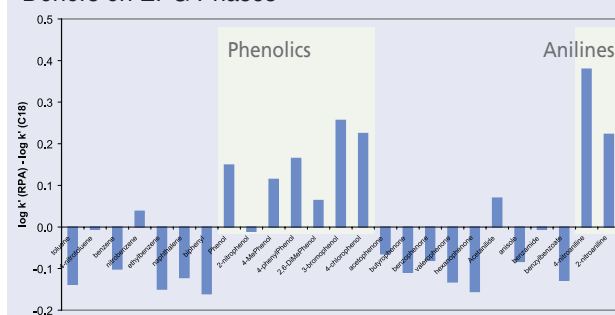
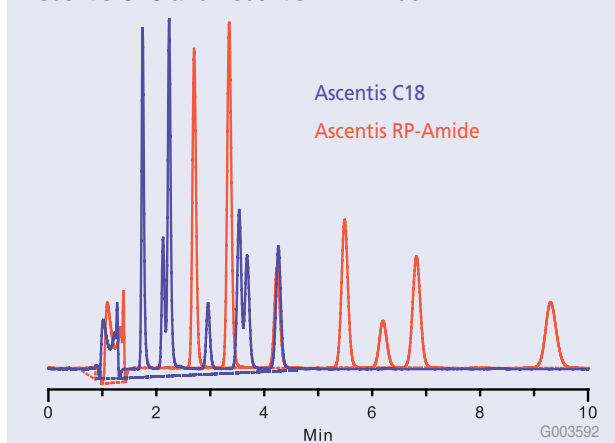


Figure 2. Separation of Catechols and Resorcinols on Ascentis C18 and Ascentis RP-Amide



demonstrates both enhanced retention and alternative selectivity for the hydrogen bond donors using the EPG phase.

Conclusions

Hydrogen bonding plays a key role in the alternative retention and selectivity observed for EPG phases when compared to traditional C18 chemistries. The role of hydrogen bonding is supported by column classification studies, fundamental LSER investigations and in specific application. When stationary phase chemistries other than traditional alkyl phases are required for the separation of hydrogen bond donating analytes, EPG phases such as the Ascentis RP-Amide should be explored as a viable alternative.

References

1. M.R. Euerby, McGeown, A. P., Petersson, P., J. Sep. Sci. 26 (2003) 295.
2. M.R. Euerby, P. Petersson, J. Chromatogr. A 994 (2003) 13.
3. L.C. Tan, Carr, P. W., Abraham M. H., J. Chromatogr. A 752 (1996) 1.
4. A. Wang, P.W. Carr, J. Chromatogr. A 965 (2002) 3.
5. J. Zhao, P.W. Carr, Anal. Chem. 70 (1998) 3619.

Table 2. LSER Multiple Linear Regression Analysis Results

Stationary Phase	c	v	e	s	a	b
Ascentis RP-Amide	-0.496±0.06	2.23±0.07	0.145±0.08	-0.385±0.07	0.068±0.05	-2.51±0.13
Ascentis C18	-0.421±0.09	2.30±0.11	0.267±0.11	-0.731±0.10	-0.256±0.08	-2.10±0.18

Polar Compound Retention using Aqueous Normal-Phase (ANP/HILIC) Chromatography

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Introduction

Aqueous Normal-Phase (ANP) Chromatography, frequently referred to as Hydrophilic Interaction Liquid Chromatography (HILIC), is a variation of normal-phase chromatography with the distinction that one of the major components of the mobile phase is water. Typical eluents of ANP consists of 50-95% acetonitrile in water or an aqueous buffer. The high volatility of the eluent makes ANP LC-MS friendly where one can realize a dramatic increase in sensitivity compared to reversed-phase chromatography. A requirement for ANP is a polar stationary phase. Common phases include bare silica, fluorinated phases such as Discovery HS F5, cyano, diol, and even certain phenyl phases such as the Ascentis Phenyl.

Many classes of polar compounds can be retained in ANP. These include polar neutrals, polar acids, and polar and non-polar basic amines. Both polar and ionic interactions appear to contribute to retention and selectivity in this mode of chromatography. HILIC retention can be simply described as the partitioning of a polar analyte into and out of an adsorbed water enriched layer on the surface of the column. On the other hand, charged polar analytes can also undergo ion-exchange with the silanol groups on the surface. It is important to understand that either mechanism can dominate depending on the analyte and the mobile phase conditions.

Benefits of ANP/HILIC Separation

- Retain polar compounds that are too water soluble to be retained by reversed-phase chromatography
- Achieve complimentary selectivity to reversed-phase chromatography
- Gain enhanced sensitivity in mass spectrometry
- Facilitate recovery of preparative chromatography fractions due to high-volatility mobile phase

Biogenic amines is one class of compounds that can benefit from the use of ANP chromatography. Shown in Figure 1 is the analysis of four biogenic amines utilizing the Ascentis Si column under ANP conditions. These small, polar compounds can be difficult to analyze by traditional reversed-phase chromatography utilizing C18 or other hydrophobic columns.

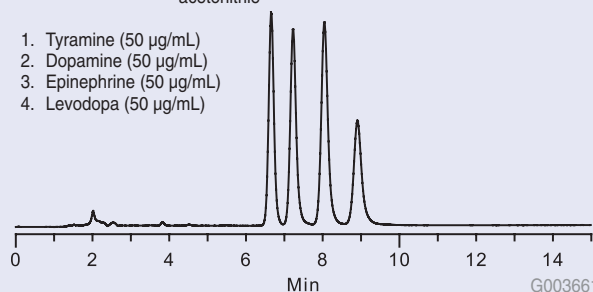
Method Considerations

A consideration when using ANP chromatography is that many features are the opposite of reversed-phase chromatography. For example, add more organic solvent to

Figure 1. Separation of Biogenic Amines on Ascentis Si

column: Ascentis Si, 15 cm x 4.6 mm I.D., 5 µm particles (581512-U)
mobile phase: 15:85, 0.1% ammonium acetate in water (pH unadjusted):
0.1% ammonium acetate in acetonitrile
temp.: 35 °C
flow rate: 1 mL/min
det.: UV at 280 nm
injection: 10 µL
sample: as indicated in 0.1% ammonium acetate in 10:90, water:
acetonitrile

1. Tyramine (50 µg/mL)
2. Dopamine (50 µg/mL)
3. Epinephrine (50 µg/mL)
4. Levodopa (50 µg/mL)



the mobile phase to increase retention. Run gradients from low aqueous content to high aqueous content. Dilute samples with the organic solvent, not water. Finally, ion-exchange can be a dominant mechanism, so adjusting buffer pH and ionic strength can have a dramatic effect on retention.

HPLC Columns for ANP/HILIC

Supelco offers four phases suitable for ANP chromatography. Each phase will perform differently in ANP chromatography but the following table provides a guide to get started.

Supelco Phase	Analytes
<i>Ascentis Si</i>	polar neutrals, polar bases, polar acids
<i>Ascentis Phenyl</i>	polar and non-polar bases
<i>Discovery HS F5</i>	polar and non-polar bases
<i>Discovery Cyano</i>	polar neutrals, polar and non-polar bases

+ Related Products

Description	Cat. No.
Ascentis Si HPLC Columns	
5 cm x 2.1 mm I.D., 3 µm particles	581500-U
15 cm x 4.6 mm I.D., 3 µm particles	581506-U
5 cm x 2.1 mm I.D., 5 µm particles	581507-U
10 cm x 2.1 mm I.D., 5 µm particles	581508-U
15 cm x 4.6 mm I.D., 5 µm particles	581512-U
25 cm x 4.6 mm I.D., 5 µm particles	581513-U

! Related Information

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

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Silica

C18

C8



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- Low bleed for sensitive LC-MS and UV applications.
- High reproducibility for easy method validation.

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Supelco Scientists have developed hundreds of applications for all Ascentis phases. These applications have been conveniently placed on a searchable CD to assist you with method development and choosing appropriate columns. Our technical service department will gladly provide you with an Ascentis Application CD and other technical advice before and after the sale.

For more information on Supelco's Ascentis HPLC Columns, contact your local sales office, technical service or visit our website at our website at sigma-aldrich.com/ascentis

MOLECULAR BIOLOGY

CELL CULTURE

CELL SIGNALING

PROTEOMICS

ANALYTICAL

LABORATORY ESSENTIALS

DRUG DISCOVERY

LC-MS Ionization Effect from Solid Phase Extraction Extractables

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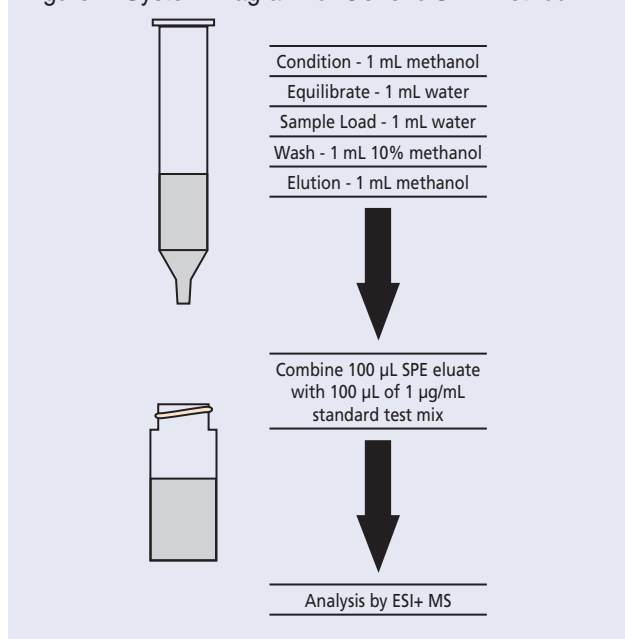
Abstract/Introduction

Ionization suppression (ion-suppression) in electrospray ionization (ESI) is an ever-growing concern for LC-MS users. Ion-suppression and/or -enhancement can greatly inhibit the ability for accurate quantitation. There have been many studies on the effect of matrix-induced ion suppression from biological samples and each use some form of sample preparation to remove interfering matrices. The process of solid phase extraction (SPE) itself can often induce ionization effects during MS analysis. This is potentially due to extractables endogenous to the SPE hardware (polypropylene cartridges/ 96-well plates) that co-elute with analytes of interest during SPE methodology. Although SPE is widely used in mass spectrometry, there have been few studies on the effect of extractables during the process of SPE on ionization. Known impurities in cartridges such as polyethylene glycols (PEGs) and phthalates are readily extracted with

commonly used organic solvents. Elution conditions for solid phase extraction typically require high organic solvents modified for pH or ionic strength. Although the SPE cartridges are designed for one time use, such elution conditions can cause deterioration of the stationary phase. These impurities are then eluted with the sample and may affect ionization either by suppression or enhancement.

The objective was to study what effect the SPE phases contribute to ionization suppression/enhancement in electrospray mass spectrometry. Several commonly used SPE phase chemistries were evaluated with a generic elution condition to measure the ionization effects from extractables. The phases that were evaluated were a standard C18, strong anion-exchange, strong cation-exchange, mixed-mode and a hydrophilic polymer. An empty polypropylene SPE cartridge with frits was also evaluated. A range of acidic, neutral and basic compounds (standard test mix) was used as a control to measure the effect of solid phase extraction extracts on MS signal suppression/enhancement.

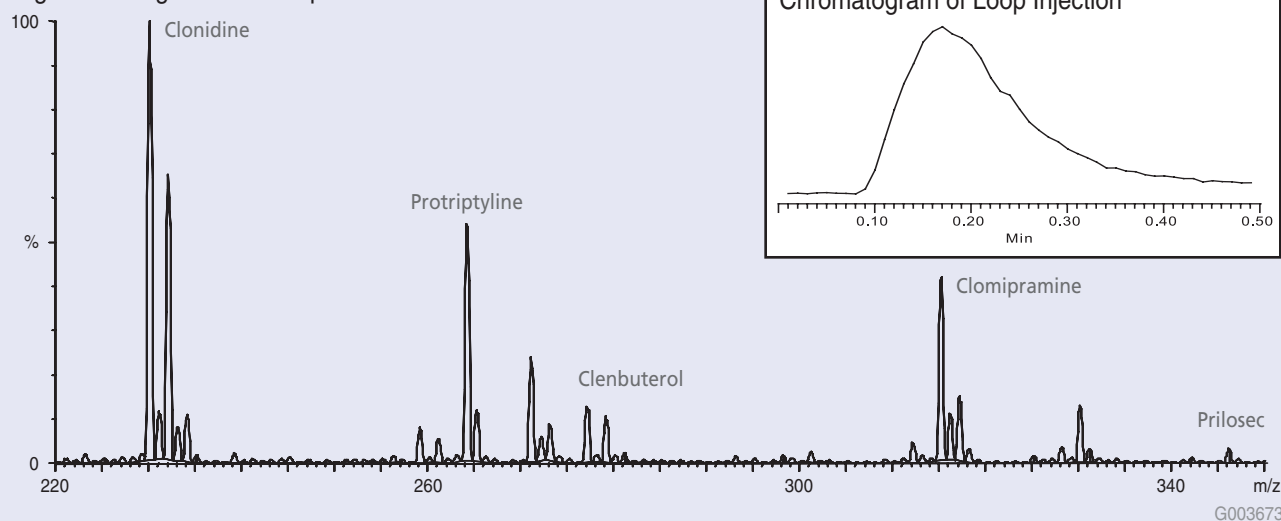
Figure 1. System Diagram for Generic SPE Method



Methodology

The generic sample preparation procedure is detailed in Figure 1. Pure DI water was loaded onto each cartridge pre-conditioned with methanol and DI water. The cartridges were then washed with a 10% methanol followed by methanol elution. After collecting the eluate from the SPE, an aliquot of the eluate was spiked with an equal volume of the 1.0 µg/mL standard test mix. The post-SPE spiked eluate was injected directly into the LC-MS instrument for analysis (no analytical chromatography). All testing was performed on a Waters Micro Mass ZQ single quadrupole mass spectrometer with electrospray ionization in positive ion mode (ESI+). The sample was introduced directly into the MS by performing an automated loop injection with a carrier solvent of 50:50 methanol:water (LC-MS grade). The analysis was performed by taking a cumulative spectral scan across the entire chromatographic peak as shown in Figure 2. The resulting mass spectrum was then integrated, and the area for each standard ion was tabulated. The quantitation of samples was performed using a bracketed calibration table. This was necessary to account for drift in signal response that occurred due to contamination of the source.

Figure 2. Integrated Mass Spectra of Standard Mixture

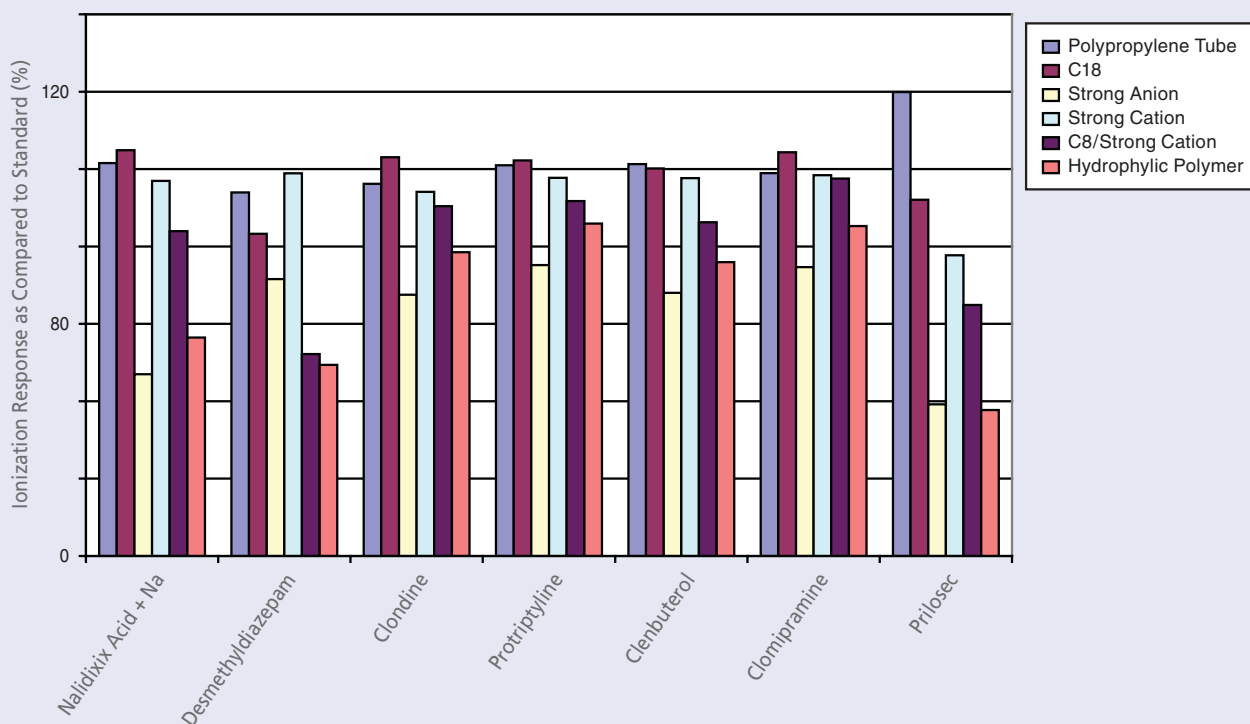


Results

The results of the ion-suppression study are summarized in Figure 3. The effect on ionization varies significantly when comparing the response from the different phase chemistries. The non-polar hydrophobic C18 reversed phase chemistry contributed little effect on the ionization of the standard compounds, this was also observed with the extractables from the polypropylene tube. As to be expected, extractables from the strong anion-exchange phase had the greatest effect on ionization with up to

60% ion-suppression. This can be explained from the mechanism of charge competition in the ESI+ source as these extractables are readily ionizable in positive ionization mode. The strong cation-exchange stationary phase exhibited some ionization effect but not significant. The acidic functionality of this phase is not as readily ionizable in ESI+ mode as compared to the strong anion exchange phase. Though ion-suppression, due to ion-pairing from the strong cation-exchange SPE extractables

Figure 3. Ionization Effect of SPE Extractables Using Generic SPE Method



with the basic standard compound producing a neutral species was expected, this was not observed. The mixed mode C8/ strong cation exchange also contributed up to 50% ion-suppression. We suspect that this was due to the total concentration of extracted ionizable species. The high concentration of extractables increases the surface tension of the ESI+ droplet preventing charged ions from forming. The polymeric phases also exhibited up to a 60% ion-suppression. This can be due to the modification performed on the polymer to enhance the hydrophilic properties. These polar functional groups are also readily ionizable and act in competition with the test compounds.

Conclusion

This observation supports the need for calibration standards to be prepared in the blank eluate derived from the SPE method. Often when measuring absolute recovery from SPE cartridges, the stock standard is used for calibration. By doing so, this does not account for the signal suppression from the phase bleed and is assumed to be due to low recovery from the SPE cartridge. Using

the eluate from the SPE cartridge to prepare the calibration standards enables a more accurate measurement of the absolute recovery from the SPE cartridge.

References

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Silica				
23 x 53 mm	20-45	11	15	1.1
23 x 110 mm	20-45	23	30	2.3
40 x 75 mm	45-75	51	60	5.0
40 x 150 mm	45-75	102	120	10.0
80 x 150 mm	45-75	410	480	41.0
80 x 300 mm	45-75	820	960	82.0
110 x 300 mm	45-75	1320	1850	130.0
C18				
23 x 53 mm	20-45	15	11	1.5
23 x 110 mm	20-45	30	20	3.0
40 x 75 mm	45-75	70	30	7.0
40 x 150 mm	45-75	140	60	14.0
80 x 150 mm	45-75	515	240	51.0
80 x 300 mm	45-75	1050	480	105.0
110 x 300 mm	45-75	1920	910	190.0

[▲] Several factors influence the maximum loading capacity of the column including: similarity of compounds, sample matrix, concentration of reaction products and the elution solvent used.



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A Selection of New Applications on the Supelco Low Bleed SLB-5ms

Katherine Stenerson, Jay Jones,
and Michael D. Buchanan
kstenerson@sial.com

Introduction

In past Reporter articles, we have highlighted the many features and benefits of the new SLB-5ms column, especially for US EPA Method 8270. In addition to Method 8270, we have also investigated the use of this column for analyses in other applications. In this article, we will highlight several of these applications.

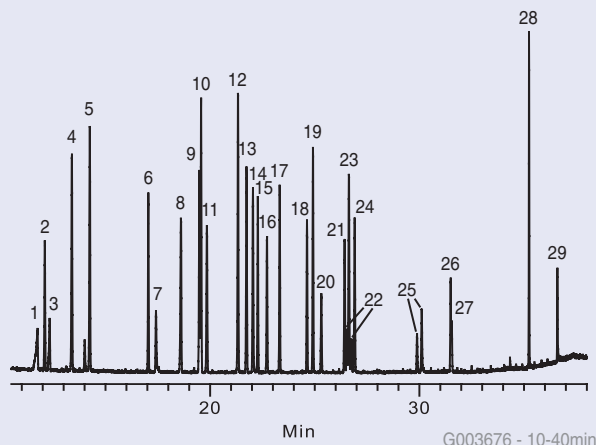
Agricultural Chemicals on Golf Courses

The run-off from golf courses treated with a variety of pesticides represents a potential environmental problem. Specifically, in Japan, the increased use of these compounds on golf courses has raised concerns. Figure 1 shows the separation of 29 "golf course pesticides" (compounds classified as pesticides, insecticides, herbicides, and fungicides) that were included in a list published by the

Figure 1. Agricultural Chemicals on Golf Courses

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m (28471-U)
oven: 50 °C (0.2 min.), 100 °C/min. to 100 °C, 5 °C/min. to 250 °C,
15 °C/min. to 325 °C (5 min.)
inj.: 250 °C
MSD interface: 300 °C
scan range: m/z 40-450
carrier gas: helium, 30 cm/sec constant
injection: 1 μ L, splitless (0.75 min.)
liner: 2.6 mm I.D., straight
sample: 10 ppm pesticides in ethyl acetate

- | | | |
|----------------------------|---------------------|---------------------------|
| 1. Acephate | 11. Chlorothalonil | 21. Butamifos |
| 2. Etridiazol | 12. Terbucarb | 22. Siduron isomers |
| 3. Chlorofos (trichlorfan) | 13. Toclofos-methyl | 23. Napropamide |
| 4. Chloroneb | 14. Metalaxyl | 24. Isoprothiolane |
| 5. Mecoprop (methyl ester) | 15. Dithiopyr | 25. Propiconazole isomers |
| 6. Benfluralin | 16. Fenitrothion | 26. Pyridaphenthion |
| 7. Thiodicarb | 17. Chlorpyrifos | 27. Iprodione |
| 8. Simazine | 18. Pendimethalin | 28. Ethofenprox |
| 9. Propyzamide | 19. Isufenphos | 29. Azoxystrobin |
| 10. Diazinon | 20. Captan | |



Japanese Ministry of the Environment in 2003, and were found in a large number of run-off samples taken from golf courses around the country (1).

Drugs of Abuse

A challenge for forensic and clinical chemists is the need to rapidly screen for a wide range of possible drugs. As such, accuracy and sensitivity are critical. Figure 2 shows the separation of 16 common drugs of abuse on an SLB-5ms column. The inertness of the SLB-5ms resulted in symmetrical peak shapes for these compounds. In addition, the low bleed of the SLB-5ms will allow for greater sensitivity and better quality mass spectral confirmation at trace levels.

Agricultural Pesticides in Wine

Figure 3 demonstrates the usefulness of Solid Phase Microextraction (SPME) in the low-level extraction of agricultural pesticides from wine, and the use of the SLB-5ms in the subsequent analysis. The pesticides chosen for the analysis represent a group of insecticides and fungicides that could be found in wines due to their use in commercial

Figure 2. Drugs of Abuse

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.50 μ m (28473-U)
oven: 45 °C (2.0 min.), 30 °C/min. to 110 °C (1.0 min.), 15 °C/min.
to 200 °C (1.0 min.), 4 °C/min. to 310 °C (5.0 min.)
inj.: 250 °C
MSD interface: 330 °C
scan range: m/z 40-450
carrier gas: helium, 0.7 mL/min. constant
injection: 0.5 μ L, pulsed splitless, 30 psi. (0.20 min.), purge on
(1.50 min.), purge flow (50.0 mL/min.)
liner: 2mm I.D., straight
sample: drugs of abuse standard diluted to 100 ppm in methanol

- | | | |
|--------------------|------------------|--------------|
| 1. Methamphetamine | 7. Methadone | 13. Morphine |
| 2. Nicotine | 8. Amitriptyline | 14. Heroin |
| 3. Caffeine | 9. Cocaine | 15. Prazepam |
| 4. Diphenhydramine | 10. Desipramine | 16. Fentanyl |
| 5. Lidocaine | 11. Codeine | |
| 6. Phenobarbital | 12. Diazepam | |

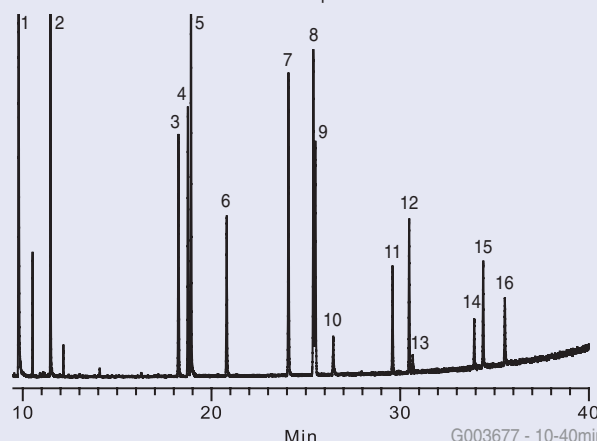
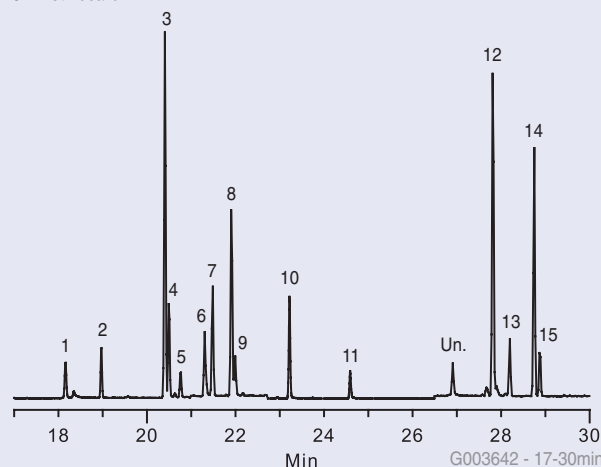


Figure 3. Agricultural Pesticides in Wine

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m (28471-U)
 SPME fiber: 85 μ m polyacrylate (57304)
 extraction: immersion, room temp. (30 min.)
 desorption: 5 min. at 250 $^{\circ}$ C
 oven: 60 $^{\circ}$ C (1 min.), 15 $^{\circ}$ C/min. to 100 $^{\circ}$ C, 7 $^{\circ}$ C/min. to 300 $^{\circ}$ C (1 min.)
 MSD interface: 325 $^{\circ}$ C
 scan range: SIM
 carrier gas: helium, 0.7 mL/min., constant
 liner: 0.75 mm I.D. SPME liner
 sample: white wine spiked with 50 ppb pesticides

- | | | |
|------------------------|--------------------|----------------------|
| 1. Dieldrin | 7. Dichlofluanid | Un. Unknown |
| 2. Diazinon | 8. Parathion-ethyl | 12. Imidan (Phosmet) |
| 3. Chlorpyrifos-methyl | 9. Triadimefon | 13. Dicofol |
| 4. Vinclozolin | 10. Procymidone | 14. Phosalone |
| 5. Carbaryl | 11. Myclobutanil | 15. Azinphos-methyl |
| 6. Methiocarb | | |



vineyards (2). These compounds contain a variety of polar functional groups, and the polyacrylate fiber provided the selectivity necessary for extraction from a wine matrix. The inertness and low bleed of the SLB-5ms enabled subsequent low-level analysis of these compounds by GC-MS.

Trichloroanisole in Wine

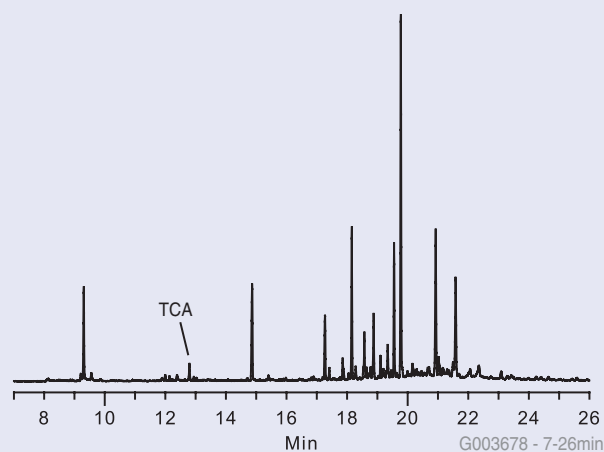
The ability of headspace SPME, in combination with analysis on the SLB-5ms, to detect a low level of 2,4,6-trichloroanisole (TCA) in wine is shown in Figure 4. TCA is often the source of the musty smell in wine resulting from tainted corks. Red wine obtained in a bottle fitted with a metal screw cap was spiked with TCA and the headspace was extracted using SPME. Analysis was done by GC-MS/SIM on the SLB-5ms. The compound was detected in a spiked wine sample at 5 parts-per-trillion (ppt), which is well below the 40 ppt sensory threshold level in red wines (3).

Conclusion

The SLB-5ms column has been designed to provide the user with the low bleed and inertness to meet the demands of today's sensitive GC-MS and GC applications. Additional applications using the SLB-5ms can be viewed or downloaded at sigma-aldrich.com/slb. If you have an idea for an application that you would like to see on the

Figure 4. Trichloroanisole in Wine

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m (28471-U)
 SPME fiber: 100 μ m PDMS metal fiber (57928-U)
 extraction: headspace, room temp (30 min.)
 desorption: 3 min. at 250 $^{\circ}$ C
 oven: 60 $^{\circ}$ C (2 min.), 8 $^{\circ}$ C/min. to 200 $^{\circ}$ C (10 min.)
 MSD interface: 300 $^{\circ}$ C
 scan range: SIM, m/z = 195, 197, 210, 212
 carrier gas: helium, 1 mL/min. constant
 liner: 0.75 mm I.D. SPME
 sample: red wine spiked with 5 ppt TCA



SLB-5ms, or any Supelco column, please feel free to drop us an e-mail at the address listed at the beginning of this article.

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1. News Release, FY 2003 Survey Results of Water Pollution by Agricultural Chemicals Used at Golf Courses, Japanese Ministry of the Environment, 11/12/2004, www.env.go.jp/en/press/2004/1112a.
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3. Riu, M.; Mestres, M.; Busto, O.; Guasch, J., Determination of 2,4,6-Trichloroanisole in Wines by Headspace Solid-phase Microextraction and Gas Chromatography-electron-capture detection. *J. Chromatogr. A*, 2002, 977: 1-8.

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SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m, Beta = 250	28471-U
SLB-5ms, 30 m x 0.25 mm I.D., 0.50 μ m, Beta = 125	28473-U
SPME fiber, 85 μ m polyacrylate on fused silica, 24 gauge, pack of 3	57304
SPME fiber, 100 μ m PDMS on metal, 23 gauge, pack of 1	57928-U

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For more information on SPME, request T199925 (CJQ) or visit sigma-aldrich.com/supelco-spme

Separate Source Chemical Standards Meet Auditor Requirements

Vicki Yearick

vyearick@sial.com

NELAC (National Environmental Laboratory Accreditation Conference) requires that standards used for initial instrument calibration to be of a separate source from daily continuing verification standards. This covers a wide range of US EPA methods. Separate source standards from Supelco afford laboratories the convenience of dealing with a single vendor for obtaining these separate standards, as well as, eliminating the need to buy and inventory raw materials to prepare their own separate source standards.

Separate source standards are pairs of products having identical composition that are prepared from independently sourced raw materials and are independently quality controlled. The extensive environmental standards product line offered by Supelco includes more than 90 different separate source reference standards for use by accredited environmental testing facilities.

Supelco's internal processes prevent the use of the same raw material lot for both the primary and secondary standards. This can be verified by comparing the raw

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Separate source standards include neat compounds, single component solutions, and mixtures. They are available for the US EPA drinking water, wastewater, and solid waste methods. The standards are identified by the words "separate source" at our website and by the catalog number (4- and 4S-, 5 and 5S-, 8- and 8S-). For example, 4S8913 is the separate source standard for 48913. As an added tool, a link at the Sigma-Aldrich Analytical Standards website directs you to the separate source products.

Listed below are several examples of our separate source standards. To view a complete list of all Supelco brand separate source reference standards, including analytes, solvent matrices, and concentrations, please visit our Analytical Standards web site sigma-aldrich.com/standards

Description	Concentration	Cat. No.
EPA 502/524 Volatiles Organic Calibration Mix A (w/o gases)	2000 µg/mL each component in methanol	502111
	<div style="display: flex; flex-wrap: wrap;"> <div style="width: 33%;"> <p>1,1,1,2-Tetrachloroethane</p> <p>1,1,1-Trichloroethane</p> <p>1,1,2,2-Tetrachloroethane</p> <p>1,1,2-Trichloroethane</p> <p>1,1-Dichloroethane</p> <p>1,1-Dichloroethene</p> <p>1,1-Dichloropropene</p> <p>1,2,3-Trichlorobenzene</p> <p>1,2,3-Trichloropropane</p> <p>1,2,4-Trichlorobenzene</p> <p>1,2,4-Trimethylbenzene</p> <p>1,2-Dibromo-3-chloropropane</p> <p>1,2-Dibromoethane</p> <p>1,2-Dichlorobenzene</p> <p>1,2-Dichloroethane</p> <p>1,2-Dichloropropane</p> <p>1,3,5-Trimethylbenzene</p> <p>1,3-Dichlorobenzene</p> </div> <div style="width: 33%;"> <p>1,3-Dichloropropane</p> <p>1,4-Dichlorobenzene</p> <p>2,2-Dichloropropane</p> <p>2-Chlorotoluene</p> <p>4-Chlorotoluene</p> <p>Benzene</p> <p>Bromobenzene</p> <p>Bromochloromethane</p> <p>Bromodichloromethane</p> <p>Bromoform</p> <p>Carbon tetrachloride</p> <p>Chlorobenzene</p> <p>Chloroform</p> <p>Dibromochloromethane</p> <p>Dibromomethane</p> <p>Ethylbenzene</p> <p>Hexachlorobutadiene</p> <p>Isopropylbenzene</p> </div> <div style="width: 33%;"> <p>Methylene chloride</p> <p>Naphthalene</p> <p>Styrene</p> <p>Tetrachloroethene</p> <p>Toluene</p> <p>Trichloroethene</p> <p>cis-1,2-Dichloroethene</p> <p>cis-1,3-Dichloropropene</p> <p>m-Xylene</p> <p>n-Butylbenzene</p> <p>n-Propylbenzene</p> <p>o-Xylene</p> <p>p-Isopropyltoluene</p> <p>p-Xylene</p> <p>sec-Butylbenzene</p> <p>tert-Butylbenzene</p> <p>trans-1,2-Dichloroethene</p> <p>trans-1,3-Dichloropropene</p> </div> </div>	<div style="display: flex; align-items: center;"> SS 5502111 </div>
Trihalomethanes Calibration Mix	200 µg/mL each component in methanol	
	<div style="display: flex; flex-wrap: wrap;"> <div style="width: 33%;"> <p>Bromodichloromethane</p> <p>Bromoform</p> </div> <div style="width: 33%;"> <p>Chloroform</p> <p>Dibromochloromethane</p> </div> </div>	<div style="display: flex; align-items: center;"> SS </div>
EPA VOC Calibration Standards Kit		48804
	<p>EPA Volatile Organic Compounds Mix 1 (48775), 1 mL</p> <p>EPA Volatile Organic Compounds Mix 2 (48777), 1 mL</p> <p>EPA Volatile Organic Compounds Mix 3 (48779), 1 mL</p> <p>EPA Volatile Organic Compounds Mix 4 (48786), 1 mL</p> <p>EPA Volatile Organic Compounds Mix 5 (48797), 1 mL</p> <p>EPA Volatile Organic Compounds Mix 6 (48799-U), 1 mL</p>	<div style="display: flex; align-items: center;"> SS 458804 </div>
VOC Mix 6	2000 µg/mL each component in methanol	48799-U
	<div style="display: flex; flex-wrap: wrap;"> <div style="width: 33%;"> <p>Bromomethane</p> <p>Chloroethane</p> <p>Chloromethane</p> </div> <div style="width: 33%;"> <p>Dichlorodifluoromethane</p> <p>Trichlorofluoromethane</p> <p>Vinyl chloride</p> </div> </div>	<div style="display: flex; align-items: center;"> SS 458799 </div>

Separate Source Chemical Standards Meet Auditor Requirements

Description	Concentration	Cat. No.	
CLP Semivolatile Calibration Mix	1000 µg/mL each component in methylene chloride:benzene (75:25)	506508 5506508	
	1,2,4-Trichlorobenzene 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol 2,4-Dichlorophenol 2,4-Dimethylphenol 2,4-Dinitrophenol 2,4-Dinitrotoluene 2,6-Dinitrotoluene 2-Chloronaphthalene 2-Chlorophenol 2-Methylnaphthalene 2-Methylphenol 2-Nitroaniline 2-Nitrophenol 3-Nitroaniline 4,6-Dinitro-2-methylphenol 4-Bromophenylphenyl ether 4-Chloro-3-methyl phenol 4-Chloroaniline	4-Chlorophenyl phenyl ether 4-Methylphenol 4-Nitroaniline 4-Nitrophenol Acenaphthene Acenaphthylene Anthracene Azobenzene Benzo[a]anthracene Benzo[a]pyrene Benzo[b]fluoranthene Benzo[ghi]perylene Benzo[k]fluoranthene Bis (2-chloroisopropyl) ether Bis(2-chloroethoxy)methane Bis(2-chloroethyl) ether Bis(2-ethylhexyl) phthalate Butyl benzyl phthalate Carbazole Chrysene Di-n-butyl phthalate Di-n-octyl phthalate	Dibenzo[a,h]anthracene Dibenzofuran Diethyl phthalate Dimethyl phthalate Fluoranthene Fluorene Hexachlorobenzene Hexachlorobutadiene Hexachlorocyclopentadiene Hexachloroethane Indeno[1,2,3-cd]pyrene Isophorone N-Nitrosodi-n-propylamine N-Nitrosodimethylamine Naphthalene Nitrobenzene Pentachlorophenol Phenanthrene Phenol Pyrene
TCL Polynuclear Aromatic Hydrocarbons Mix	2000 µg/mL each component in methylene chloride:benzene (50:50)	48905-U 458905	
	Acenaphthene Acenaphthylene Anthracene Benzo[a]anthracene Benzo[a]pyrene	Benzo[b]fluoranthene Benzo[ghi]perylene Benzo[k]fluoranthene Chrysene Dibenzo[a,h]anthracene	Fluoranthene Fluorene Indeno[1,2,3-cd]pyrene Naphthalene Phenanthrene
Pyrene			
TCL Pesticides Mix	2000 µg/mL each component in hexane:toluene (50:50)	48913 458913	
	α-BHC β-BHC γ-BHC (Lindane) δ-BHC 4,4'-DDD 4,4'-DDE	4,4'-DDT Aldrin Dieldrin Endosulfan I Endosulfan II Endosulfan sulfate	Endrin Endrin aldehyde Endrin ketone Heptachlor Heptachlor epoxide Methoxychlor

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5 µL	75ASN	26s ga. cone tip	43 mm	21314
5 µL	75ASN	23s - 26s ga. cone tip	43 mm	24571
Hamilton Gastight® Syringes				
5 µL	175ASN	23s ga. cone tip	43 mm	26700-U
5 µL	175ASN	23s - 26s ga. cone tip	43 mm	24577
SGE Syringes				
5 µL	SK-5F-HP-0.63	23 ga. cone tip	42 mm	21911
5 µL	SK-5F-HP-0.47	26 ga cone tip	42 mm	21910
5 µL	5F-HP-0.63/0.47	23 - 26 ga. cone tip	42 mm	26887-U
Hamilton Syringes				
10 µL	701ASN	23s ga. cone tip	43 mm	21317
10 µL	701ASN	26s ga cone tip	43 mm	21316
10 µL	701ASN	23s - 26s ga. cone tip	43 mm	24574
Hamilton Gastight Syringes				
10 µL	1701ASN	23s ga. cone tip	43 mm	26701
10 µL	1701ASN	23s - 26s ga. cone tip	43 mm	24580
SGE Syringes				
10 µL	SK-10F-HP-0.63	23 ga. cone tip	42 mm	21544
10 µL	SK-10F-HP-0.47	26 ga. cone tip	42 mm	21912
10 µL	10F-HP-0.63/0.47	23 - 26 ga cone tip	42 mm	26889-U
SGE Superflex® Syringes				
10 µL	SK-10FX-0.47	26 ga. cone tip	42 mm	23971
10 µL	SK-10FX-0.63	23 ga. cone tip	42 mm	23972
SGE Gastight Syringe				
10 µL	10F-HP-0.63/0.47	23 - 26 ga. cone tip	42 mm	26891-U

Syringe for Agilent 7670, 7671, and 7672 Autosamplers

Hamilton Syringe				
10 µL	701N	26s ga. bevel tip	51 mm	20779

Syringes for General Use

Hamilton Syringe				
10 µL	701RN	26s ga. bevel tip	51 mm	20793
SGE Syringes				
10 µL	10FX	26 ga. bevel tip	50 mm	23966
10 µL	10RX removable needle	26 ga. bevel tip	50 mm	23967

Did you know...?

Many GC methods require the use of on-column injections on wide-bore capillary columns. Syringes with a narrow, 26 gauge needle are needed to make these injections. However, 26 gauge needles do not stand up well to the repeated septum penetrations made by an autosampler. We recommend using a dual gauge needle with autosamplers. Dual gauge needles have a 23 gauge base and narrow down to 26 gauge at the tip. They provide the strength of the thicker 23 gauge needle while still allowing on-column injections.



Related Information

For more information on syringes, request JCS by ticking the box on the enclosed BRC.



Product Spotlight on FocusLiner™ Inlet Liners

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Introduction

Supelco now offers the FocusLiner series of inlet liners. These liners incorporate a unique design that prevents shifting of the wool plug during repeated injections or sudden inlet pressure changes. The use of a wool plug in inlet liners has been used for many years to promote the rapid vaporization of the entire sample, minimize mass discrimination, and prevent non-volatile material from entering the column. In addition, by wiping the sample from the needle tip, a wool plug significantly improves reproducibility by preventing droplet formation. The position of the wool plug is critical. It must be located so that the wool plug surrounds the needle tip as the sample is injected.

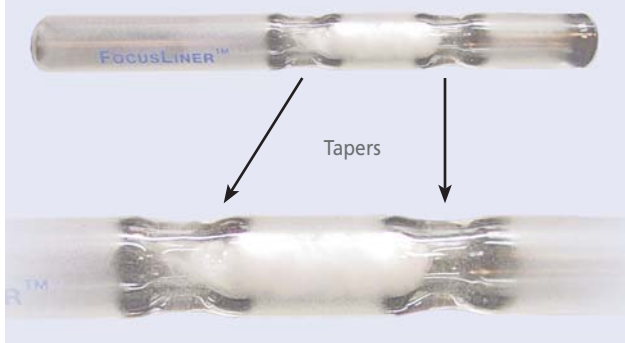
The Problem: Traditional Liners with Wool

With traditional inlet liners, the speed of many auto-samplers is sufficient to force the wool plug down below the optimal position for effective needle wiping. If there is an imperfection in the needle, such as a slight burr, this can grab the wool plug and pull it up towards the septum. Pressure changes, such as those that occur in pulsed splitless injection, can also move the wool plug. Even changing the column or septum can cause a rapid pressure drop and alter the position of the wool plug. Unfortunately, there is no guarantee that once a traditional liner is installed in the injector, the wool plug will stay in the correct position.

The Solution: FocusLiner Inlet Liners

With a FocusLiner inlet liner, tapers inside the liner stabilize the position of the wool plug, preventing the plug from shifting up or down. Figure 1 depicts this design. The wool plug is always correctly located. This position, unlike that found in a traditional inlet liner, provides a larger

Figure 1. FocusLiner Inlet Liner



surface area, allowing the sample to mix well prior to entering the column. More importantly, the location of the wool plug lets the tip of the needle penetrate the wool for maximum vaporization. The wool plug will also wipe any residual liquid sample from the needle tip to prevent droplet formation. This can improve reproducibility as much as 10 times, reduce solvent tailing, and lower mass discrimination during split injections. The effect is dramatic and typically reduces injection variability by at least 96%.

Conclusion

The use of a FocusLiner inlet liner provides precise, accurate, and reliable sample injections resulting in enhanced sample vaporization and maximum detection level sensitivity. Supelco offers FocusLiner inlet liners for all major GC instruments, most injection techniques, as well as for 0.10 mm I.D. Fast GC columns.

References

1. Technical Article TA-0004-A, 0.2% RSD's? It's Now a Reality with SGE's FocusLiner, SGE (www.sge.com)
2. Technical Article TA-0043-A, FocusLiner: Improve GC Accuracy and Reproducibility 10 Fold, SGE (www.sge.com)

+ Featured Products[▲]

Description	Cat. No.
Agilent, Split/Splitless, 78.5 x 6.3 x 4.0 mm	2879805-U
Agilent, Split/Splitless, 78.5 x 6.3 x 4.0 mm, single taper	2879905-U
PerkinElmer® AutoSystem™ and Clarus 500, Split/Splitless, 92 x 6.2 x 4.0 mm	2879205-U
PerkinElmer AutoSystem and Clarus 500, Split/Splitless, 92 x 6.2 x 4.0 mm, single taper	2879105-U
Shimadzu® 14/15A/16 with SPL-14 Injector, Split/Splitless, 99 x 5.0 x 3.4 mm	2878105-U
Shimadzu 14/15A/16 with SPL-14 Injector, Split/Splitless, 99 x 5.0 x 3.4 mm, single taper	2877805-U
Shimadzu 17A with SPL-17 Injector, Split/Splitless, 95 x 5.0 x 3.4 mm	2878605-U
Shimadzu 17A with SPL-17 Injector, Split/Splitless, 95 x 5.0 x 3.4 mm, single taper	2878405-U
Varian® 1075/1077 Injector, Split, 78.5 x 6.3 x 4.0	2875405-U
Varian 1075/1077 Injector, Split, 78.5 x 6.3 x 4.0, single taper	2874805-U
Varian 1078/1079 Injector, Split/Splitless, 54 x 5.0 x 3.4	2875705-U
Varian CP-1177 Injector, Split/Splitless, 78.5 x 6.3 x 4.0	2879805-U
Varian CP-1177 Injector, Split/Splitless, 78.5 x 6.3 x 4.0, single taper	2879905-U

▲ All of these FocusLiner inlet liners are packs of 5 and are packed with quartz wool. Additional pack sizes can be viewed at sigma-aldrich.com/focusliner

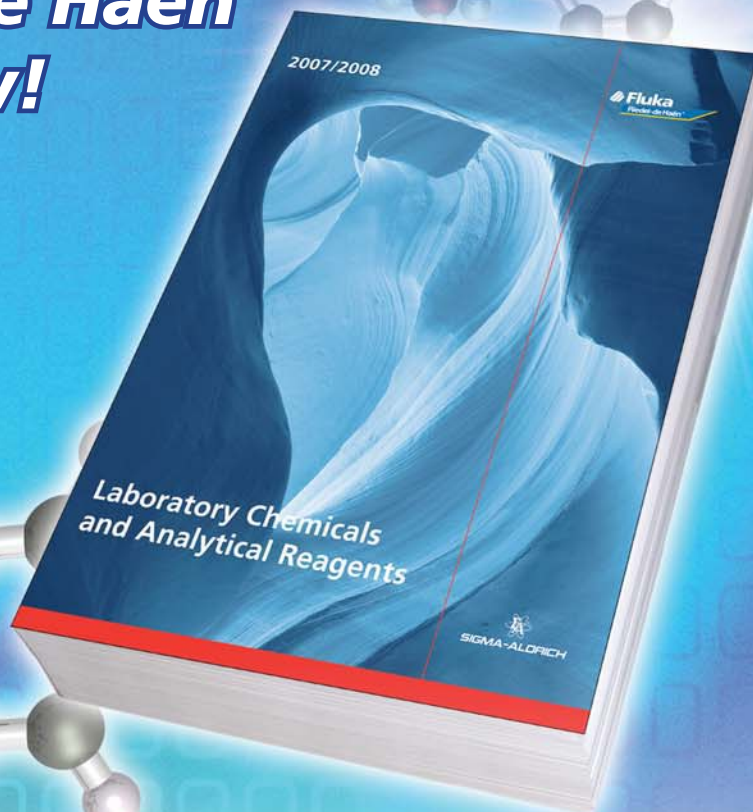
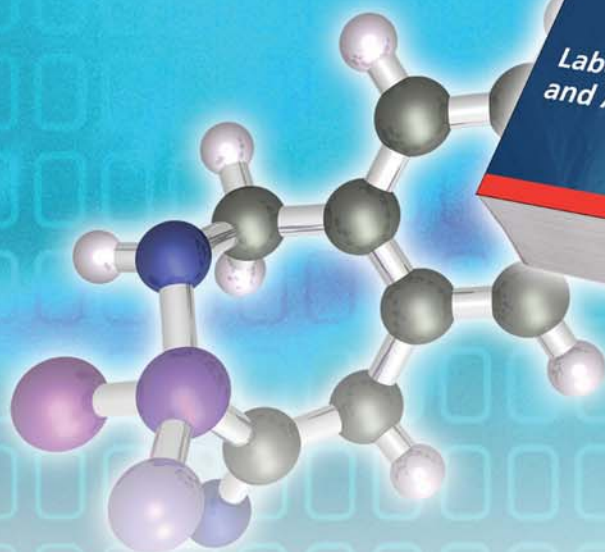
! Related Information

Our full line of FocusLiner inlet liners can be viewed at sigma-aldrich.com/focusliner. For more information on inlet liners, request T196899 (BBB) Bulletin 899 - *Capillary GC Inlet Liner Selection Guide* and T404081 (HCH) - *Selecting the Appropriate Inlet Liner Poster*.

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