

Reporter

Volume 28.2



Peptide Mapping with Ascentis Express Peptide ES-C18



The Fused-Core Advantage for Bioseparations

- Liquid Chromatography
- Sample Handling
- Gas Chromatography
- Standards
- Accessories
- Chiral Chromatography



Tracy Ascah

Product Manager

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Dear Colleague:

This year, one of Supelco's important educational outreach programs to the chromatography community is our “*Innovations in Chromatography and Sample Prep*” seminar series that we are conducting in many cities across North America.

How are the seminars educational? The answer is three-fold. First, we describe our innovative products in the context of the fundamental chromatography relationships. You will see not only that they work, but how they work (and possibly brush up on your van Deemter as well, if you're not careful!). Second, we show how these innovations can be put to use today, on existing instruments, so you can reap the benefits of speed, sensitivity or selectivity that they offer. Third, we have included guest speakers from academia, industry and government research who will talk on their particular areas of expertise in separation science.

We strive to make the seminars educational and interesting. Hopefully, they will also demonstrate that Supelco has a healthy pipeline of innovative products, based on sound fundamentals, to meet today's analytical challenges. If you are interested in attending or learning more, please visit the website sigma-aldrich.com/analytical-seminars for complete details and to register.

I'm speaking for the entire Supelco team when I say that I hope to see you at one of our seminars this year!

Kind regards,

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Peptide Mapping with Ascentis Express Peptide ES-C18

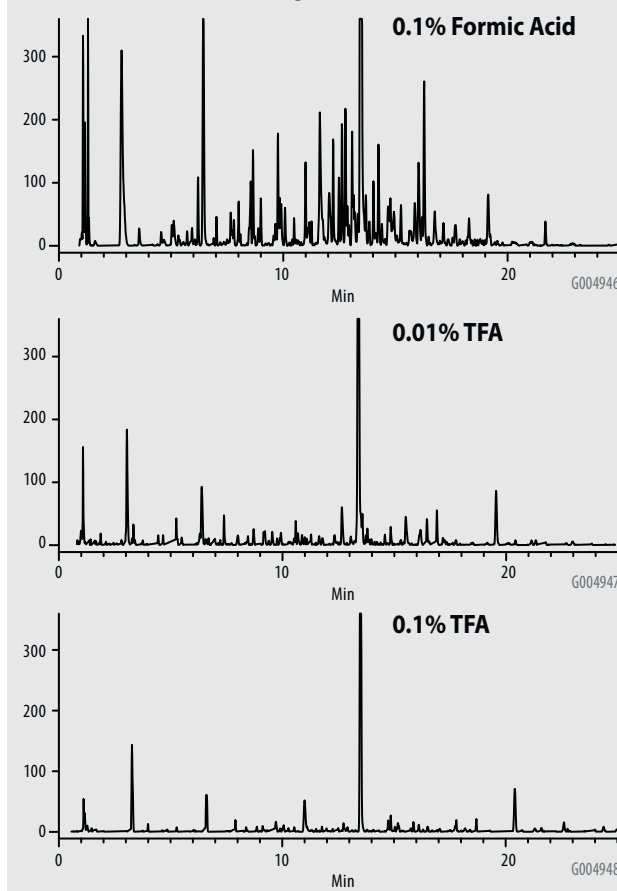
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Peptide mapping is an established technique for assessing changes to the primary structure of a protein. This has applications in areas of quality control or fundamental research in which changes to the protein sequence or to the chemical modification of amino acids is to be monitored. Essentially, the protein(s) is cleaved (digested) in a sequence-dependent manner to generate a finite number of peptide fragments. The mixture of peptide fragments is then resolved chromatographically. The exact pre-treatment protocol of the protein prior to digestion varies and may depend on the sort of questions the researcher is posing. However, a general protein pre-treatment protocol involves denaturation, reduction, and alkylation. Alkylation is performed to control the oxidation state of free sulfhydryls that otherwise can cause undesirable heterogeneity of the resulting chromatograms. Reduction is performed to ensure the sulfhydryls are reduced prior to alkylation. Denaturation is performed to ensure all sulfhydryls are accessible for chemical modification, as well as make the polypeptide chain fully accessible for digestion.

Peptide mapping is generally done by reversed-phase liquid chromatography. Traditionally this was done in conjunction with low-UV detection. A good match with the low-UV detection, is the use of perfluorinated organic acids as ion pairing reagents. TFA was ideal in this regard. As a strong acid, at low levels, it maximizes retention of polypeptides on silica-based columns by keeping the pH well below that of peptidyl carboxyls, and at the same time it ion pairs with basic moieties to optimize peak shape. Thus, TFA-mobile phases became the default method for peptide mapping with UV detection. However, the advent of mass-spectrometry as a preferred method of detection for peptide mapping necessitated the redevelopment of a standard mobile phase; TFA at levels typical for UV-based peptide mapping (0.1%) cause severe reduction in sensitivity as compared to other organic acids, as the mobile phase additive (1). This has been best described as a consequence of higher surface tension of the charged droplet in the ESI source and strong ion pairing in the gas phase, between TFA and peptide basic moieties (1). Acetic acid and formic acid have been used as alternatives to TFA for LC-MS of peptides, but formic acid has become more common. This is likely due, again, to it being a stronger acid, thus minimiz-

Figure 1. Sensitivity Comparison of Tryptic Digest with Different Ion Pair Reagents



ing the ionization of peptidyl carboxyls so as to enhance retention. An example of the difference in MS sensitivity with formic acid TFA is shown in Figure 1.

Supelco has recently released a new column on the Fused-Core™ particle platform designed for polypeptide analysis, Ascentis Express Peptide ES-C18. As such, it

exhibits high plate counts by virtue of the uniform column packing of the monodisperse particles, and a possible shorter diffusion path of the porous shell. Table 1 summarizes the major features of the Ascentis Express Peptide particle.

Table 1. Ascentis Express Peptide ES-C18 Fused-Core Particle

Silica	High Purity, type B
Solid Core	1.7 μm
Porous Shell	0.5 μm
Pore Size	160 \AA
Phase	Sterically Protected C18

(continued on page 4)

(continued from page 3)

$$P_c = \frac{t_g}{W_{ave}}$$

The major benefit of this new high-efficiency packing for peptide mapping is the resultant improved peak capacity (P_c), where t_g is the gradient time and w_{ave} is the average peak width.

Peak capacity provides a theoretical maximum number of peaks that can be chromatographically resolved over the gradient run time. A more efficient column will have lower average peak width, and will therefore exhibit higher peak capacity. An example of this is shown in Figure 2 and Table 2. Figure 2 displays chromatograms of a complex tryptic digest on a typical wide-pore C18 column, versus the Ascentis Express Peptide ES-C18. While visual inspection suggests a greater number of resolved peaks in the case of the Ascentis Express Peptide ES-C18 column, a more convincing quantitative evaluation comes from a sampling of peaks across the chromatogram, as shown in Table 2. Ascentis Express Peptide ES-C18 is able to resolve, statistically, a greater number of peaks.

Figure 2. Comparison of Tryptic Digest with Ascentis Express and Conventional Wide Pore, 5 μ m Column

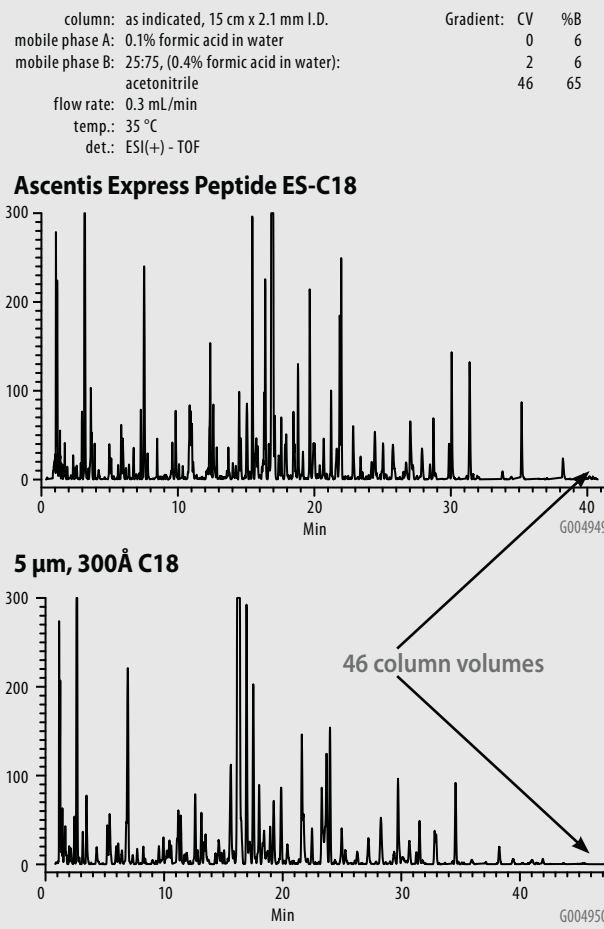


Table 2. Comparison of Peak Capacities

Column	t_g^*	w_{ave}	P_c
Ascentis Express Peptide ES-C18	41.3	0.1213	340
5 μ m 300Å C18	47.2	0.1951	242

*gradient time is scaled to the same number of column volumes. t_g and w are units of minutes.

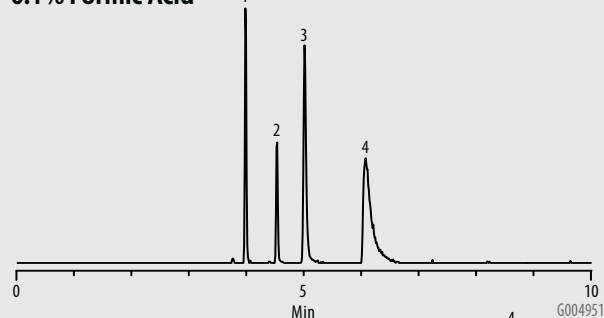
While 0.1% formic acid has become a de facto standard for peptide mapping by LC-MS, it is not without some concerns. It has been noted that peak efficiency and peak shape in the presence of 0.1% formic acid are not as good as with 0.1% TFA (2). This is particularly evident with basic peptides as shown in Figure 3. Also note the enhanced retention of peptides in the presence of TFA, presumably due to the greater hydrophobicity of TFA relative to formic acid.

Figure 3. Peak Shape Comparison of Basic Peptides with Different Ion Pair Reagents

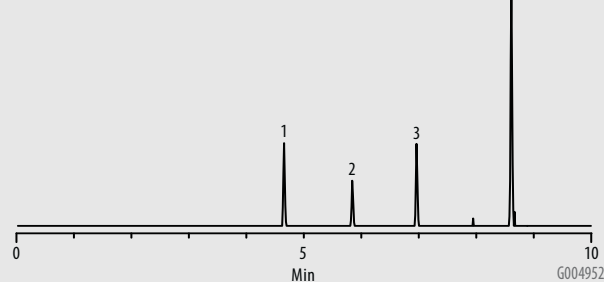
column: Ascentis Express Peptide ES-C18, 15 cm x 2.1 mm I.D. (53307-U)
 mobile phase A: 0.1% additive in water
 mobile phase B: 25:75, (0.4% additive in water):acetonitrile
 gradient: 15 – 44% B in 10.1 min
 flow rate: 0.3 mL/min
 temp.: 35 °C
 det.: ESI(+)-TOF

- | | |
|--------------------|---------------------------|
| 1. ac-GGGLGGAGGLKG | monoisotopic mass: 941.5 |
| 2. ac-KYGLGGAGGLKG | monoisotopic mass: 1118.6 |
| 3. ac-GGAVKALKGLKG | monoisotopic mass: 1139.7 |
| 4. ac-KYALKALKGLKG | monoisotopic mass: 1330.8 |

0.1% Formic Acid



0.1% TFA



McCalley (2) has best explained the poorer peak shape in the presence of formic acid, as reduced peak capacity of the retained analyte (peptides) due to ionic repulsion in the absence of adequate ion pairing, that would otherwise neutralize the analyte charge (2). This can be further understood in reference to Table 3.

Table 3. Ionization of Ion Pair Reagents

Reagent	pKa	pH of 0.1% Solution	% Ionized
TFA	0.3	2.0▼	98
Formic acid	3.7	2.6▼	7
Formic acid	3.7	3.5	36

▼ pH unadjusted

As a 0.1% aqueous solution, TFA is 98% ionized while that of formic acid is 7% ionized. Obviously then, there is far greater anion available to ion pair with basic moieties in the case of TFA than in the case of formic acid. This is certainly a plausible explanation consistent with McCalley's hypothesis. A further approach to testing this idea is to adjust the pH of the formic acid solution higher, such that greater formate anion is available for ion pairing. Adjusting the pH to 3.5 results in a 7-fold increase in the formate anion concentration. Indeed, this minor alteration in the mobile phase has dramatic impact on peak shape of basic peptides as seen in Figure 4.

The improved peak shape at pH 3.5 is best explained as a result of the increase in formate anion concentration, and not an effect of the ammonium cation, because the peak shape with a similar concentration of ammonium formate at pH 3, is not nearly as improved as at pH 3.5 (data not shown). Were the effect similar, it would argue for a more significant role of the ammonium cation in affecting poor peak shape, via mitigation of ion-exchange activity between the peptides and the silica surface.

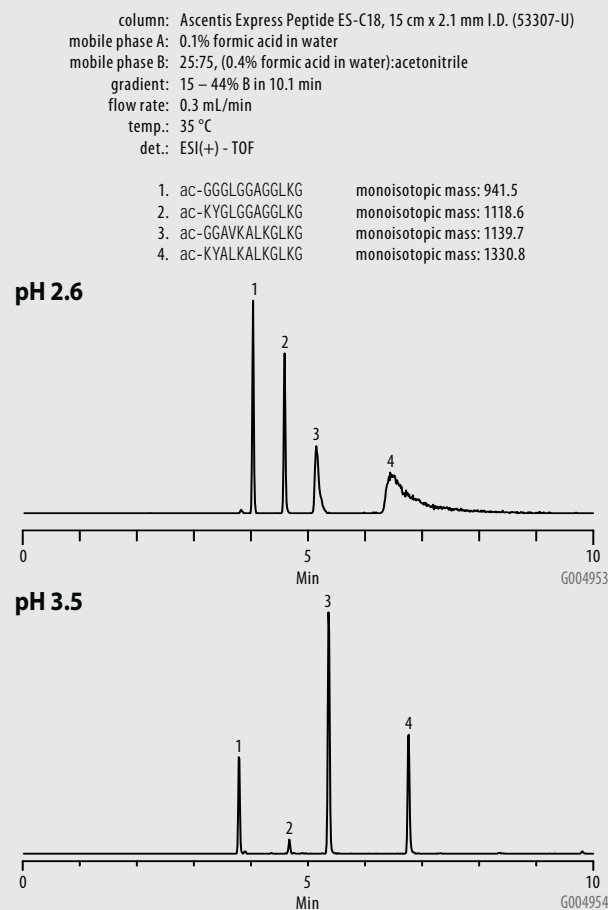
Ascentis Express Peptide ES-C18 is optimized for peptide separations up to 20,000 daltons

Ascentis Express Peptide ES-C18, then, is the latest addition to the Ascentis Express product line that can yield state-of-the-art performance for reversed-phase chromatography of polypeptides, be they complex proteomic mixtures or samples derived from peptide drug research and analysis.

* all concentrations, shown as percentages, are v/v

References

1. Apffel, A. et. al. 1995. Enhanced sensitivity for peptide mapping with electrospray liquid chromatography-mass spectrometry in the presence of signal suppression due to trifluoroacetic acid-containing mobile phases. *J. Chrom A* 712: 177-190.
2. McCalley, D.V. 2004. Effect of buffer on peak shape of peptides in reversed-phase high performance liquid chromatography. *J. Chrom A* 1038: 77-84.

Figure 4. Peak Shape Comparison of Basic Peptides at Different pH

+ Related Products

Ascentis Express Peptide ES-C18 Columns

I.D. (mm)	Length (cm)				
	3	5	7.5	10	15
2.1	53299-U	53301-U	53304-U	53306-U	53307-U
3.0	53308-U	53311-U	53312-U	53313-U	53314-U
4.6	53316-U	53318-U	53323-U	53324-U	53328-U

+ Related Information

For more information on the Ascentis Express Peptide ES-C18, request brochure T410043 (MII).

Ascentis ES Cyano – A New “Extra Stable” Cyano Phase Shown as a Confirmatory Column for US EPA Method 8330

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Introduction

Military installations and armament manufacturers have experienced closures in the last several years due to non-proliferation treaties and disarmament agreements. These closures have forced the military and environmental regulators to closely examine the explosive materials left behind in water, soil and sediment.

US EPA Method 8330

The United States Environmental Protection Agency (US EPA) developed Method 8330 (1) for the trace analysis of

explosive residues by HPLC with ultraviolet detection (LC-UV). This method is used to identify and determine levels of the explosives shown in Figure 1, and recommends the use of two columns for confirmatory analysis. For instrument calibration, the fourteen analytes are divided in two mixes, termed Mix A and Mix B, with 5-point calibration curves required for both. After calibration, the instrument software combines the retention time and response factor data from both mixes into a single file. The results of the subsequent analyses of sample extracts are then compared to this combined calibration file.

Primary (C18) and Confirmatory (Cyano) Columns

Figure 2 shows the analysis of these two mixes on an Ascentis C18 column. Using identical instrument conditions, both mixes were also analyzed on an Ascentis ES cyano column (chromatograms shown in Figure 3). These two columns contain different ligands with their own analyte interactions, resulting in a different selectivity for each column. This differing selectivity is necessary for confirmatory analysis.

The choice of the Ascentis ES Cyano column to provide alternate selectivity for the explosives is based on π - π and/or dipole-dipole interaction on a cyano phase (2). The π acidic solutes, such as the nitro-substituted aromatic explosives have been shown to have preferential retention for a cyano phase. (2,3,4). In addition, the method mobile phase of 50:50 water:methanol has been shown to enhance retention on a cyano phase (5).

Figure 1. Explosive Analytes

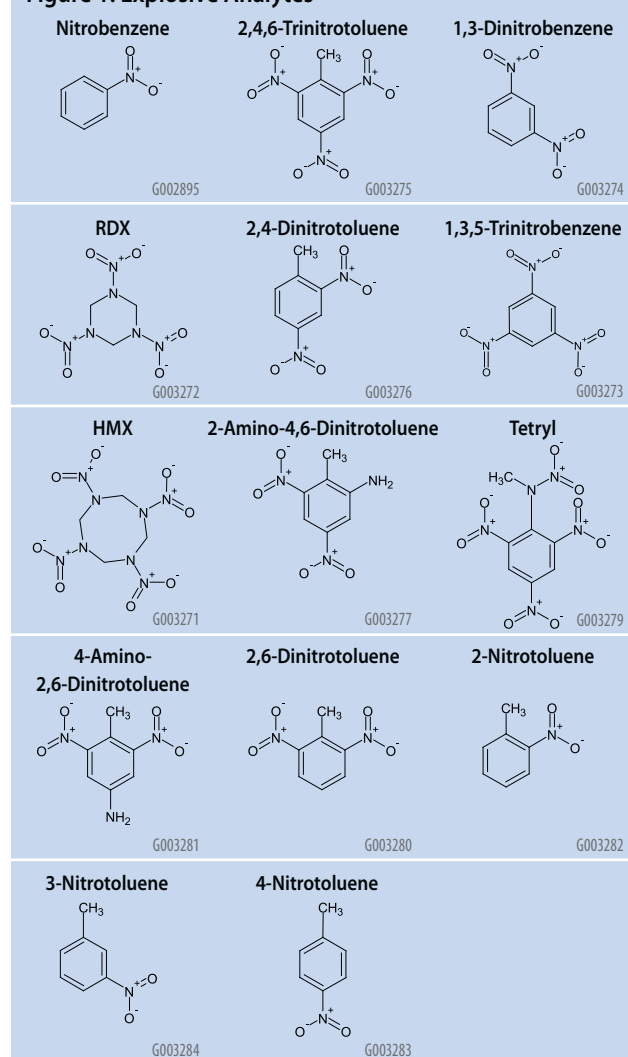
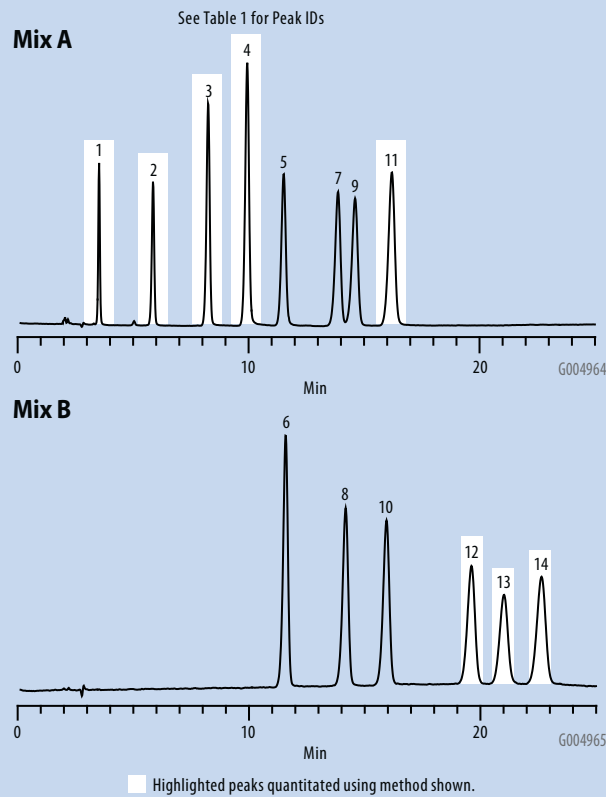


Table 1. Peak IDs, Retention Times, and Quantitation Strategy

Peak ID	Analyte	C18 (min.)	Cyano (min.)	Quantitate Using
1	HMX	3.54	15.85	C18
2	RDX	5.88	14.67	C18
3	1,3,5-Trinitrobenzene	8.26	10.55	C18
4	1,3-Dinitrobenzene	9.66	10.55	C18
5	Nitrobenzene	11.54	8.92	Cyano
6	Tetryl	11.59	23.81	Cyano
7	2,4,6-Trinitrotoluene	13.89	15.24	Cyano
8	4-Amino-2,6-dinitrotoluene	14.19	17.24	Cyano
9	2-Amino-4,6-dinitrotoluene	14.63	19.09	Cyano
10	2,6-Dinitrotoluene	15.96	14.01	Cyano
11	2,4-Dinitrotoluene	16.23	14.67	C18
12	2-Nitrotoluene	19.62	11.88	C18
13	4-Nitrotoluene	21.04	12.32	C18
14	3-Nitrotoluene	22.66	12.77	C18

Figure 2. Analysis of Explosives on Ascentis C18 using EPA Method 8330

column: Ascentis C18, 25 cm x 4.6 mm I.D., 5 μ m particles (581325-U)
 mobile phase: 50:50 water:methanol
 flow rate: 1 mL/min.
 temp.: 35 $^{\circ}$ C
 det.: UV, 254 nm
 injection: 2 μ L
 sample: each analyte at 100 μ g/mL in 50:50 water:methanol

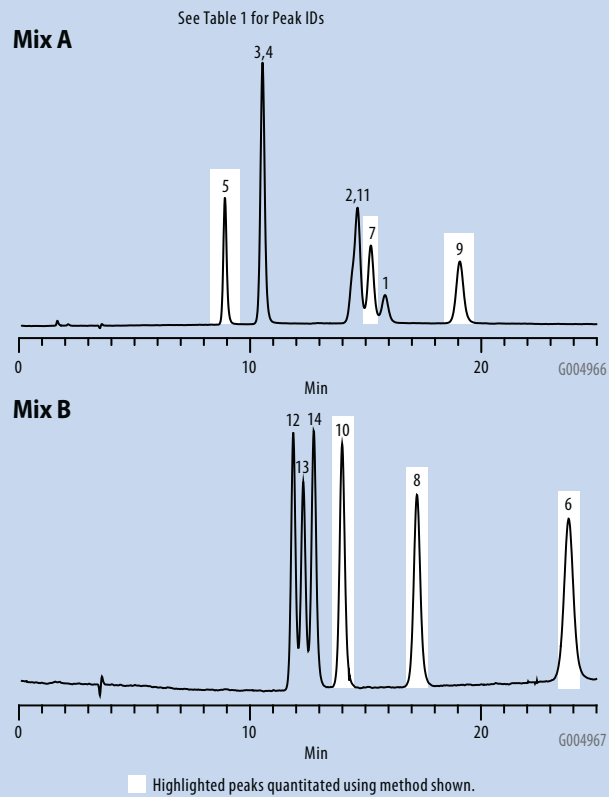


Analyte Identification and Quantitation

The results obtained from the primary (C18) column can be used for eight of the analytes, as indicated in Table 1. A number of co-elutions and partial co-elutions exist on the primary (C18) column, so it can be seen that an alternative selectivity column would be necessary to confirm the identity of each peak. As an illustration, Table 1 shows that the analyte pair nitrobenzene and tetryl will co-elute on the Ascentis C18, with retention times of 11.54 and 11.59 minutes, respectively. Retention times on the Ascentis ES Cyano are also listed in Table 1. Note the large differences in retention times for this analyte pair, 8.92 and 23.81 minutes, respectively. Another example, also shown in Table 1, are the partially co-eluting analyte pair 4-amino-2,6-dinitrotoluene and 2-amino-4,6-dinitrotoluene. When these analytes are run on the Ascentis ES Cyano phase, their retention times are nearly two minutes apart. Table 1 also shows the column that would be used when this column pair is used for quantitation.

Figure 3. Analysis of Explosives on Ascentis ES Cyano using EPA Method 8330

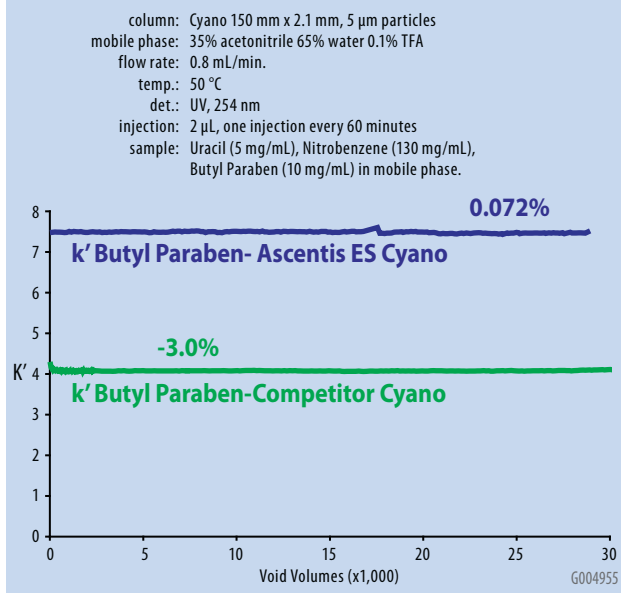
column: Ascentis ES Cyano, 25 cm x 4.6 mm I.D., 5 μ m particles (577307-U)
 mobile phase: 50:50 water:methanol
 flow rate: 1 mL/min.
 temp.: 35 $^{\circ}$ C
 det.: UV, 254 nm
 injection: 2 μ L
 sample: each analyte at 100 μ g/mL in 50:50 water:methanol



Ascentis ES Cyano Column Stability

An important issue that has arisen in the past is the stability of a cyano phase. Figure 4 shows results of a stability test run under rigorous conditions. At 50 $^{\circ}$ C, with a mobile phase containing of 0.1% TFA in 65% water and 35% acetonitrile, a test mixture of uracil, nitrobenzene and butyl paraben was injected every hour for several days. The leading competitive cyano column was included in this test as a control. The Y-axis shows the number of column void volumes pumped through the column during the test, with the X-axis showing retention (k'). Note the 2.2% loss in k' for the nitrobenzene on the competitor cyano phase versus the 0.78% loss on the Ascentis ES Cyano phase. Recall that the nitrobenzene is also one of the explosive compounds included in EPA Method 8330, discussed above. Additionally, butyl paraben shows a 3% loss of k' on the competitor Cyano, while a negligible loss of 0.072% is seen on the Supelco Cyano phase. This data shows the stability of this

(continued on page 8)

Figure 4. Stability of Ascentis ES Cyano vs. Competitor

(continued from page 7)

new phase under extreme conditions of pH and temperature in LC analysis.

Conclusion

US EPA Method 8330 is used to determine explosive residues in water, soil and sediment. It specifies the use of

two columns with alternative selectivity to confirm results. The data presented here shows that the Ascentis C18 and Ascentis ES Cyano are an ideal column pair for this analysis. Stability of the Ascentis ES Cyano is favorable when compared to the leading competitive cyano phase.

References

1. US EPA Method 8330A, "Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)" Revision 1 (February 2007), obtained from the www.epa.gov web site.
2. K. Croes et al. J. Chromatogr. A 1098 (2005) 123.
3. A. Tchaplá, S. Heron, E. Lesellier, J. Chromatogr. A 656 (1993) 81.
4. S. Heron, A. Tchaplá, J. Chromatogr. A 725 (1996) 205.
5. J. Horak, N.M. Maier, W. Lindner, J. Chromatogr. A 1045 (2004) 43.

+ Featured Products

Description	Cat. No.
Ascentis C18, 25 cm x 4.6 mm I.D., 5 μ m particles	581325-U
Ascentis ES Cyano, 25 cm x 4.6 mm I.D., 5 μ m particles	577307-U
US EPA Method 8330 Mix A	47283
8 analytes, each at 100 μ g/mL in acetonitrile, 1 mL ampul	
2-Amino-4,6-dinitrotoluene	Nitrobenzene
1,3-Dinitrobenzene	RDX
2,4-Dinitrotoluene	1,3,5-Trinitrobenzene
HMX	2,4,6-Trinitrotoluene
US EPA Method 8330 Mix B	47284
6 analytes, each at 100 μ g/mL in acetonitrile, 1 mL ampul	
4-Amino-2,6-dinitrotoluene	3-Nitrotoluene
2,6-Dinitrotoluene	4-Nitrotoluene
2-Nitrotoluene	Tetryl

Newly-Expanded Chiral Screening Services

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Supelco is distinct in offering chiral separation products, chiral method development and chiral purification services that cover a wide range of analyte types, separation modes, solvent systems, and assay requirements. This short article provides some background on the benefits of using chiral services and the uniqueness of our offering.

Method development for chiral separations is a challenging task. Even the veteran chiral separation scientist cannot accurately predict what stationary phase/mobile phase combination will be successful. This difficulty stems from the highly complex and interdependent retention mechanisms that manifest themselves into retention and enantioselectivity.

Compounds with similar structures, especially in the region of a chiral center, often provide enantiomeric selectivity under similar conditions. However, structural changes, even as small as the addition of a methyl group, often lead to the need for further method development.

Column screening at the inception of method development not only promises to provide a method that works, but also may provide combinations of stationary and mobile phases that work better for a given analysis. For example, if an analytical method for release testing is desired, reversed-phase conditions may be better than normal-phase. As another example, methods intended for trace analysis or preparative purposes may be improved by reversing the elution order of stereoisomers. Chiral screening provides, in a relatively short period of time, a more complete picture of possible solutions. The data acquired can form the basis of many development efforts and provide a complete record, should unforeseen events arise, such as new coeluting impurities.

Sigma-Aldrich/Supelco now provides a comprehensive and unparalleled screening package intended to assist customers in their method development efforts, no matter what the needs of the assay are. Analyte screening in both high-performance liquid chromatography (HPLC) and gas chromatography (GC) is available.

Table 1. HPLC Chiral Screening Conditions

Column	RP1	RP2	PIM	NP1	NP2	POM
Astec CHIROBIOTIC® TAG	X	X	X			
Astec CHIROBIOTIC V2	X	X	X			
Astec CHIROBIOTIC T	X	X	X			
Astec CYCLOBOND® I 2000	X	X				
Astec CYCLOBOND I 2000 HP-RSP	X	X				
Astec CYCLOBOND I 2000 DMP	X	X				
Kromasil® CelluCoat				X	X	X
Kromasil AmyCoat				X	X	X
Kromasil TBB				X	X	X
Kromasil DMB				X	X	X
Astec P-CAP™ DP				X	X	X
Astec P-CAP				X	X	X

RP1 = 70:30, 20 mM ammonium acetate (pH 4.0):acetonitrile
 RP2 = 50:50, 20 mM ammonium acetate (pH 4.0):methanol
 PIM = 100:0.1:0.1, methanol:acetic acid: triethylamine
 NP1 = 80:20, heptane: isopropanol (with 0.1% TEA and 0.1% TFA)
 NP2 = 50:25:25, heptane: isopropanol (with 0.1% TEA and 0.1% TFA):MTBE
 POM = 95:5, acetonitrile:isopropanol (with 0.1% TEA and 0.1% TFA)

Our semiautomated HPLC chiral screening protocol includes a combination of 12 different stationary phases and 6 different elution systems. Details of the combinations are presented in Table 1. The highly-effective protocol includes normal phase, polar organic, polar ionic, and reversed-phase modes of elution. Stationary phases range from cellulosic/amylosic chemistries to the more polar CHIROBIOTIC phases, each exhibiting unique separation qualities for a wide range of analyte structures. Detection, by default, is ultraviolet absorption. Protocols modified for evaporative light scattering, fluorescence, refractive index and mass spectrometry are also available.

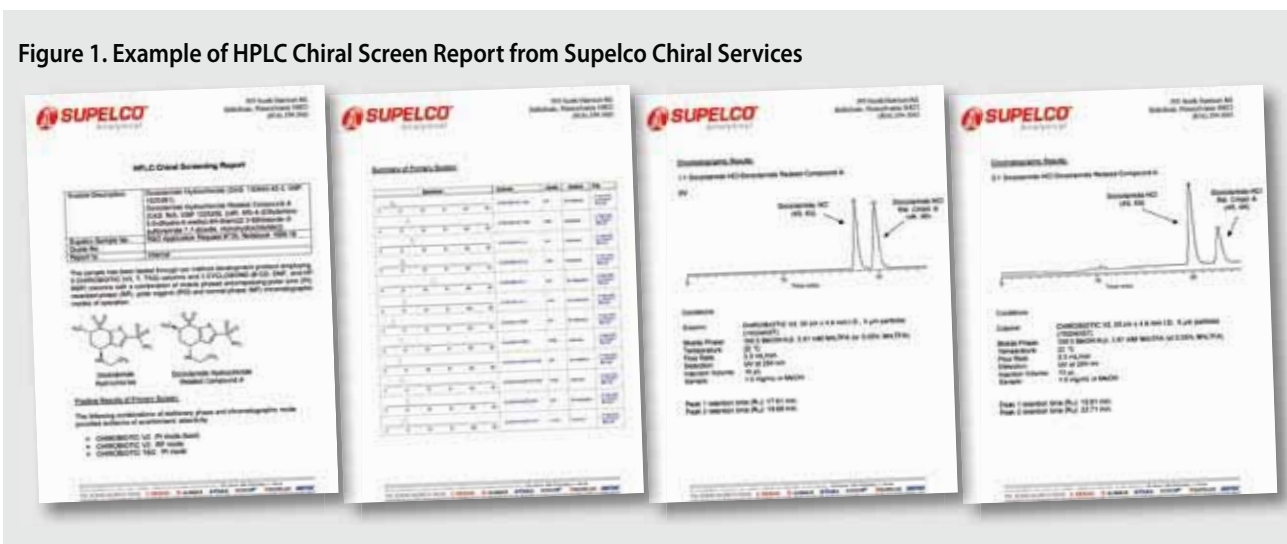
The output from the comprehensive screening study is tabulated and evaluated for suitability based on the input from the customer. The chosen set of conditions deemed most appropriate is then confirmed on a second system utilizing a second column and mobile phase preparation. Limited variable optimization may also be applied. A report is then generated including all acquired data and confirmatory results, as well as recommendations for further method development. An example of such a report is provided in Figure 1.

The GC protocol involves the manual exploration of not less than three selected chiral columns and employs a thermal gradient. Once a stationary phase chemistry has been identified, an isothermal system is generated to further optimize the separation. Mass spectrometric detection is used to confirm derivatization if needed. As with HPLC, a full report is generated including acquired data, spectral analysis (if needed) and recommendations.

Stationary phase screening is a necessary and integral part of chiral method development. Data provided by the screening protocols provide a comprehensive picture of the possible solutions. Methods intended for analytical analysis as well as preparative efforts can be derived from this screening data. Sigma-Aldrich/Supelco offers comprehensive HPLC and GC screening services. Costing less than the price of a single analytical column, employment of the program makes sound financial sense.

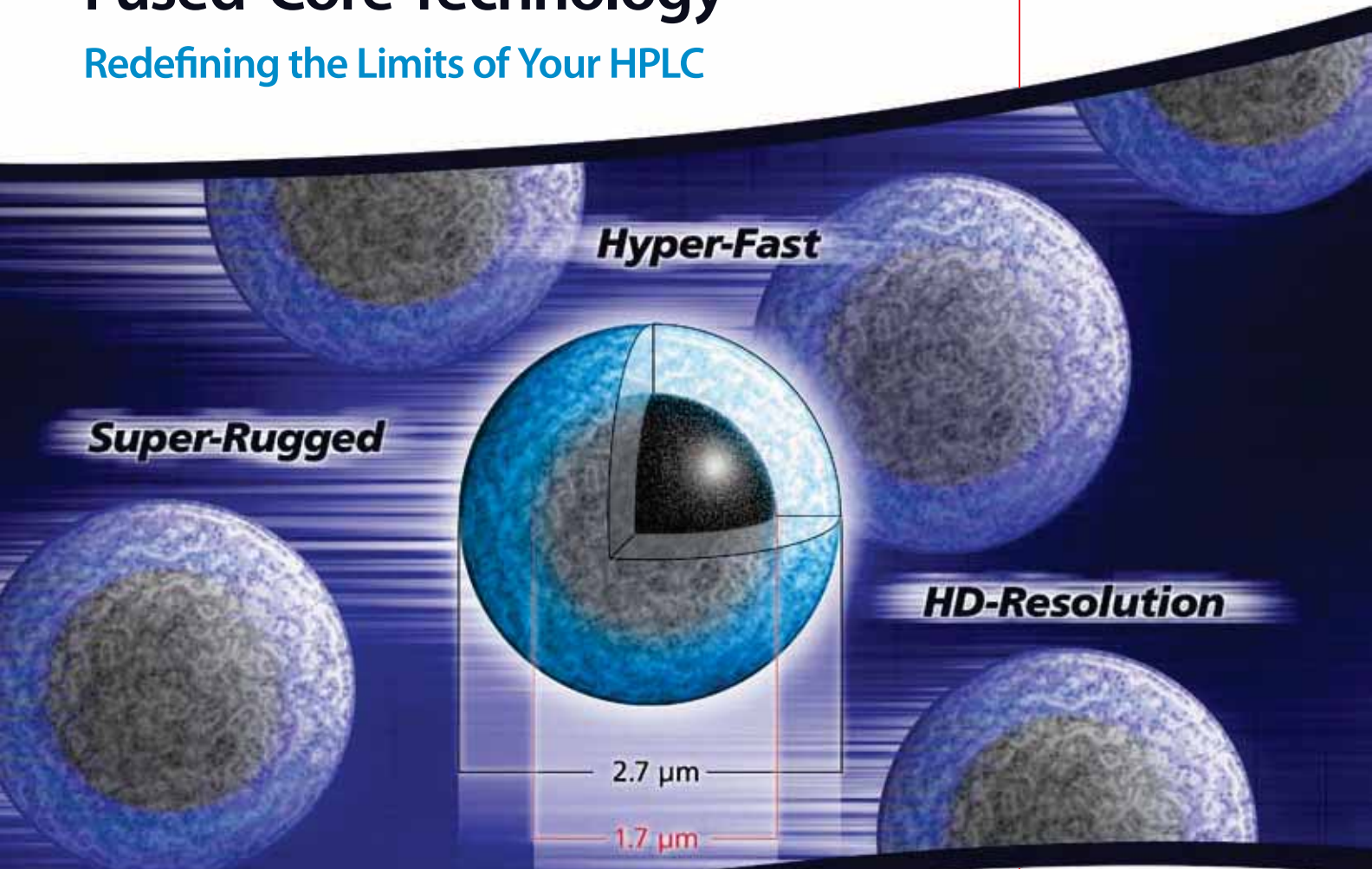
View our complete offering of chiral chemistry, chiral chromatography, and chiral services at our website: sigma-aldrich.com/chiral

Figure 1. Example of HPLC Chiral Screen Report from Supelco Chiral Services



Ascentis Express Fused-Core Technology

Redefining the Limits of Your HPLC



Based on Fused-Core particle technology, Supelco's Ascentis Express columns provide a breakthrough in HPLC column performance.

- Double the efficiencies of conventional 3 µm particles
- Equal efficiencies of sub-2 µm columns at half of the backpressure
- Rugged design capable of high pressure operation

Now available — Ascentis Express Peptide ES C18 for peptides and small proteins

For additional information, call our technical experts at 800-359-3041/814-359-3041 or visit us on the web: sigma-aldrich.com/express

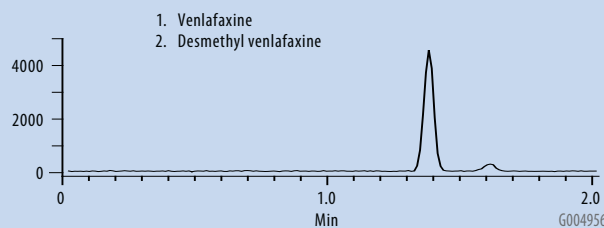
Minimizing Phospholipid Matrix Effects in HILIC LC-MS using HybridSPE-Precipitation Small Volume Plates

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This article discusses phospholipid matrix effects in HILIC chromatography. HILIC chromatographic separations have become widely accepted in LC-MS applications due to increased retention of polar compounds and enhanced ionization of analytes. Though the retention mechanism of phospholipids in reversed-phase chromatography is different than in HILIC chromatography, phospholipid buildup still occurs in HILIC separations, causing poor analyte response. In this study, rat plasma samples spiked with venlafaxine and metabolite were processed using standard protein precipitation methods and compared with samples prepared using the HybridSPE-PPT Small Volume 96-well plate. Spiked sample concentrations ranged from 100 ng/mL to 1200 ng/mL in plasma. Analyte response was compared between the two sample prep techniques along with total amount of extracted phospholipid matrix. The phospholipid matrix was measured by scanning mass range 400-950 m/z.

Figure 1. HILIC Separation of Venlafaxine and Metabolite



Samples prepared using the HybridSPE Small Volume plate consisted of applying 20 μ L of plasma to the plate, followed by 60 μ L of 1% formic acid in acetonitrile. The plate was then vortexed for one minute, then placed on a vacuum manifold with 10" Hg vacuum for two minutes. The filtrate was collected and analyzed directly using an Ascentis Express HILIC column. Samples prepared using the standard protein precipitation method consisted of adding 100 μ L of plasma to a centrifuge vial, followed by 300 μ L of 1% formic acid in acetonitrile. Samples were then vortexed for one minute and centrifuged for two minutes at 15000 rpm. The supernatant was collected and analyzed directly.

Using a standard protein precipitation technique, significant phospholipid matrix interference occurred directly in the elution region of venlafaxine and metabolite. As depicted in Figure 2, a continued buildup of phospholipids occurred throughout the study. The blue trace represents phospholipid interference from the initial injection;

Figure 2. TIC Phospholipid Monitoring of Standard Protein Precipitation, initial injection (blue), 50th injection (red)

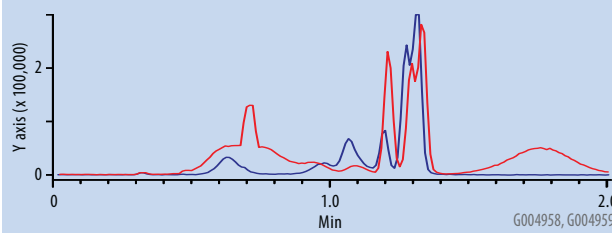
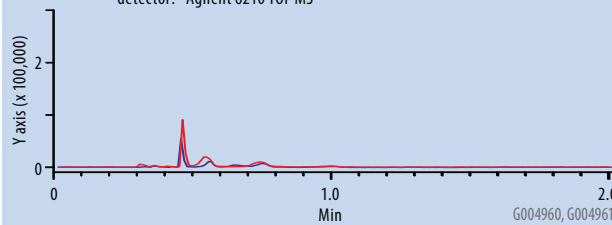


Figure 3. TIC Phospholipid Monitoring using HybridSPE Small Volume, initial injection (blue), 50th injection (red)

column: Ascentis Express HILIC, 10 cm x 2.1 mm I.D., 2.7 μ m (53939-U)
mobile phase: 5 mM ammonium formate in 90:10 acetonitrile:water, pH 6.87
flow rate: 0.6 mL/min.
temp.: 35 $^{\circ}$ C
instrument: Agilent 1200SL Rapid Resolution
detector: Agilent 6210 TOF MS



the trace in red represents the amount of phospholipid interference at the 50th injection. The continual increase in matrix buildup resulted in significant analyte suppression, even at the highest concentration level. When samples were prepared using the HybridSPE Small Volume technique, the phospholipid matrix interference was depleted, resulting in no matrix interference. As shown in Figure 3, no matrix interference is observed from the initial injection in blue to the final injection in red.

The HybridSPE Small Volume technique is a simple and effective method for sample preparation of small volume plasma samples, resulting in depletion of phospholipid matrix interference, eliminating the concern for matrix buildup in both reversed phase and HILIC separations.

+ Featured Products

Description	Cat. No.
HybridSPE-PPT Small Volume Plate, 15 mg/well	52794-U
Ascentis Express HILIC, 10 cm x 2.1 mm I.D., 2.7 μ m	53939-U

ordering: 800-247-6628 (US only) / 814-359-3441 technical service: 800-359-3041 (US and Canada only) / 814-359-3041

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Analysis of Organophosphorous Pesticides in Produce by Solid/Liquid Extraction and Dual-Layer Amino-Silica/Carbon SPE Cleanup and GC-NPD

Olga Shimelis, Katherine Stenerson, Michael Ye,
Carmen T. Sanatasania, Michael Monko
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Introduction

Organophosphorous pesticide use in agriculture is widespread due to the fact that they are more amenable to environmental degradation in comparison to organochlorine or organonitrogen compounds. There is a large number of



pesticides in the organophosphorous group, and because of their potential health effects, they are of particular concern on produce imported from areas in which they are commonly used. In this work we analyzed the representative organophosphorous pesticides from cabbage, green onions, apples and

mushrooms. The extraction was performed using a custom-made mixture of salts and liquid-liquid partitioning, with clean-up using dual-layer Supelclean™ ENVI-Carb™-II/PSA Solid Phase Extraction (SPE) tubes.

Sample Preparation

A 10 g homogenized food sample was spiked at 10 ng/g with pesticides, and 10 mL of acetonitrile was added. The sample was mixed with custom-made extraction salts – 4 g dry magnesium sulfate and 1 g sodium chloride. The sample was centrifuged and the supernatant was mixed with 1 g of dry magnesium sulfate. 5 mL of the resulting sample was evaporated down to 1mL for SPE loading. A Supelclean ENVI-Carb-II/PSA 500 mg/500 mg 6 mL tube was pre-conditioned with 5 mL acetonitrile:toluene (65:35). The sample was

Did you know...?

The poster presented at AOAC 2009 titled *Analysis of Organophosphorous Pesticides in Produce by Dispersive Solid Phase Extraction, Dual-Layer Amino-Silica/Carbon SPE Cleanup and GC-NPD* contains many details not included in this article (including full chromatograms and a much longer list of organophosphorous pesticide recoveries). An electronic file of this poster can be obtained by contacting Supelco Technical Service at 800-359-3041 (US and Canada only) 814-359-3041, or at techservice@sial.com and requesting T409161.

Table 1. Percent Recovery and (% RSD) of Representative Organophosphorus Pesticides Spiked into Vegetables at 10 ng/g (n=3)

Pesticide	Cabbage	Onion	Mushroom	Apple
Methamidophos	76 (10%)	70 (45%)	70 (5%)	58(3%)
Dichlorvos	masked	57 (8%)	89 (7%)	76 (6%)
Ethoprophos	63 (9%)	66 (11%)	77 (9%)	75 (7%)
Methyl parathion	65 (2%)	66 (11%)	81 (7%)	77 (6%)
Profenfos	72 (3%)	77 (11%)	92 (7%)	82 (6%)
Coumaphos	78 (8%)	69 (8%)	94 (12%)	88 (6%)

loaded and eluted with 10 mL of acetonitrile:toluene (65:35). The elution fraction was evaporated to 0.5 mL and reconstituted to 1mL with ethyl acetate.

Conclusions

Most of the pesticides tested showed good recovery and reproducibility. Recoveries for some pesticides were still matrix-dependent, such as dichlorvos from cabbage (Table 1). GC analysis using an SLB™-5ms column was optimized by using nitrogen phosphorus detection (NPD). Although NPD is highly sensitive for nitrogen containing compounds (such as organophosphorus pesticides), the high level of sulfur-containing compounds found in cabbage and onions still contributed to matrix interference as observed in Table 1. Custom-made mixes of extraction salts saved total sample preparation time.

+ Featured Products

Description	Cat. No.
Supelclean ENVI-Carb-II/PSA Tube, 500 mg/500 mg, 6 mL, pk. of 30	54067-U
SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm	28471-U

+ Related Products

Description	Cat. No.
Methamidophos	33395
Dichlorvos	45441
Ethoprophos	45306
Methyl Parathion	PS94
Profenfos	45632
Coumaphos	45403

+ Related Information

If you are interested in further information or would like to request a quotation on a custom or existing dispersive SPE product, please visit our website: sigma-aldrich.com/quechers.

Performance Comparison of TDS³ Storage Containers to Swagelok Fittings and Glass Storage Containers

Kristen Schultz and Jamie Brown

Kristen.schultz@sial.com

Introduction

The typical analytical process for air sampling using thermal desorption tubes almost always involves shipping and storing the sampling tube before and after sampling prior to analysis. The most common approach to preventing contamination of the tube during shipment and storage has been to attach Swagelok end-cap fittings, using PTFE ferrules to both ends of the tubes before and after sampling. Another common technique is to place the sampling tube in a glass vial-like container, constructed to seal at one end with a Teflon[®]-faced screw cap.

The TDS³ (Thermal Desorption Storage & Sampling System) offers advantages over both the Swagelok end-cap fittings and glass storage containers because it is designed to eliminate internal dead volume, minimize the area of migration of the sample from the adsorbent during the storage period, and eliminate breakage risks when shipping and handling in contrast to glass storage containers bearing this same risk. The TDS³ storage container holds the tube in its hard polycarbonate shell and seals with inert end-caps fitted with Teflon-faced silicone septa that are easily replaced. This eliminates the need for extensive cleaning or thermal conditioning of the device before it can be used for storing another tube.

Experimental

The performance of the TDS³ storage container was measured by its effectiveness for storing a collected sample relative to Swagelok end-cap fittings and glass storage containers.

A mix of twelve analytes were spiked onto twelve Carbotrap[™] 300 thermal desorption tubes (6 mm O.D x 4 mm ID x 11.5 cm L), containing three carbonaceous adsorbents: Carbotrap C, Carbotrap B, and Carbosieve[™] S-III. The sampling tubes were spiked with 40 ng of each analyte in 0.2 µL of methanol, using flash vaporization and 0.5 liters of inert nitrogen (50 mL/min for 10 min) to transfer the vaporized analytes onto the sampling tube.

Figure 1. TDS³ Container with Carbotrap 300 Glass-Fritted TD Tube



The clear body of the TDS³ is shorter than the actual tube (as shown), so the septa seals on the end of the thermal desorption tube, creating an air-tight seal.

The twelve tubes were assigned to three sets, each stored in a different storage device. Tubes in Set One were stored in TDS³ storage containers, Set Two were fitted with brass Swagelok nut and end-cap fittings, using PTFE ferrules; and Set Three were stored in a threaded glass vial-type container which seals with a Teflon screw cap at one end.

After spiking, the tubes were quickly sealed and placed in a paint can (all tubes in the same can) containing a small amount of activated charcoal and placed in a laboratory freezer for 14 days at -24 °C. After 14 days, the samples were removed from the paint can and thermally desorbed to a gas chromatograph.

Results

Percent recoveries in Table 1 were calculated by comparing peak areas for the desorbed analytes to those for calibration standards spiked onto the Carbotrap 300 tubes in the same manner on the day of analysis. Values shown are means for the three samples. Area counts for the first six

(continued on page 14)

Analyte	Set One	Set Two	Set Three
	TDS ³ Storage Container	Swagelok Fittings with PTFE Ferrules	Glass Storage Container
Chloroform	97.7 ± 6.8	97.9 ± 2.3	99.2 ± 3.5
1,1,1-Trichloroethane	102.4 ± 1.7	102.6 ± 1.5	104.4 ± 1.4
Carbon tetrachloride	98.7 ± 2.8	95.8 ± 1.3	98.8 ± 2.6
1,2-Dichloroethane	98.6 ± 3.0	95.8 ± 1.7	98.6 ± 1.0
Trichloroethylene	100.7 ± 2.8	96.7 ± 0.7	105.0 ± 2.0
1,2-Dichloropropane	99.3 ± 2.5	97.5 ± 1.4	102.9 ± 2.7
1,3-Dichloropropane	101.3 ± 6.0	94.4 ± 9.2	87.8 ± 9.4
Tetrachloroethylene	99.5 ± 5.3	101.7 ± 4.4	93.8 ± 6.6
Ethylene dibromide	114.2 ± 6.2	97.1 ± 18.3	76.2 ± 8.7
Chlorobenzene	95.4 ± 4.4	95.9 ± 6.9	95.0 ± 5.7
Bromoform	107.7 ± 11.3	98.9 ± 5.5	91.7 ± 10.4
Bromobenzene	96.0 ± 4.8	95.4 ± 8.4	91.1 ± 6.8

(continued from page 13)

listed compounds were normalized to an internal standard, bromodichloromethane; the last six compounds were normalized to 1,3-dichlorobenzene.

Conclusion

The results in Table 1 demonstrate that the TDS³ storage containers are equivalent to both Swagelok end-cap fittings and glass storage containers in terms of sample stability during storage.

+ Featured Products

Description	Pk.	Cat. No.
TDS³ Storage Container by Instrument Manufacturer/Model		
Supelco, DANI, Markes, PerkinElmer, Shimadzu	1	25097-U
CDS/Dynatherm Standard Tubes	1	25096-U
Chrompack TD Tubes	1	25098-U
Gerstel TDS/TDS2/TDSA Tubes	1	25095-U
Gerstel 60 mm Tubes	1	28307-U
Teledyne/Tekmar AEROTrap 6000 Tubes	1	25099-U
Envirochem, 810 Tubes	1	25100-U
TDS³ Storage Container Accessories		
Sampling caps w/washers for ¼ in. OD Tubes	10	25069
Replacement Septa for all TDS ³ containers	50	25073
Male Luer Plug	12	504351
Female Luer Cap	12	57098
Tubing Adapter for Use With:		
1/8 in. tubing to male luer	20	21016
3/16 in. tubing to male luer	20	23364
¼ in. tubing to male luer	10	24856
Tubing Coupler		
For use with male to male luer	20	25064-U

Did you know...?

In addition to its ability to maintain sample integrity, the TDS³ storage container offers the user versatility during the sampling process. It can be converted to a sampling device by installing optional sampling caps that allow the user to easily connect the tube in the TDS³ to a sampling pump. An optional tube holder is also available.

Figure 2. Carbotrap 300 TD Tubes with Escort Elf Pump and Twin Port Sampler



E001122

+ Related Products

Description	Qty.	Cat. No.
Equipment		
Escort Elf Sampling Pump	1	28160-U
Gemini Twin Port Sampler	1	28118-U
12 Volt Battery Charger	1	28155-U
110 Volt Battery Charger	1	28158-U
240 Volt Battery Charger	1	28159-U
Chemical Standards		
Chloroform	5 mL	02487
1,1,1-Trichloroethane	1 mL, 5 mL	02669
Carbon tetrachloride	1 mL, 5 mL	02671
1,2-Dichloroethane	1 mL, 5 mL	02562
Trichloroethylene	5 mL	46267
1,2-Dichloropropane	1 mL	02577
1,3-Dichloropropane	250 mg	45439
Tetrachloroethylene	1 mL, 5 mL	02666
Ethylene dibromide	1 g	31040
Chlorobenzene	5 mL, 25 mL	08650
Bromoform	1 g	36972
Bromobenzene	500 mg	442495



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Particle Size, Pore Size	Pkg. Size	Cat. No.
40-63 μm , 60Å	500 g, 1 Kg, 2.5 Kg, 5 Kg, 25 Kg	60737
35-70 μm , 60Å	25 g, 1 Kg, 25 Kg	60738
63-200 μm , 60Å	1 Kg, 6x1 Kg, 25 Kg	60741

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P000616

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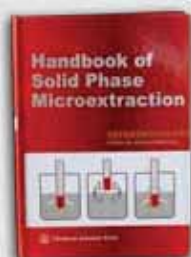
Sigma-Aldrich also offers a wide range of low pressure liquid chromatography (LPLC) hardware, separations media and accessories for flash chromatography and routine purification challenges including:

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Sample Handling

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Gas Cylinder Pressure Regulators

New Features and Better Performance from Airgas®

Robert F. Wallace
bob.wallace@sial.com

Introduction

The importance of gas purity to a successful gas chromatography (GC) analyses has been widely understood by chromatographers for years. Because gas purifiers play such a large part in the purification process, the role of the gas regulator is often overlooked. The regulator is often the first component of a gas delivery system and the first opportunity

Figure 1. Single Stage (above) and Two-Stage Regulators (below)



E001124



E001123

for contaminants to enter the system. Additionally, the design of the regulator must ensure contaminants cannot hide inside the body and emit at an unpredictable time. We have updated our line of gas cylinder pressure regulators with new models from Airgas, a recognized worldwide leader in gas delivery products. All are smaller/lighter models that include inlet check valves, a feature that significantly increases performance.

A Bar Stock Regulator or a Forged Body Regulator?

There are two basic regulator designs, forged body and bar stock. Forged body regulators have a rough internal surface with a larger internal volume. Bar stock regulators, preferred for most chromatographic applications, are machined with a smoother internal surface and lower internal volume.

Our bar stock regulators, with smooth internal cavities, provide a non-pitted surface, allowing more efficient and direct gas flow. This design eliminates the large cavities and pockets associated with forged body regulators and thus contain minimal internal surfaces to which contaminants can cling. The small internal volume (5% of the internal volume of a forged body regulator) provides a more direct gas flow path through the regulator. As a result, all areas come in contact with the gas stream, so any contaminant that enters the regulator is quickly swept out of the body.

Single Stage and Two-Stage

Contrary to widespread belief, a gas pressure regulator is not a flow control device. The primary function of the pressure regulator is to reduce the high gas pressure in a cylinder or delivery line to a level that can be used by an instrument or process. A single-stage regulator (Figure 2) reduces the pressure to an intermediate pressure that will be further reduced by a secondary regulator (either an in-line regulator or regulator built into the instrument). Two-stage cylinder regulators (Figure 3) reduce the high pressure from the cylinder to a range that can be accepted by the instrument. The two-stage regulator functions as two, single-stage regulators in series. The first stage reduces the inlet pressure to an internally set intermediate pressure, typically 300-500 psig. The second stage, adjusted by turning the control knob, reduces the immediate pressure to the desired delivery pressure.

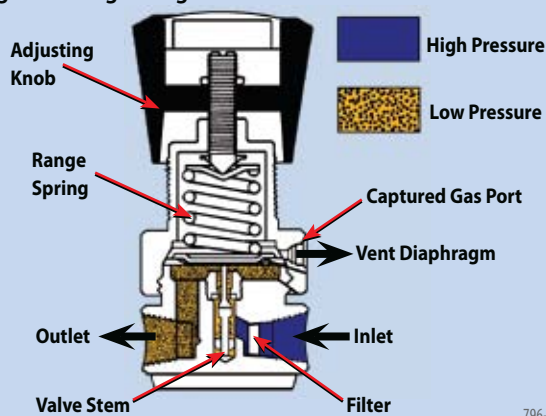
Which do I Choose?

Which regulator will work best for my application? The following are suggestions based on the need inside the laboratory. The Supelco high purity (HP) and high purity plus (HP+) gas cylinder regulators use a stainless steel diaphragm with a captive PTFE seal. Under most GC conditions, with the exception of the most sensitive applications, this diaphragm will ensure reliable and exceptional gas delivery. For more sensitive applications an ultra high purity (UHP) regulator is recommended. These regulators have a stainless steel diaphragm with a metal-to-metal rather than PTFE seal providing a better seal that will not allow contaminants to diffuse into the internal chamber.

New Regulator Features

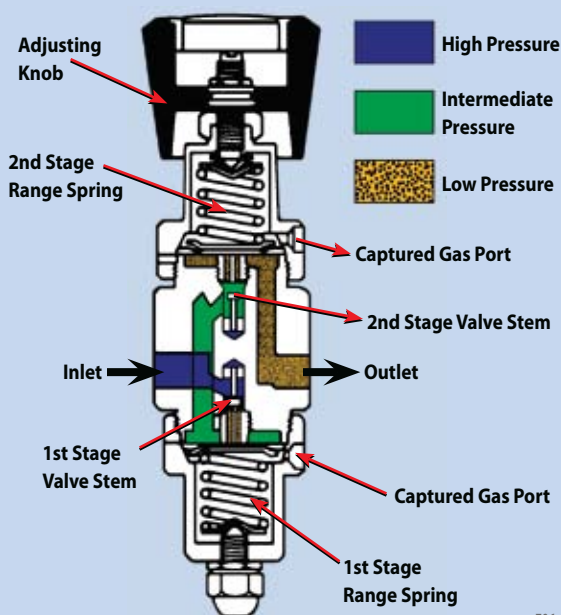
Gas cylinder regulators from Supelco now feature a check valve on the inlet fitting that keeps air from entering the regulator body during cylinder change-out, thus eliminating the need for a long purge time following the installation of a new gas cylinder. At the body outlet of the regulator, a shut-off valve keeps air from entering the system during change-out of the downstream gas purification/management components. There are two types of shut-off valves to choose from; a needle shut-off valve on HP models allows for flow control, whereas a diaphragm shut-off valve in HP+ and UHP models has higher leak integrity. The bonnet of the HP and HP+ are made of nickel-plated zinc, giving the regulator a cosmetically better look than nickel-plated brass. The UHP, which is machined brass, can be used in

Figure 2. Single Stage Gas Flow Path



796-0054

Figure 3. Two Stage Gas Flow Path



796-0055

panel mounting applications. There is a 1/8" male stainless steel Swagelok fitting on the regulator outlet, which is compatible with both copper and stainless steel tubing.

Conclusion

The gas pressure regulator should be designed to minimize the amount of incoming contaminants and remove them before they enter the chromatographic system. This helps guarantee the highest quality gas, resulting in maximum column life and ultimate performance. With the need of the chromatographer in mind, our newly updated line of gas cylinder pressure regulators provides a quality product for inert gases that will reduce and maintain the pressure from a cylinder to a level that can be used by the system, ensuring safety to both operator and instrument.

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Stage	Grade	Pressure (psi/bar)	Cat. No.
CGA 580 Regulators			
Two	HP	0-100 / 0-6.9	29557-U
Single	HP	0-100 / 0-6.9	29556-U
Two	HP+	0-100 / 0-6.9	29574-U
Two	HP+	0-150 / 0-10.3	29575-U
Single	HP+	0-100 / 0-6.9	29573-U
Two	UHP	0-100 / 0-6.9	29585-U
Single	UHP	0-100 / 0-6.9	29584-U
DIN 6 Regulators			
Two	HP	0-100 / 0-6.9	29559-U
Single	HP	0-100 / 0-6.9	29558-U
Two	HP+	0-100 / 0-6.9	29577-U
Two	HP+	0-150 / 0-10.3	29578-U
Single	HP+	0-100 / 0-6.9	29576-U
Two	UHP	0-100 / 0-6.9	29588-U
Single	UHP	0-100 / 0-6.9	29587-U
CGA 350 Regulators			
Two	HP	0-100 / 0-6.9	29561-U
Single	HP	0-100 / 0-6.9	29560-U
Two	HP+	0-100 / 0-6.9	29581-U
Single	HP+	0-100 / 0-6.9	29579-U
Two	UHP	0-100 / 0-6.9	29591-U
Single	UHP	0-100 / 0-6.9	29589-U
DIN 1 Regulators			
Two	HP	0-100 / 0-6.9	29563-U
Single	HP	0-100 / 0-6.9	29562-U
Two	HP+	0-100 / 0-6.9	29583-U
Single	HP+	0-100 / 0-6.9	29582-U
Two	UHP	0-100 / 0-6.9	29593-U
Single	UHP	0-100 / 0-6.9	29592-U
CGA 320 Regulators			
Two	HP	0-100 / 0-6.9	29567-U
Single	HP	0-100 / 0-6.9	29564-U
CGA 590 Regulators			
Two	HP	0-100 / 0-6.9	29569-U
Single	HP	0-100 / 0-6.9	29568-U
DIN 13 Regulators			
Two	HP	0-100 / 0-6.9	29572-U
Single	HP	0-100 / 0-6.9	29571-U

+ Related Information

CGA	DIN	Gas
580	6	helium, nitrogen, argon
350	1	hydrogen, methane, argon/methane
320	-	carbon dioxide
590	13	compressed air

Did you know...?

Gas cylinder pressure can have an affect on the quality of the carrier gas entering the regulator. Two-stage pressure regulators will not operate effectively at pressures less than 300 psig. Below this pressure they begin to function more like a single stage regulator, resulting in variable pressure. Without constant pressure, retention times will vary and irreproducible chromatography may result. To avoid this from happening we recommend changing gas cylinders when the pressure drops between 500 and 300 psig.

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GC-MS Tuning Solutions, Internal and Surrogate Standards

Jim Walbridge and Kathy Kiefer
techservice@sial.com

Because of its sensitivity and specificity, GC-MS is a widely used technique in environmental and forensic chemistry. Some important applications within these markets include many EPA methods, drugs of abuse monitoring and fire and explosives residue investigations. To get the most out of your GC-MS analysis, it is important to choose the proper internal standards, surrogates and tuning solutions, like those offered by Sigma-Aldrich under its Supelco brand.

Tuning Solutions

Tuning solutions are utilized to “tune” or optimize MS detector parameters. This maximizes the accuracy of mass and ion abundance measurements. Some tuning solutions also contain compounds used to assess injection port inertness and GC column performance. Our tuning solutions are formulated as specified in particular EPA methods and are important tools for the qualification of GC-MS systems.

Internal Standards

GC-MS protocols typically utilize the internal standard method of quantification. Internal standards gauge the method’s ability to extract target analytes from the sample matrix, and are used to monitor and adjust instrument fluctuations. Pentafluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene- d_4 and chlorobenzene- d_5 are examples of internal standards.

Surrogate Standards

Surrogate standards are used to monitor the entire analytical process from sample preparation to instrument performance. Toluene- d_8 , 4-bromofluorobenzene and dibromofluorobenzene are examples of surrogate standards.

The table below lists a sample of the many GC-MS standards and tuning solutions available from Sigma-Aldrich. A Certificate of Analysis is supplied with each product. The single and multi-component solutions are certified for identity, purity and concentration. Each component is guaranteed to be within +/-0.5% of the stated concentration.

To view our complete offering of GC-MS standards, please visit sigma-aldrich.com/standards.

+ Featured Products

Description	Concentration	Pkg. Size	Cat. No.
Surrogate Standards			
8270 Surrogate Standard <i>2,4,6-Tribromophenol, 2-Fluorobiphenyl, 2-Fluorophenol, Nitrobenzene-d_5, Phenol-d_6, p-Terphenyl-d_{14}</i>	4000 µg/mL in methylene chloride	1 x 1 mL	47960-U
Toluene- d_8	2000 µg/mL in methanol	1 x 1 mL	48593
1-Bromo-4-fluorobenzene	2000 µg/mL in methanol	1 x 1 mL	48083
Tuning Solutions			
EPA 8270 GC-MS Tuning Solution <i>Benzidine, 4,4-DDT, Pentachlorophenol, DFTPP</i>	50 µg/mL each in methylene chloride	1 x 1 mL	47387
Decafluorotriphenylphosphine (DFTPP)	–	100 mg	442543
Decafluorotriphenylphosphine (DFTPP)	1000 µg/mL in acetone	1 x 1 mL	47941
1-Bromo-4-fluorobenzene	25000 µg/mL in methanol	1 x 1 mL	48800
Internal Standards			
EPA 8620 Internal Standards Mix <i>1,4-Dichlorobenzene-d_4, 1,4-Difluorobenzene, Chlorobenzene-d_5, Pentafluorobenzene</i>	2000 µg/mL in methanol	1 x 1 mL	48958
Semivolatiles Internal Standards Mix <i>1,4-Dichlorobenzene-d_4, Acenaphthene-d_{10}, Chrysene-d_{12}, Naphthalene-d_8, Perylene-d_{12}, Phenanthrene-d_{10}</i>	2000 µg/mL in methylene chloride	1 x 1 mL	46955-U

¹³C isotope-labeled Mycotoxin Standards for GC- and LC-MS-MS

Pat Myers

techservice@sial.com

Mycotoxins are a diverse group comprised of hundreds of secondary metabolic products of various fungal species. Many show at least some toxicity in one or more organisms. Several show marked toxicity in humans with the effects of high-level exposure including death, hepatic toxicity, renal toxicity, neurotoxicity, immunosuppression, and cancer. The effects of long-term low-level exposure have not been studied well.

Contamination of the food supply with mycotoxins is increasingly prevalent. This is likely due to increasing environmental and economic stresses. Contamination with fungi can occur during growth, harvest, transportation, processing, or storage of the food product. Water stress (either too much, too little, or at the wrong time), temperature stress, or infestation with insects can all lead to fungal contamination. Improper storage and transportation conditions can also cause or exacerbate the problem.

Human exposure to mycotoxins can be either by direct consumption of contaminated food sources or indirectly by consumption of products from animals fed contaminated feed. Techniques to reduce mycotoxin concentration after contamination are expensive, unreliable and sometimes reversible. Prevention of contamination and removal of contaminated products from the food chain are the only viable means of eliminating human exposure. Sensitive and accurate detection of very low levels of these compounds is critical to governmental efforts to eliminate contaminated food sources from the market-

place. Accredited local laboratories capable of running validated and certified analyses of mycotoxins are a necessary part of the regulatory system.

A common method used to ensure the traceability to SI units required for validation and certification is the use of a primary-ratio method of analysis. Double Isotope Dilution Mass Spectroscopy (dIDMS) is recognized by the Comité consultatif pour la quantité de matière (CCQM) of the Bureau International des Poids et Mesures (BIPM) as a primary method of measurement.

Both fully ¹³C isotope-labeled analogs of the mycotoxins and corresponding natural composition standards are required for dIDMS. In dIDMS, the sample and the analytical standard are each spiked with the isotope-labeled analog. Either GC-MS-MS or LS-MS-MS is then used to determine isotope ratios in the sample and standard. The change in isotope ratios caused by the addition of a known amount of ¹³C isotope-labeled analyte analog is used to calculate the concentration of the analyte in the original sample. Additionally, because the ¹³C isotope-labeled analogs behave chemically identical to the natural composition analytes, variations in sample preparation have little impact on the uncertainty of the analyses.

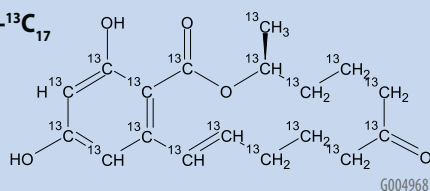
Sigma-Aldrich now provides a full range of ¹³C isotope-labeled mycotoxin standards along with the required natural composition standards for use in dIDMS, the newest of which are listed below. Please visit sigma-aldrich.com/mycotoxins to view the entire line of mycotoxin standards.

References

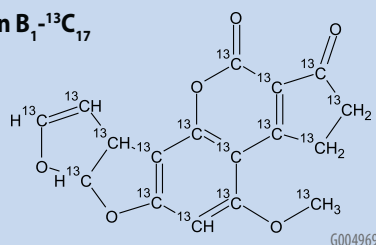
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3. Fernandes-Whaley, Maria. The role of organic chemical metrology in food safety for South Africa. Pretoria: National Metrology Institute of South Africa, 2009.

Figure 1. Fully ¹³C isotope-labeled Mycotoxins

Zearalenone-¹³C₁₇



Aflatoxin B₁-¹³C₁₇



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Description	Concentration	Pkg. Size	Cat. No.
Aflatoxin B ₁		5 mg	32754
Aflatoxin B ₁ - ¹³ C ₁₇ solution	0.5 µg/mL in acetonitrile	1 mL	32764
Aflatoxin B ₂		5 mg	32755
Aflatoxin B ₂ - ¹³ C ₁₇ solution	0.5 µg/mL in acetonitrile	1 mL	32771
Aflatoxin G ₁		5 mg	32756
Aflatoxin G ₁ - ¹³ C ₁₇ solution	0.5 µg/mL in acetonitrile	1 mL	32772
Aflatoxin G ₂		5 mg	32757
Aflatoxin G ₂ - ¹³ C ₁₇ solution	0.5 µg/mL in acetonitrile	1 mL	32777
Zearalenone- ¹³ C ₁₈ solution	0.5 µg/mL in acetonitrile	1 mL	32758

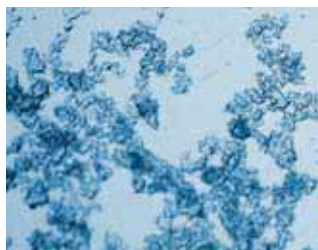
Sigma-Aldrich – Your Source for Physical Property Standards

Matthias Nold
techservice@stal.com

For the determination of the identity and purity of a substance, measuring of physical properties plays an important role. To ensure the accuracy of these measurements, the measurement equipment needs to be regularly calibrated using accurate analytical standards with known properties.

Sigma-Aldrich is well-known for providing a huge portfolio of analytical standards used in the quantitative and qualitative analysis and for identity control by chromatographic and spectroscopic methods. However, Sigma-Aldrich also offers a wide range of physical property standards for various applications in the chemical, food and material science industries. Taking into account the growing need for certified reference materials (CRM), our physical property standards offering includes many products from recognized manufacturers of high-quality CRM such as IRMM, Whitehouse Scientific, H&D Fitzgerald or Paragon Scientific.

The product offering covers a wide variety of physical properties:



For calibration of melting point instruments, a range of **melting point standards** is available. The melting points of the standards are measured in the thermodynamic mode and are traceable to primary reference material.

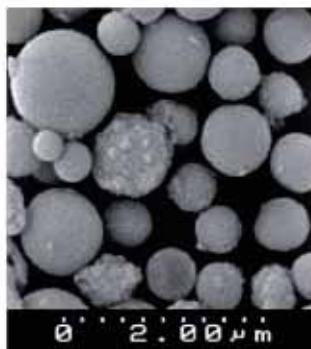
Our **density standards** are manufactured and certified by respected density metrologists H&D Fitzgerald from the UK and are ISO 9001 and ISO 17025 compliant.



Recently, a big range of certified **viscosity standards** have been added to the Sigma-Aldrich product offering. These products are also produced under ISO 17025 by Paragon Scientific.

The Fluka **conductivity standards** comply with ISO 7888, which describes the use of electrical conductivity for monitoring water quality.

Also under the Fluka brand, we offer two sets of **turbidity standards**. These can be directly used for calibration of the AQUANAL-plus Spectro Turbidity instrument but are also suitable for any other turbidity instrument.



For the calibration of particle size measurement equipment and for the calibration of sieves we offer NIST and NPL traceable **particle size and sieve calibration standards**. These products are manufactured and certified by the recognized specialists from *Whitehouse Scientific*.

The most obvious characteristic of a material is the appearance, especially the color. Sigma-Aldrich offers a series of **color reference solutions** according to Ph. Eur, USP and ASTM/APHA.



The Sigma-Aldrich **color chart for solids** (Cat. No. 91711) has been especially developed for the use in quality control of all the Sigma-Aldrich production sites. It consists of a selection of color chips from the Munsell Books

of Color. The colors included in the chart are the ones we identified as the most frequently occurring in chemical quality control.

Last year, the **IRMM** (Institute of Reference Materials and Measurements), an institute of the Joint Research Center of the European Commission, renewed its contract with Sigma-Aldrich. As an official distributor, Sigma-Aldrich offers the whole range of the IRMM certified reference materials, including about 80 CRMs for physical properties (thermal, mechanical, morphological and optical properties).

A comprehensive brochure containing product listings and technical information about the Sigma-Aldrich physical property standards has been issued recently. To order this brochure, please visit our webpage at sigma-aldrich.com/standards and follow the "Request Literature" link. You can also browse the complete and up-to-date product offering for physical property standards at sigma-aldrich.com/physicalproperties

Precleaned Vials



US EPA Methods 8260, 624, and 524 all cite the use of “clean vials” for sample collection to reduce the possibility of contamination from trace volatile organics found on the glass surface. The methods also provide instructions for the proper cleaning of the vials. These instructions state that the components of the vial system should be washed with soap and water, rinsed with distilled deionized water, and then dried in an oven at 100 °C for approximately one hour.

To reduce the possibility of sample contamination and to save time, Supelco offers a line of precleaned sampling vials that conform to the EPA’s definition of clean vials. These vials are manufactured from Type I borosilicate glass and are cleaned for water sampling according to US EPA Title 40: Protection of Environment, Part 136 Guidelines establishing test procedures for the analysis of pollutants.

All vials are shrink wrapped immediately after cleaning to ensure their cleanliness. They are offered in clear and amber glass, and in sizes ranging from 2 to 40 mL. All are assembled with a tan PTFE/white silicone septa and an open-top, black, polypropylene cap. The 2 to 22 mL vials are shipped with a Certificate of Conformity, and the 40 mL EPA/VOA vial with a Certificate of Analysis. Labels are conveniently provided with all package sizes.

Capacity	Dimensions	Septa Thickness PTFE/Silicone		Pkg. Size	Cat. No.
		in.	mm		
Clear Glass					
2 mL	12 x 32 mm	0.005/0.055	0.127/1.397	100	27339
4 mL	15 x 45 mm	0.005/0.055	0.127/1.397	100	27340
7 mL	17 x 60 mm	0.005/0.055	0.127/1.397	100	27341
15 mL	21 x 70 mm	0.010/0.115	0.254/2.921	100	27342
22 mL	23 x 85 mm	0.005/0.055	0.127/1.397	100	27343
40 mL	28 x 98 mm	0.010/0.090	0.254/2.286	72	23188
Amber Glass					
2 mL	12 x 32 mm	0.005/0.055	0.127/1.397	100	27344
4 mL	15 x 45 mm	0.005/0.055	0.127/1.397	100	27345
7 mL	17 x 60 mm	0.005/0.055	0.127/1.397	100	27346
15 mL	21 x 70 mm	0.010/0.115	0.254/2.921	100	27347
22 mL	23 x 85 mm	0.005/0.055	0.127/1.397	100	27348
40 mL	28 x 98 mm	0.010/0.090	0.254/2.286	72	23189

Silanization Service for Vials

High concentrations of silanol groups (Si-O-H) on untreated glass vial surfaces can catalyze decomposition of unstable compounds or adsorb polar compounds through hydrogen bonding. Quantitative analyses of these sensitive compounds become unreliable, recoveries are

reduced and analyses can be complicated by decomposition byproducts.



Supelco uses an environmental friendly process to silanize glass vials. This organosilanization process derivatizes surface silanols at a high temperature, shielding the active groups from contact with the active hydrogens on the sample components.

Our experience includes silanizing vials ranging from 2 mL to 40 mL, and maintaining a stock of popular 2 mL and 4 mL silanized vials. If your application requires a different vial, you may choose from the large assortment of Supelco brand vials, or send us your own vials for treatment.

To obtain a quote or learn more about our silanization process, please contact our Technical Service department at techservice@sial.com

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Ion Pair Chromatography

Hansjörg Tinner, Michael Kiselewsky
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Chromatography is omnipresent in analytical research in spite of the development of instruments of extreme ranges of sensitivity and selectivity. Several traditional variations of chromatography are still widely used though many computer assisted advanced analytical instruments are now available. One such chromatographic variation is Ion Pair Chromatography (IPC).

In the past, the approach used to separate charged analytes was ionic suppression. By changing the pH value of the mobile phase, charged analytes become non-ionized. This approach of method development can be time consuming and is better suited for single analyte or simple mixtures where pK_A's of the analytes are close.

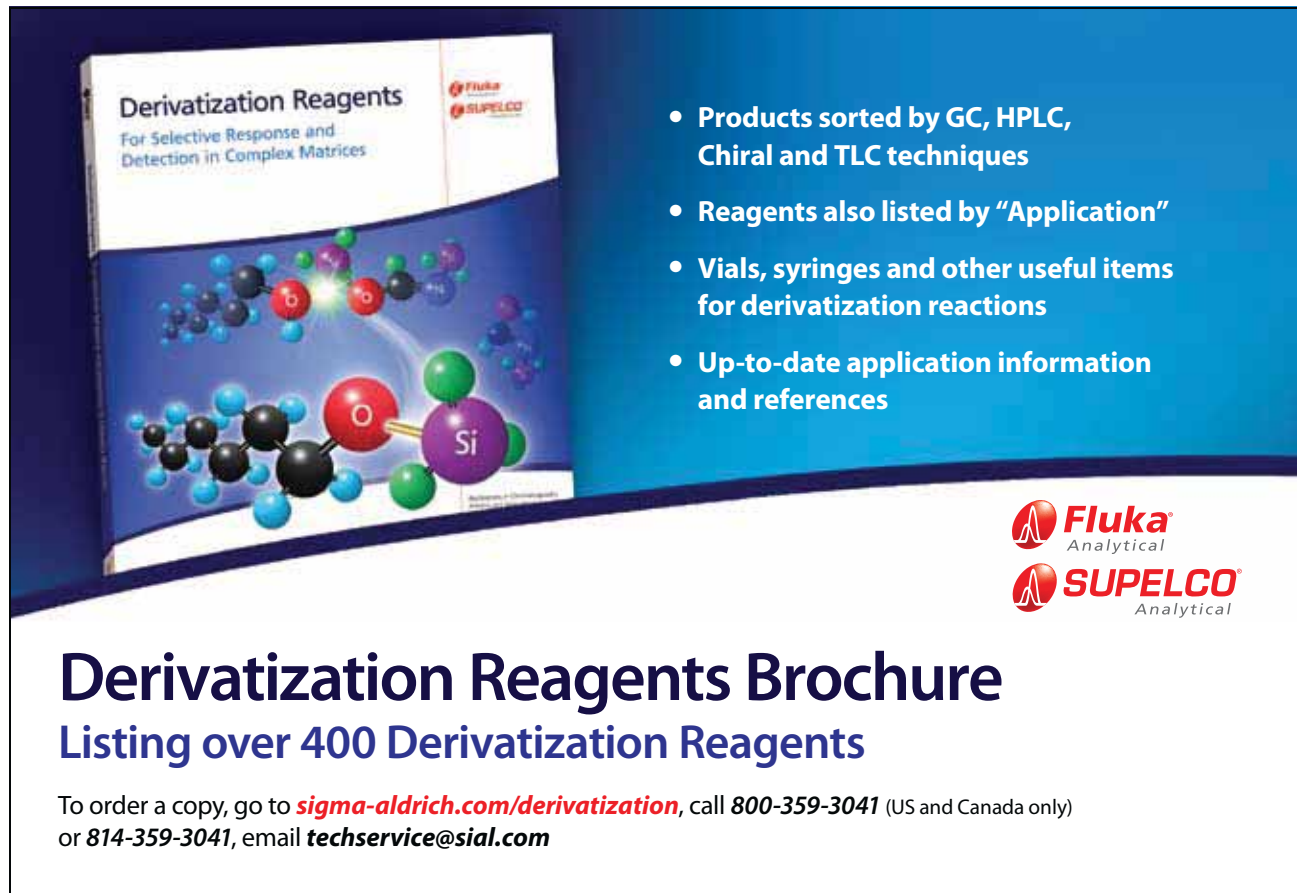
On the other hand, IPC is a more general and applicable approach that allows the separation of complex mixtures of very polar and ionic molecules. The mobile phase is supplemented with an ion-pairing reagent. Ion-pairing reagents consist of large ionic molecules having a charge opposite to the analyte of interest as well as a substantial hydrophobic region that allows interaction with the stationary phase, plus

associated counter-ions. In total, IPC results in different retention of analytes to facilitate separation. IPC is an established and reliable technique that provides:

- Reduced separation times
- Highly reproducible results
- Sharper peak shapes
- Simultaneous separation of ionized and non-ionized analytes
- Wide choice of additives to improve separation

Figure 1 presents an example of polar compounds that were successfully separated on a Supelco Ascentis Express C18 column using IPC additives. The analytes are imidazolium and pyridinium derivatives. The column was subsequently rinsed with solvent in order to prevent ion pair reagent agglomeration. A gradient consisting of acetonitrile with a buffer of 1.1 g sodium 1-heptanesulphonate monohydrate (Fluka 51832) and 700 µL phosphoric acid 85% (Fluka 79606) in 1 L water was used.

The more sensitive modern instruments become, the easier they will detect any impurity added by auxiliaries in addition to the analytes of interest. Therefore, the purity of any kind of eluent additives will influence performance and



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Derivatization Reagents Brochure

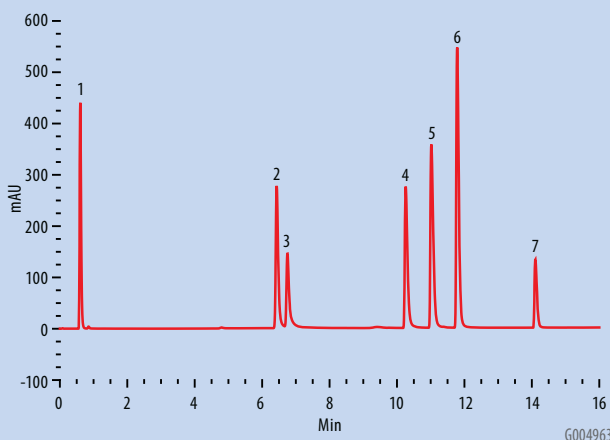
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Figure 1. Separation of Polar Compounds Using Ion Pair Additives

column: Ascentis Express C18, 7.5 cm x 4.6 mm I.D., 2.7 μ m (53819-U)
 sample volume: 5 μ L
 flow rate: 1.0 mL/min
 temp: 25 $^{\circ}$ C
 UV detection: 210 nm
 gradient: t = 0.2% acetonitrile
 t = 1 min to 10 min 2 – 20% acetonitrile
 t = 15 min 35% acetonitrile.

1. Anions (e.g. Br)
2. 1-(3-Cyanopropyl)-3-methylimidazolium dicyanamide
3. 1-Methyl-2-vinylpyridinium triflate
4. 1-Butyl-3-methylimidazolium bromide
5. 1-Butyl-2,3-dimethylimidazolium tetrafluoroborate
6. 1-Benzyl-3-methylimidazolium tetrafluoroborate
7. 1-Hexyl-3-methylimidazolium chloride



accuracy. Only products that have been tested for suitability and carefully analyzed for purity will guarantee the quality and performance in an application.

Sigma-Aldrich has a long tradition of offering superior quality analytical reagents. We are proud to offer a wide range of accurately tested IPC reagent products under our Fluka brand. Our reagents are of the highest purity and

exhibit minimal extinction in the low UV region. These reagents exhibit excellent transparency down to 200 nm, even at high concentrations. In addition, they are tested for the absence of insoluble matter. Non-absorbing impurities like redox-traces that can interfere with the sample are also tested. The suitability tests are carefully performed using a very steep gradient.

A complete list of products is available on our website:

sigma-aldrich.com/ipc

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Anionic and Cationic Reagents for Ion Pair Chromatography (IPC)

Description	Carbon Length	Cat. No.
Ion-pair Additives for Cationic Separation		
Sodium 1-pentanesulphonate monohydrate	C5	76952
Sodium 1-hexanesulphonate monohydrate	C6	52862
Sodium 1-heptanesulphonate monohydrate	C7	51832
Sodium 1-octanesulphonate monohydrate	C8	74882
Sodium octyl sulphate	C8	75073
Sodium 1-nonanesulphonate	C9	74316
Sodium 1-decanesulphonate	C10	30631
Sodium dodecyl sulphate	C12	71726
Ion-pair Additives for Anionic Separation		
Tetramethylammonium chloride	C1	74202
Tetrapropylammonium bromide	C3	88103
Tetrabutylammonium chloride	C4	86852
Tetrabutylammonium bromide	C4	86857
Tetrabutylammonium bisulphate	C4	86853
Tetrahexylammonium hydrogensulphate	C6	87299
Tetraheptylammonium bromide	C7	87296
Tetrakis(decyl)ammonium bromide	C10	87578
Hexadecyltrimethylammonium bromide	C16	52367

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