

# the Reporter

EUROPE

Volume 21, May 2006 International, Issue

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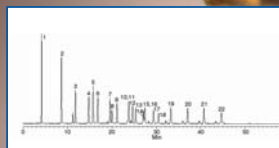
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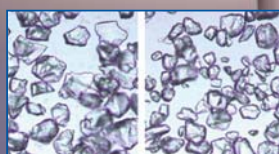
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**SIGMA-ALDRICH**

## EDITORIAL

## NEW Sigma-Aldrich Analytical Standards Portal

## Dear Reader,

With the burgeoning use of the internet as a source for information, especially via search engines, it should come as no surprise that we set a priority on leveraging our website to maximise its utility as a resource for application, technical and product information. Customers using the internet to obtain such information expect speed and relevance; they want the right information, quickly, with minimal amount of clicks or rabbit holes.

Another growing area is the use of analytical standards. From forensics to pharmaceuticals, foods to petrochemicals, the availability of reliable standards and certified reference materials improves both the accuracy of the analysis and, with more and more samples to analyse, reduces the time required by laboratory personnel to prepare and validate standards.

Growing use of internet, growing use of analytical standards; that's why we focused on developing the best website for sourcing our analytical standards.

In this issue of the Reporter Europe, we introduce our Analytical Standards portal – your gateway to the extensive collection of thousands of standards and reference materials for all areas of analytical chemistry and spectroscopy from our trusted Fluka, Riedel-de Haën, Sigma and Supelco brands. The portal allows

you to browse our collection via a new search engine: the Standards Explorer that permits multiple keyword searching by name, compound, CAS number and even molecular formula.

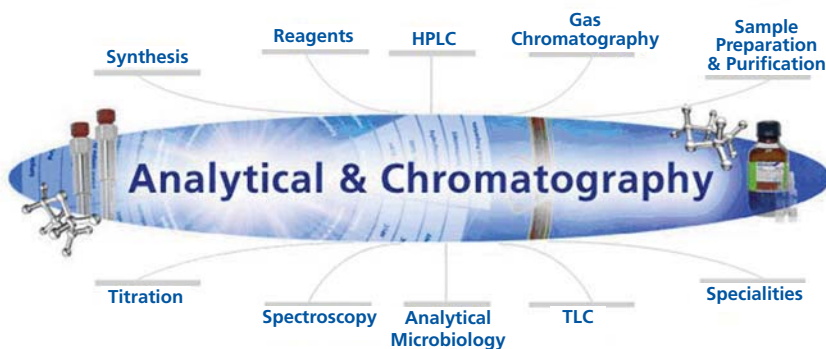
The Analytical Standards portal represents our commitment to bringing you the right information, and presenting it in a fashion that is modern, fast, convenient and, perhaps this is a bit of a stretch, fun to use.

I hope you find the Analytical Standards portal and the rest of the articles in this issue of the Reporter Europe useful. Please feel free to send me any comments and suggestions you might have. I look forward to hearing from you!

Kind regards



Ingo Haag, PhD  
Analytical Marketing Manager



[www.sigma-aldrich.com/analytical](http://www.sigma-aldrich.com/analytical)

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## HPLC ARTICLE

## HPLC Analysis of DNPH-Derivatives of Carbonyl Compounds on Ascentis RP-Amide Universal Reversal-Phase Columns

Hugh Cramer Applications Technician and Jacinth McKenzie, Ph.D. Sr. Applications Chemist

**Introduction**

Versatility is one of the primary reasons why HPLC has become a ubiquitous technique in analytical laboratories across nearly all market segments. The ability to alter the stationary phase chemistry to achieve the desired separation is one of the main contributors to this versatility. In this short communication we describe the separation of twenty-one aldehydes and ketones as their DNPH-derivatives on an Ascentis™ RP-Amide column. Ascentis™ RP-Amide has universal applicability; it provides excellent peak shape, retains and resolves both polar and non-polar compounds, has unique selectivity compared to C18 and can be used with all HPLC detectors, including sensitive, low-bleed LC-MS analysis.

**Importance of carbonyl analysis**

Carbonyls – aldehydes and ketones – are found everywhere, including chemical and plastics manufacturing, food and the environment. They occur naturally, serving as reactants or products in many biochemical processes (1, 2). Unfortunately, many aldehydes and ketones, especially formaldehyde, are associated with air pollution and are known to contribute to ozone depletion (3). Others carbonyls are eye, skin and respiratory system irritants and many have also been identified as animal teratogens, mutagens and carcinogens (4).

**Analytical challenges of carbonyls**

The analysis of carbonyl compounds using HPLC or GC is complicated by three factors. First, they often lack significant chromophores and therefore are difficult to detect at low levels using UV detection. Second, the lower molecular weight carbonyls are highly polar and therefore difficult to retain by reversed-phase HPLC columns. Third, carbonyls are highly reactive with glass and metal surfaces so there are quantification problems when trying to analyse them using GC.

Derivatisation overcomes these problems associated with carbonyl analysis. One of the most commonly used derivatisation reagents is 2,4-dinitrophenylhydrazine (DNPH). Carbonyls react with DNPH under acidic conditions with the formation of the respective hydrazones as shown in Figure 1. The resulting DNPH derivatives have greatly increased UV sensitivity and improved hydrophobic retention. This reaction can be performed using either liquid phase or solid-phase synthesis techniques.

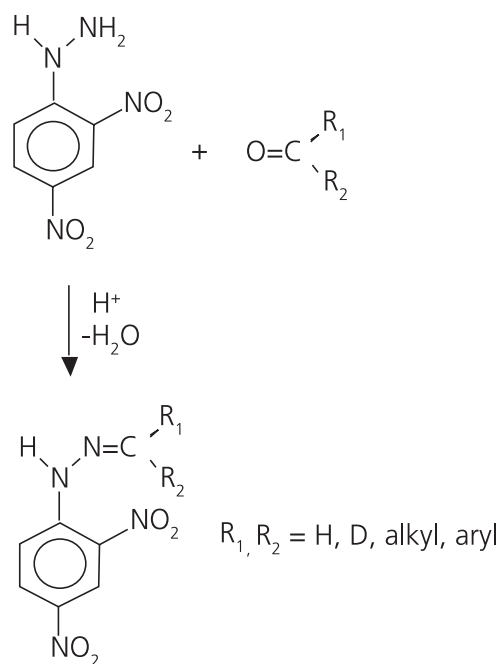
**Sigma-Aldrich solutions for carbonyl analysis**

Sigma-Aldrich offers a complete solution for the analysis of aldehydes and ketones, including pre-derivatized DNPH-carbonyl standards, derivatization reagents, solvents and chromatographic columns. For solid-phase derivatisation, the Supelco LpDNPH S10 adsorbent cartridge is pre-coated with DNPH and used for sampling carbonyls from ambient and indoor air as described in US EPA and ASTM methods.

To achieve the HPLC separation of DNPH-derivatised carbonyls, we offer the C18 columns specified in EPA Method 8315 (aqueous and solid waste, soil, stack gas, and indoor air samples) and EPA Method 554 (drinking water) (5). However, as discussed previously, carbonyls are of interest in many areas besides environmental. The separation of carbonyls and polar compounds in general can benefit from the different selectivity offered by alternate HPLC stationary phases, such as the Ascentis RP-Amide. This stationary phase operates by a reversed-phase

mechanism, but the polar embedded group, in this case an amide, gives it enhanced polar retention and different selectivity compared to a C18. Often, an Ascentis C18 column and an Ascentis RP-Amide can be used in conjunction; their differing selectivities being applied to aid in peak confirmation.

**Figure 1.** Reaction of carbonyl compounds with DNPH

**Materials and Methods**

The separation of twenty-one pre-derivatised DNPH-carbonyl compounds (Standard TO11/IP-6A Aldehyde/Ketone-DNPH Mix, 47285-U) was run on a 15 cm x 4.6 mm Ascentis RP-Amide column packed with 3  $\mu\text{m}$  particles (565322-U). The chromatographic system was a Waters 2690 equipped with a 2996 PDA detector (set at 360 nm) and column heater (set at 30 °C). The gradient elution profile used the high purity gradient-grade G CHROMASOLV® solvents to ensure minimal baseline rise and reduce the occurrence of spurious peaks.

**Results: Unique selectivity of Ascentis RP-Amide**

Figure 2 shows the optimised gradient elution of twenty-one DNPH-derivatised carbonyl compounds on the Ascentis RP-Amide column. The separation was primarily on the basis of hydrophobicity, with low molecular weight carbonyls eluting first, followed by single ring carbonyl and finally the longer alkyl chain carbonyls. Of particular importance is the high degree of resolution between the reagent peak (DNPH, peak 1) and the first carbonyl peak (formaldehyde-2,4-DNPH, peak 2). This spacing is necessary in order to detect low levels of formaldehyde; with insufficient resolution the formaldehyde peak is lost and cannot be reliably quantified.

Although the analysis described here was run with UV detection at 360 nm, an additional benefit of the Ascentis RP-Amide column over other embedded polar group HPLC phases is that it has minimal bleed enabling both low UV and LC-MS conditions.

## References


1. Pamplona, R.; Dalfó, E.; Ayala, V.; Josep Bellmunt, M.; Prat, J.; Ferrer, I.; Portero-Otin, M.; Proteins in human brain cortex are modified by oxidation, glycooxidation and lipoxidation; *J. Biol. Chem.* 2005; 280, 21522 – 21530.
2. Gibbons, S.; Anti-staphylococcal plant natural products; *Natural Product Reports* 2004, 21(2), 263 – 277.
3. Derwent, R.G.; Jenkin, M.E.; Saunders, S.M.; Pilling, M.J.; Passant, N.R.; Multi-day ozone formation for alkenes and carbonyls investigated with a master chemical mechanism under European conditions; *Atmospheric Environment* 2005; 39 (4), 627 – 635.
4. Schultz, T.W.; Yarbrough, J.W.; Trends in structure-toxicity relationships for carbonyl-containing  $\alpha,\beta$ -unsaturated compounds; *SAR and QSAR in Environmental Research* 2004; 15(2); 139 – 146.
5. "Monitoring Carbonyls in Air Using the LpDNPH S10 Cartridge with Analysis by HPLC" Supelco Application Note 92 (T396092A).

## Ordering information

Prod. No.	Description	Pack Size
565322-U	Ascentis RP-Amide, 3 $\mu$ m, 15 cm x 4.6 mm	Each
565371-U	Ascentis RP-Amide guard column kit, 2 cm x 4 mm (Holder, one cartridge, fittings)	1 kit
47285-U	TO11/IP-6A Aldehyde/Ketone-DNPH Mix 15 components, each 15 $\mu$ g/mL in acetonitrile, suitable for ASTM D5197, EPA IP-6A, TO-11, TO-5	1 mL ampoule
21024-U	LpDNPH S10 Cartridge Starter Kit (10 cartridges, adapters)	1 kit
34877	G CHROMASOLV® Water	1 L, 2.5 L
34998	G CHROMASOLV® Acetonitrile	1 L, 2.5 L

For more information on Ascentis columns, please visit our website [www.sigma-aldrich.com/ascentis](http://www.sigma-aldrich.com/ascentis)

For more DNPH standards please refer to our website or Supelco catalogue.

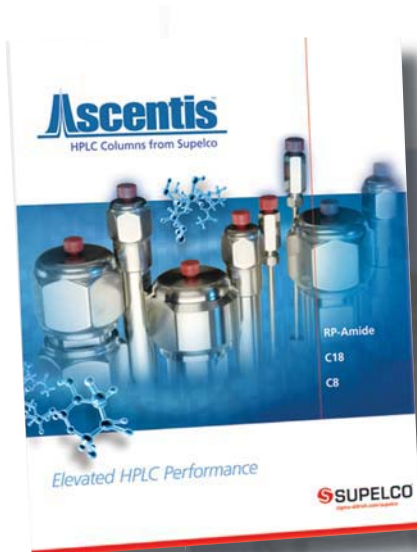
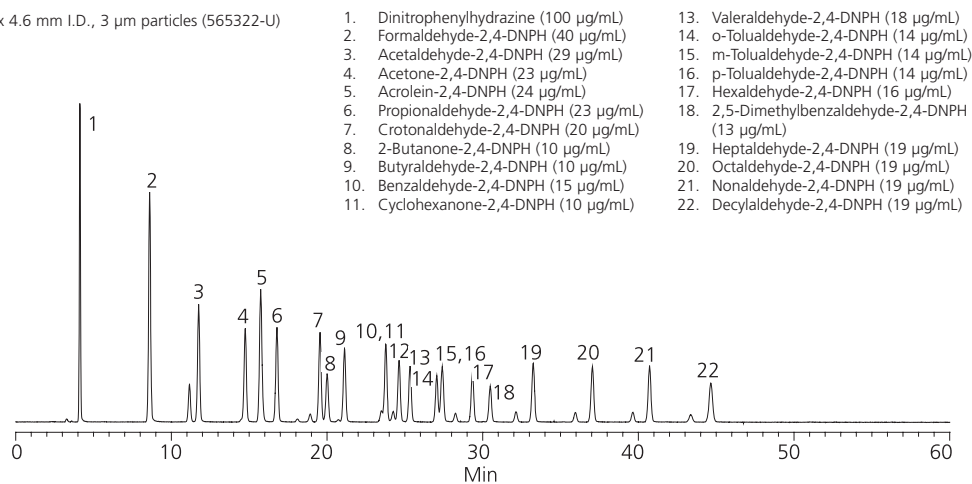
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**Figure 2.** Separation of carbonyl compounds on Ascentis RP-Amide

column: Ascentis RP-Amide, 15 cm x 4.6 mm I.D., 3  $\mu$ m particles (565322-U)  
 mobile phase A: 60:40, water:acetonitrile  
 mobile phase B: 25:75, water:acetonitrile  
 flow rate: 1.5 mL/min.  
 temp: 30 °C  
 det.: UV at 360 nm  
 injection: 10  $\mu$ L  
 sample: as listed, in mobile phase

Gradient:

Min	%A	%B
0	100	0
5	100	0
25	40	60
40	0	100
60	0	100



For information on the complete Ascentis product line featuring the RP-Amide, C18 and C8 phases, request the Ascentis Brochure: Elevated HPLC Performance. (Code HLV)

Additional information is available on our web site: [www.sigma-aldrich.com/ascentis](http://www.sigma-aldrich.com/ascentis)

## HPLC ARTICLE

# Ascentis™ RP-Amide Universal RP-HPLC Column – First Choice for Both Routine and Challenging HPLC Separations

By Klaus Herick, Ph.D. Sales Development Manager HPLC Europe

There is likely to be no argument against the statement that C18 is the most commonly used stationary phase chemistry for reversed phase HPLC (RP-HPLC). But with today's alternative chemistries, should it remain at the top? In this short communication, we hope to convince you that C18 may not always be your first choice and that others, specifically the alkyl-amide embedded polar group (EPG) phases, are definitely worthy of consideration.

### Stationary Phase Chemistry and Selectivity

When developing a new RP-HPLC method, there are generally two instances when the analyst needs to look beyond the primary column: when the column cannot perform the separation or when a confirmation column is required. In both cases, it is often selectivity proffered by the stationary phase that needs to be altered.

Modifying the stationary phase chemistry, for example, by inserting functional groups within the alkyl chain, changes the selectivity of the separation by changing the ways which analyte molecules interact with stationary phase molecules. This, in turn, changes the equilibrium distribution. The pure van der Waals (dispersive) interactions of the alkyl-only C18 chains can be supplemented by H-bond, dipole-dipole, ionic or other types of interactions.

However, any stationary phase modification done to enhance selectivity cannot cause diminution of other performance attributes, like retention, efficiency, reproducibility and signal to noise ratio, nor limit its compatibility with HPLC instruments and mobile phases.

### Universal RP-HPLC Phase: Ascentis™ RP-Amide

Representing nearly two decades of refinements in both silica and bonded phase technologies (refer to article starting on page 6), Ascentis RP-Amide meets these requirements. No longer sequestered to specialty applications, Ascentis RP-Amide is a truly universal RP-HPLC column, suitable for both a first choice column and confirmation column. Compared even to C18, the Ascentis RP-Amide has wide applicability because of its intrinsic characteristics.

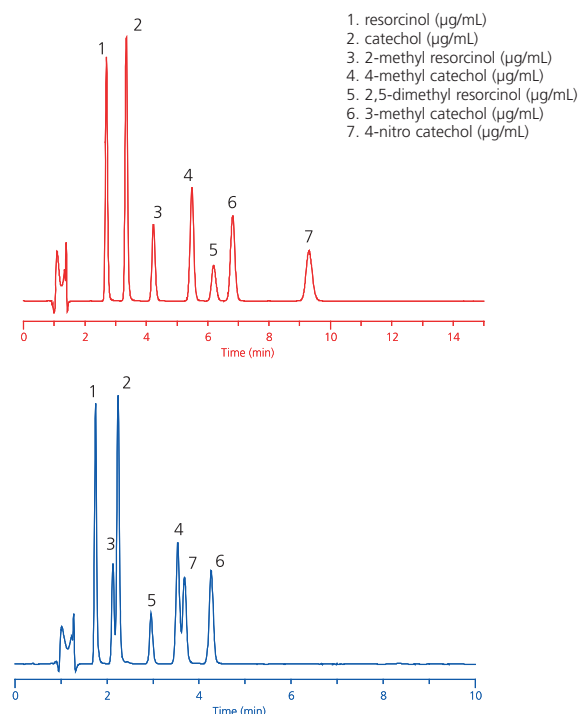
#### Ascentis RP-Amide Characteristics:

- Predictable reversed-phase mechanism
- Enhanced polar compound retention
- Ultra-low bleed for LC-MS compatibility
- High retentivity (important for LC-MS and preparative applications)
- Different selectivity compared to C18, C8, cyano, etc.
- Excellent peak shape for acids, bases, chelators and zwitterions
- Reproducible, rugged and stable
- No phase collapse under 100% aqueous conditions
- Scalability from microbore to preparative dimensions
- World-wide availability, reliable supply and expert customer support through the Sigma-Aldrich organisation

Space does not permit a complete dissertation on these characteristics. However, the accompanying figure shows one example of the improved retention and selectivity of polar compounds on the RP-Amide compared to a C18 on the same silica substrate (Figure 1). For more information, please request the Ascentis brochure, or visit our website [www.sigma-aldrich.com/ascentis](http://www.sigma-aldrich.com/ascentis)

**Figure 1.** Enhanced retention and selectivity of Ascentis RP-Amide (top) compared to C18 (bottom)

columns: 15 cm x 4.6 mm, 5 µm particles  
 mobile phase: (75:25) 20 mM phosphoric acid (pH 2.0 unadjusted):CH<sub>3</sub>CN  
 temp: 30 °C  
 flow rate: 1.5 mL/min  
 det: UV, 270 nm  
 sample: catechols and resorcinols (50 µg/mL) in 20 mM phosphoric acid (pH 2.0 unadjusted)



#### Product List: Ascentis™ RP-Amide HPLC Columns (select dimensions only)

Prod. No.	Particle Size (µm)	ID (mm)	Length (cm)
565320-U	3	4.6	5
565322-U	3	4.6	1
565303-U	5	2.1	5
565306-U	5	2.1	25
565323-U	5	4.6	5
565324-U*	5	4.6	15
565325-U	5	4.6	25

Ascentis C18 and C8 phases, guard columns and other dimensions of Ascentis RP-Amide are available. [www.sigma-aldrich.com/ascentis](http://www.sigma-aldrich.com/ascentis)

\*Dimension shown in the accompanying application

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## HPLC ARTICLE

# Embedded Polar Group Stationary HPLC Phases: A Track Record of Continuous HPLC Innovation at Supelco

By **Wayne Way Ph.D.** Market Segment Manager HPLC and **Klaus Herick Ph.D.** Sales Development Manager HPLC Europe

## Introduction

Research aimed at innovative HPLC phases at Supelco is nothing new. Supelco researchers introduced the first so-called “deactivated” C18 phase, SUPELCOSIL LC-18-DB, in the late 1970’s, when HPLC technology was still in its relative infancy. Research continued that focused on the power of both silica treatments and stationary phase modifications, particularly in the embedded polar group (EPG) technology, to improve chromatographic performance. Although research is still ongoing, today the fruits of these labours can be seen in the Ascentis™ RP-Amide universal RP-HPLC column. The evolution of the Ascentis line and the state-of-the-art RP-Amide phase is discussed briefly in this article.

## The Dawn of Modern HPLC

In the 1980’s, the HPLC community was keenly interested in techniques to improve the peak shape of basic and chelating compounds. At that time, the pure “type B” silicas we have today were not available. The first “type A” silicas based on sodium silicate had excellent mechanical and physical properties, but suffered from metal ion contamination and heterogeneity of the surface silanol population. Surface treatments had a positive effect in reducing the silanol interactions, but not to a sufficient extent. Basic compounds still exhibited excessive tailing, especially at or above their  $pK_a$ .

Because the surface activity of the type A silicas could not be fully eliminated, researchers then began looking at methods to shield sensitive analytes from the silica surface. Various techniques were applied, and many became commercially available. In 1988, Supelco pioneers patented and commercialized a novel deactivation technique which would become known as EPG, or embedded polar group, deactivation (1, 2).

## Early EPG Phases: Deactivation with Surprising Selectivity Advantages

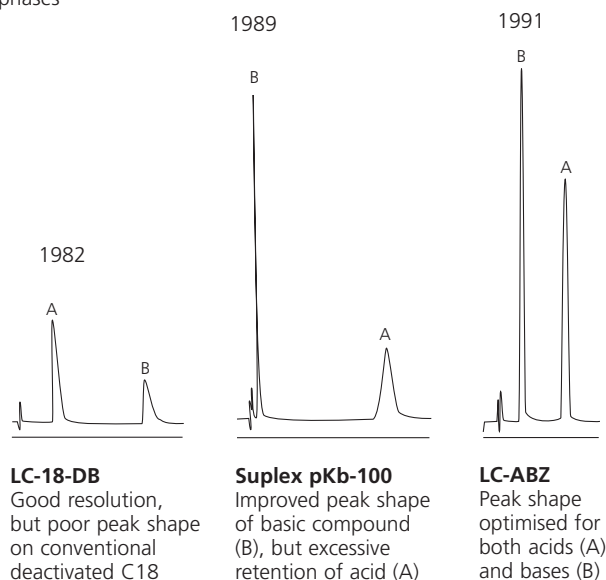
Although many functional groups were tested, a commercially successful EPG phase was made by inserting an amide group within the alkyl chain, close to where it bonds to the silica surface. This first EPG phase was called Suplex™ pKb-100; the “pKb” refers to its utility toward bases, and the “100” for the pore size of the silica. The results were dramatic and caught a lot of attention at the time. Various hypotheses on the mechanism of the deactivation abounded, including via electrostatic shielding and solvation (computer simulations demonstrated an enrichment of water molecules at the surface compared to a C18).

Soon after introduction, however, these same development chemists were surprised to find as much, if not more, interest in the unique selectivity conferred by the amide group than in its intrinsic deactivation effect. Quickly, R&D attention was turned toward understanding and optimising the performance of the EPG phases toward different classes of compounds.

The second and third members of the Supelco EPG family were SUPELCOSIL LC-ABZ and ABZ+Plus, introduced in 1990 and 1993, respectively. Both of these phases were based on type A silica and the Suplex synthesis, with slight modifications to improve their performance toward acids and zwitterions in addition to bases (the “ABZ” stands for acid, base and

zwitterions). The evolution in improvement from LC-18-DB to LC-ABZ is depicted in Figure 1.

**Figure 1.** Evolution of Improvement of Supelco deactivated HPLC phases



## The Rise of LC-MS and Pure Silica Gels: Sensitivity and Reproducibility

The mid-1990s were a time of active growth in HPLC. Instruments were evolving along with the columns. The advent of LC-MS, previously available to only a few large research institutions, was fast becoming a routine analytical tool. Its power to see and identify analytes, especially at low levels, was compromised by contaminants derived from mobile phases and column bleed. While all HPLC phases exhibit some level of column bleed, EPG phases are notorious in MS systems because the ligands are easily ionised and thus detected. Traditional C18 ligands also suffer from column bleed, but the ligands are not easily ionised, and hence not detected in positive ion mode. This fuelled research aimed at developing LC-MS compatible EPG phases.

In the mid-1990s pure type B silica gel became readily available. Made from organo-silicates rather than inorganic silicates, they have a homogenous and lower activity silica surface and are free of metal ions. Overcoming irreproducibility was one of the main concerns in the HPLC market at this time. Pure silicas provided demonstrable improvement in the reproducibility of bonded phases made from them. The first Supelco EPG phase to take advantage of this advance in silica and subsequent reduction in variability was Discovery® RP-Amide, introduced in 1997.

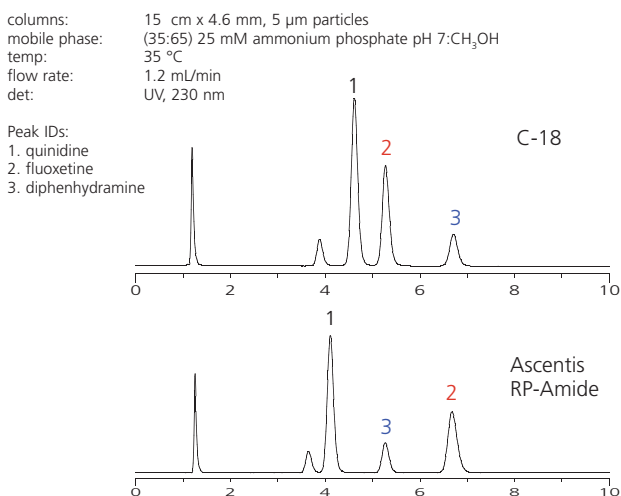
## Ascentis RP-Amide: High Retentivity and Low Bleed Optimized for LC-MS

LC-MS continued to be the driver for HPLC bonded phase development in the new millennium. With LC-MS instruments becoming more and more sensitive and affordable to nearly every laboratory, Supelco R&D efforts focused on two important column-related criteria: further reduction of bleed and increased retentivity, especially of polar compounds. High retentivity is an advantage in LC-MS because it allows for higher organic mobile phases which desolvate more rapidly than aqueous mobile

phases and lead to higher analyte ion sensitivity. High retentivity also has advantages in preparative LC, allowing higher sample loads.

In 2004, Supelco introduced Ascentis™ RP-Amide to address both of these challenges (Figure 2). Two advances facilitated its development: a new high purity, high surface area silica gel, and the use of a newly developed amide ligand. This special ligand, with a proprietary bonding scheme, creates a uniform phase network, which significantly enhances phase stability while preserving efficiency and reproducibility. This results in lower column bleed as well as the ability to work at low pH ranges for an extended time as compared to previous generation EPG phases (3).

**Figure 2.** Selectivity differences between C18 and Amide-based HPLC phases (note elution order reversal)



**Future Direction**

The Ascentis line is the culmination of nearly three decades of HPLC innovation at Supelco. Few other manufacturers can claim such longevity and consistency.

So what's next from the HPLC innovators at Supelco? We will continue to monitor the performance of our materials as they are used in more and more demanding applications. Trends in HPLC instrument design, including hyphenation, new detection methods and increased speed and sensitivity requirements will no doubt be on our radar. We also rely on our customers to give us ideas on where we can improve and what are the analytical challenges we can design phases to meet.

**References**

- (1) Ascah, T. L.; Feibush, B. J. *Chromatogr.* **1990**, 506, 357-389.
- (2) Ascah, T. L.; Kallury, K. M. R.; Szafranski, C. A.; Corman, S. D.; Liu, F. J. *Liq. Chromatogr. Rel. Technol.* **1996**, 3049-3056.
- (3) Ascentis Applications CD

**Product Table**

Prod. No.	Particle Size (µm)	ID (mm)	Length (cm)	Pack Size
<b>SUPELCOSIL ABZ+Plus HPLC column</b>				
59194C30	3 µm	3.0	15 cm	1 EA
57926	5 µm	2.1	15 cm	1 EA
<b>Discovery RP-AmideC16 HPLC Column</b>				
50501321	5 µm	2.1	15 cm	1 EA
505013	5 µm	4.6	15 cm	1 EA
<b>Ascentis RP Amide HPLC Column</b>				
565306-U	5 µm	2,1	25 cm	1 EA
565325-U	5 µm	4,6	25 cm	1 EA

Although newer technology exists, these early EPG phases are still widely used and are still available today from Sigma-Aldrich. Other dimensions and phases available. For a complete list of Supelco's EPG phases, call or visit our website [www.sigma-aldrich.com/supelco](http://www.sigma-aldrich.com/supelco).

For more information on Ascentis, please visit our website: [www.sigma-aldrich.com/ascentis](http://www.sigma-aldrich.com/ascentis)

**i** Information Request .....2101, 2102

## Request the Ascentis HPLC Applications CD

We have compiled over 200 applications showing the use of our C18, C8, and RP-Amide HPLC columns. Upon receipt of your request, we will send you a CD with valuable applications and technical information. (Code HNU)

# Elevated Performance

*"One Step" at a time*

RP-Amide  
C18  
C8



**Ascentis™**  
HPLC Columns from Supelco

Supelco has taken another step in performance and value with Ascentis RP-Amide columns. The amide functionality is the most popular of all Embedded Polar Group (EPG) phases and has resulted in the first EPG phase recognised with a USP code designation (L60). As an innovator in EPG bonding chemistry, Supelco has moved ahead of the competition again.

#### Supelco's most recent advances in bonding chemistry have resulted in:

- Better reproducibility for easy method validation
- Lower bleed for LC-MS applications providing better sensitivity
- Excellent selectivity for polar compounds increasing resolution and reducing coelution

#### Service before and after the sale

Our Technical Service Department will gladly share our knowledge and experience with you in the selection of HPLC columns and accessories, and provide detailed technical assistance for your chromatographic techniques.

**For more information** on Supelco's Ascentis HPLC columns, contact our technical experts at [EurTechServ@europe.sial.com](mailto:EurTechServ@europe.sial.com) or visit our website at [sigma-aldrich.com/ascentis](http://sigma-aldrich.com/ascentis)

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LABORATORY ESSENTIALS

DRUG DISCOVERY

## HPLC ARTICLE

# Effectively Managing Your Laboratory Time and Costs – Automated Solvent Recovery and Mobile Phase Degassing

When performing HPLC, frequently large volumes of special grade solvents are consumed and wasted, although most of the mobile phase may still be clean could be recovered and reused. Thus solvent consumption is reduced, mobile phase preparation time is saved, the environment, as well as money.

### The Solvent Recovery Systems

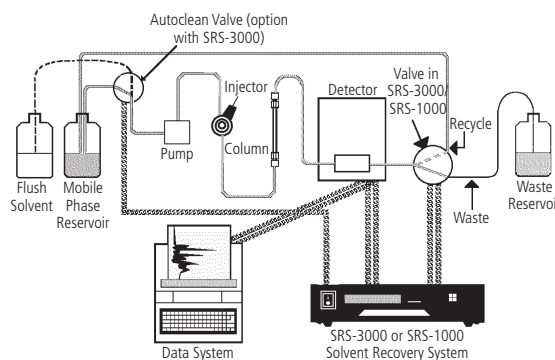
For automated retrieval of clean mobile phases during HPLC runs, the Supelco SRS-3000 and SRS-1000 Solvent Recovery Systems (Figure 1) can save money and time in any isocratic analysis. A microprocessor-controlled solvent switching valve monitors detector output and directs the solvent to the waste reservoir only when a peak is detected (Figure 2). When the baseline falls below the threshold you select, the

Both systems are ready-to-use and include a control unit with switching valve, a power cord, a 2-lead signal cable (+/-), Teflon tubing and fittings, and an instruction manual.

In addition to these components, the SRS-3000 system with the Autoclean valve has the wash valve, additional tubing and fittings, a wash start cable and a pump remote stop cable.

The SRS-3000 and SRS-1000 units meet all CE requirements; the SRS-1000 units also meet UL and CSA requirements.

Figure 2. System's Installation and Set-up



1. Connect SRS-3000 or SRS-1000 unit to detector signal output (cable is included).
2. Connect SRS-3000 or SRS-1000 unit to mobile phase and waste reservoirs and detector (Teflon tubing is included).
3. Set the threshold value and begin saving time and money.

Figure 1. The Supelco SRS-3000 (right) and SRS-1000 (left) Solvent Recovery Systems



uncontaminated solvent is directed back to the mobile phase reservoir. In a typical isocratic analysis, 80-90% of the mobile phase is uncontaminated and can be recycled (Figure 3). Settings for threshold, detection range, and delay time enable you to precisely control the switching valve.

In addition to the basic features mentioned above, the SRS-3000 unit offers validation output (included), an Autoclean option (see below), and storage for up to 10 method files. The validation output provides a continuous, auditable data trail of the solvent recycling valve position, for GMP, GLP, or ISO-9000 protocols. The valve position is recorded by superimposing tick marks over separate copy of the chromatographic signal.

### Autoclean Valve

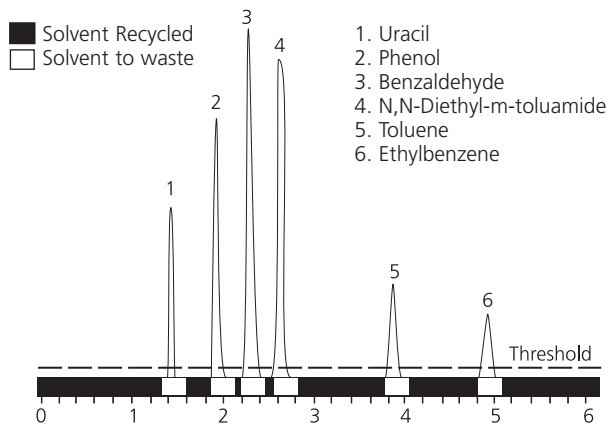
The SRS-3000 system also is available with valve that enables you to select a different solvent to flush the HPLC system. The Autoclean valve is especially useful if you are using a single pump with mobile phases containing buffer or other salts. The Autoclean valve installs between the mobile phase reservoir and the pump. It has two inlet lines, one for the mobile phase and one for the wash solvent. The valve can be factory installed, or you can order it separately and install it yourself.

### Economy-Priced Unit

The economically priced SRS-1000 includes the same solvent-saving features as the SRS-3000 unit. A simpler display and no advanced features (no validation output, Autoclean option, or method storage memory) allow us to keep the price substantially lower.

Figure 3. Function Scheme

Recovery of 80% or more of the mobile phase used in an isocratic HPLC analysis is possible.



### Supelco Mobile Phase Degassing System

Degassing of mobile phases can be tedious and time consuming, in particular in method development and routine analysis. The Supelco Mobile Phase Degassing System (Figure 4) with its Smart Sensor not only detects and alerts personnel to leaks, it also communicates with the vacuum pump. If a change in vacuum is detected due to mobile phase flow rate changes, the pump can compensate by changing its speed. The validation output records vacuum level to satisfy system compliance and validation requirements. The degassing system has a Teflon® AF membrane, with NO-OX™ fittings and tubing. Furthermore the Supelco Mobile Phase Degassing System is a four channel apparatus with a flow range of 0-5 mL/min. The degassing status is LED indicated. The Apparatus has a CE mark and an unprecedented four years warranty.

#### Ordering information

Prod. No.	Description	Pack Size
57431	SRS-3000 Solvent Recovery System	1EA
57432	SRS-3000 Solvent Recovery System, autoclean	1EA
55018-U	Mobile Phase Degassing System	1EA

Figure 4. Supelco Mobile Phase Degassing System



## TLC

### Thin Layer Chromatography – Did you know that Sigma-Aldrich sells Merck TLC plates?

Thin layer chromatography is one of the oldest and very simplest chromatographic technique. It is used for assessment, identification, purity check and for scouting of method parameters for flash chromatography. Sigma-Aldrich offers our own brand of plates as well as from other known manufacturers. One of the most well known manufacturers is Merck, however what is not so well known is that these plates can be purchased from Sigma-Aldrich. Please see below for our offering of glass plates coated with 60Å pore silica. These plates incorporate a charrable organic binder.



#### Ordering information

Cat. No.	Adsorbent Layer	Plate Dimensions (cm x cm)	Layer Thickness (µm)	Particle Size (µm)	Fluorescence Indicator <sup>1</sup>	Qty.
<b>Silica or Silica/C18 On Glass</b>						
Z292966-1PAK	Silica gel 60	10 x 20	250	5-20	no	50
Z292974-1PAK	Silica gel 60	20 x 20	250	5-20	no	25
Z292982-1PAK	Silica gel 60	2.5 x 7.5	250	5-20	yes	100
Z292990-1PAK	Silica gel 60	5 x 10	250	5-20	yes	200
Z293008-1PAK	Silica gel 60	5 x 20	250	5-20	yes	100
Z293016-1PAK	Silica gel 60	10 x 20	250	5-20	yes	50
Z293024-1PAK	Silica gel 60	20 x 20	250	5-20	yes	25
Z293032-1PAK	Silica gel 60/C18	20 x 20	250	5-20	yes	25

<sup>1</sup> Manganese-activated zinc silicate.

Please refer to our web site for further TLC plates and products.

# VersaFlash<sup>TM</sup>

## High Throughput Flash Purification

### It's in the particles!

Irregular silica particles are most commonly used as packing materials for Flash Chromatography. However, compared to spherical particles, irregular shaped particles pack in a less uniform manner and give lower column efficiency. Irregular particles also generate fines that can cause changes in bed density, channeling and band broadening, in addition to possibly contaminating preps and fouling the system. Broad sample bands give rise to dilute sample fractions, which consume more solvent and take longer to rotovap.

VersaPak cartridges eliminate the problems associated with irregular particles. Packed with spherical particles, VersaPak cartridges have a denser, more evenly-packed bed. Fines are eliminated as a source of channeling, fouling and band-broadening. Sample components elute in sharper bands and more concentrated fractions reducing solvent consumption and evaporation time. The denser bed in VersaPak cartridges also gives higher sample capacity compared to the same volume packed with irregular particles.

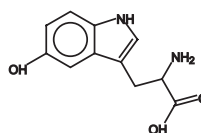
### The savings are in the particles:

- Save Solvent – by more concentrated bands/fractions
- Save Time – by faster more efficient separations with less rotovap time
- Save Money – use smaller cartridge sizes and less solvent

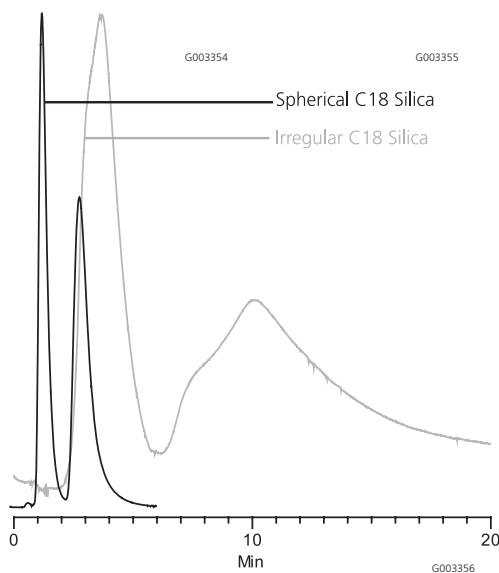
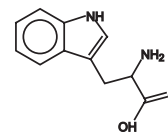


Improved efficiency on cartridges packed with spherical vs. irregular silica particles

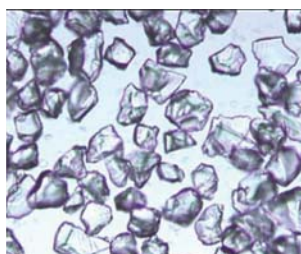
5-hydroxy-DL-tryptophan



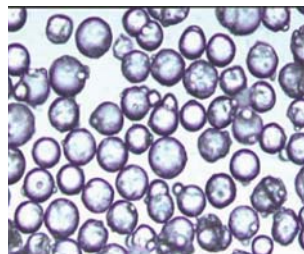
DL-tryptophan



Irregular Silica



Spherical Silica



The VersaFlash High Throughput Flash Purification system is a versatile purification system that can cover a broad range of sample sizes. Cartridge sizes range from 11g (23mm ID) to 1900g (110mm ID) that can be used on the same system.

For further information on VersaFlash products, please request the VersaFlash-High-Throughput Flash Purification Brochure (FWL).

**VERSAFLASH<sup>TM</sup>**  
Pure.... & Simple



**i** Information Request ..... 2103

## SPE ARTICLE

## Controlling SPE Selectivity Through pH and Organic Modifier Manipulation

An Trinh Product Manager Sample Prep (SPE), Laura Marlatt, David S. Bell Ph.D. Applications Group Manager

## Introduction

Solid phase extraction (SPE) methods are frequently developed by copying/modifying an existing application or choosing a generic method. Although these approaches are less time-consuming, it can often be very difficult to determine and troubleshoot the root cause(s) of problems associated with an SPE method when they arise. For example, if poor recovery is observed, is it due to: 1) poor analyte retention during sample load; 2) pre-mature analyte elution during the wash step (b/w sample load and elution); or 3) analyte over retention during elution?

Like HPLC, SPE is a form of chromatography, and as such, basic chromatographic principles should be used when developing, optimising, and troubleshooting a given SPE method. In this report, we demonstrate the use of pH and organic modifier manipulation during SPE wash/elution to control the retention and elution of three different compounds (neutral, basic, and acidic) on three different reversed-phase SPE chemistries of decreasing hydrophobicity (C18, C8, and cyanopropyl-CN).

## The Role of pH and Organic Modifiers in SPE

Most reversed-phase SPE protocols follow the general procedure in which the phase is first conditioned and equilibrated with aqueous miscible solvent (e.g., methanol or acetonitrile) followed by sample load. The sample must be aqueous because a polar mobile phase environment is necessary to drive reversed-phase retention. To elute compounds of interest, reversed-phase interactions are disrupted by decreasing polarity of the mobile phase environment. Common elution solvents include methanol and acetonitrile. Prior to elution, a wash step of intermediate solvent strength is typically employed to remove any endogenous interferences that may have co-retained with the analytes of interest (e.g., 5-20% methanol).

Most analytes contain ionisable functional groups, and a compound's ionisation state can drastically change its retention and elution characteristics on a given SPE sorbent. When an analyte is in its neutral form, it becomes more hydrophobic and retention strengthens under reversed-phase conditions. This may allow for stronger wash solvents to remove co-retained interferences prior to elution. In contrast, in the ionised form, compounds become more polar, weakening the interaction strength between analytes of interest and reversed-phase functional groups. As a result, one may be able to elute with weaker solvent conditions (e.g. 50% methanol as opposed to 100% methanol) which could possibly eliminate the evaporation/reconstitution step common in SPE protocols. Figure 1 describes the role of pH in SPE.

## Method

1 mL standards of 20 µg/mL ibuprofen (acidic), hydrocortisone (neutral), and alprenolol (basic) in 20 mM potassium phosphate, pH 7 were loaded on to three different 96-well SPE plates conditioned and equilibrated with 1 mL methanol and DI water per well. The SPE phase chemistries tested were Discovery DSC-18 (C18), DSC-8 (C8), and DSC-CN (cyanopropyl), 100 mg/well.

Figure 1. The Role of pH in Reversed-Phase SPE

Acids (e.g. Carboxylic Acids):  $R-COOH \rightleftharpoons R-COO^-$ 

HA (neutral)	$\rightleftharpoons$	H <sup>+</sup> + A <sup>-</sup> (ionized)
50%	@ pK <sub>a</sub>	50%
100%	2 pH units below pK <sub>a</sub>	0%
0%	2 pH units above pK <sub>a</sub>	100%

Bases (e.g. Amines):  $R-NH_3^+ \rightleftharpoons R-NH_2$ 

BH <sup>+</sup> + OH <sup>-</sup> (ionized)	$\rightleftharpoons$	B (neutral)
50%	@ pK <sub>a</sub>	50%
0%	2 pH units below pK <sub>a</sub>	100%
100%	2 pH units above pK <sub>a</sub>	0%

Neutral State (Blue) = Strengthens reversed-phase interaction

Ionized State (Red) = Weakens reversed-phase interaction

Respective wells were washed/eluted with 1 mL test solvents ranging from 0-100% methanol in 2% NH<sub>4</sub>OH, pH11 (high pH), DI H<sub>2</sub>O (neutral pH), and 2% CH<sub>3</sub>COOH, pH 3 (low pH). The wash/elute eluate was collected for each well, and analysed for compound breakthrough via HPLC-UV.

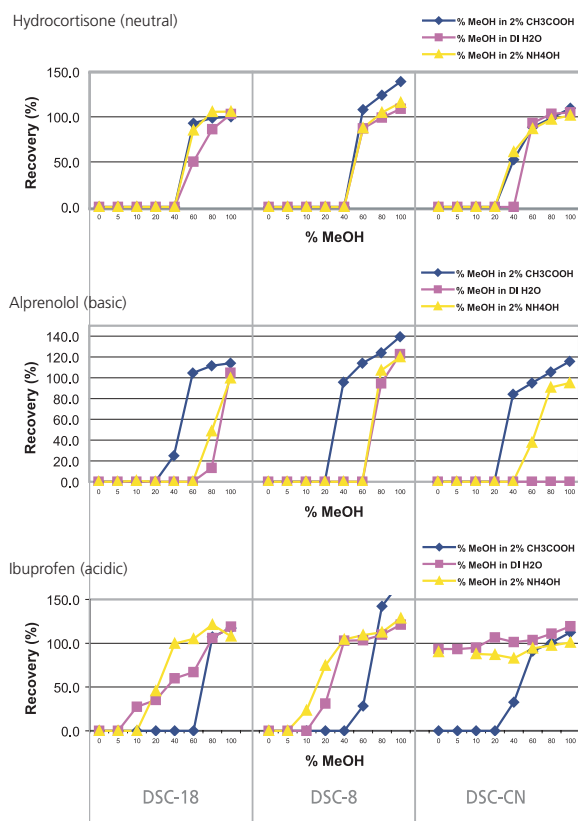
## Retention-Elution Profile for Hydrocortisone (neutral)

Figure 2 represents a retention-elution profile of the three compounds tested in which % recovery was measured against changing extraction conditions (pH vs. % organic modifier vs. phase chemistry). Hydrocortisone is a neutral compound that contains no ionisable functional groups. In Figure 2, we see that changes in pH across all three SPE chemistries had very little effect in manipulating elution selectivity. Up to 40% methanol can be used as a possible wash solvent for both DSC-18 and DSC-8. DSC-CN is much more polar reversed-phase SPE chemistry. As a result, analyte breakthrough occurs between 20-40% methanol. 100% methanol is required to completely recover this moderately polar to non-polar compound.

## Retention-Elution Profile for Alprenolol (basic)

Alprenolol is a basic compound with a pK<sub>a</sub> of ~9.5. At higher pH levels, it deprotonates into its neutral form. At low pH levels, it is in its ionised form. In contrast with hydrocortisone, pH modification has a great effect in controlling selectivity. At neutral and high pH conditions, alprenolol can withstand up to 60% methanol on DSC-18 and DSC-8 SPE before compound breakthrough occurs. At low pH conditions, compound breakthrough occurs at greater than 20% methanol. On DSC-CN, the short alkyl functional groups allow greater compound access to silanol groups which act as a secondary weak cation exchanger. As a result, at neutral pH conditions compounds are retained by both cation exchange and reversed-phase, and compounds remain retained from 0-100% methanol. At high pH conditions, alprenolol is in its neutral form disrupting secondary ionic interactions allowing

**Figure 2.** Retention-Elution Profile Hydrocortisone, Alprenolol and Ibuprofen on DSC-18, DSC-8 and DSC-CN SPE



**Conclusion**

Both pH and % organic modifier play a critical role in determining retention and elution of ionisable compounds in reversed-phase SPE. By controlling the pH of the SPE mobile phase, one can control the relative hydrophobicity of an ionisable compound allowing for stronger wash solvents resulting in improved sample cleanup. pH manipulation may also allow for weaker elution solvents possibly minimising processing time by negating the eluate evaporation/reconstitution step common in most reversed-phase procedures. By understanding how a compound interacts with the SPE phase under changing extraction conditions, one can manipulate the conditions to offer the most selective procedure.

**For more information, please request T404049 (GUX). This information is available in electronic form only. Be sure to include your email address on the request form.**

**If you are interested in a sample pack of our SPE tubes, please contact our technical service EurTechServ@europe.sial.com or sales representative.**

**For a complete listing of the SPE products please refer to the Sample Preparation/Purification chapter of the Supelco catalogue.**



**i** Information Request ..... 2104

for elution from 40-100% methanol. At low pH, alprenolol is ionised but silanol groups are protonated and neutral resulting in elution between 20-100% methanol.

**Retention-Elution Profile for Ibuprofen (Acidics)**

Ibuprofen is an acidic compound with a  $pK_a$  of ~4.2. In contrast to alprenolol, the compound is neutralised at low pH and ionised at high pH environments. On DSC-18 and DSC-8, up to 60 and 40% methanol in 2% acetic acid can be used as a possible wash solvent. At neutral and high pH levels where the compounds are ionic and thereby more polar, the retention limit is 5-10% methanol before compound breakthrough occurs. On DSC-CN, retention is very weak at high and neutral pH, and buffer alone will elute the compound. At low pH levels, wash solvents of up to 20% methanol can be employed.

**Note on High Recoveries**

Note that >100% recovery was often observed. When injecting a sample of greater solvent strength than the HPLC mobile phase, fluctuations in retention time and peak shape are often observed (data not shown) which can result in erroneously high signals. We observed this trend in our data because the SPE eluate was directly analysed, and a high level of % methanol was used during SPE elution in part of the study. Although recovery data was not accurate, the purpose of the data was to describe general recovery trends observed by systematically changing elution conditions.

## SPME TROUBLESHOOTING

### Tips to Optimize Extractions by SPME

The Solid Phase Microextraction (SPME) is an innovative, solvent free technology that is fast, economical and versatile. SPME has gained wide spread acceptance as the technique of preference for many applications. As with any analytical process however, problems occur on occasion. The most important step in correcting a problem when it occurs is identifying the root cause of the problem without wasting time. The systematic approach to troubleshooting described in the "SPME Trouble shooting Guide" will allow a quick solution to many problems. The guide contains helpful tips to prevent problems before they occur, as well as a troubleshooting table listing the symptoms of the common problems, the possible causes and suggested remedies. By following these recommendations, one can save valuable time and money. Here are some sample chapters from the guide:

#### Troubleshooting Suggestions

Make troubleshooting faster and easier by closely observing and keeping complete records of your analytical conditions. Understanding the system performance history, as related to the fiber, sampling, desorption, inlet, column, detector response, etc., is important for effective troubleshooting. Thorough documentation of system maintenance (fiber changes, inlet liner changes, etc.) are equally helpful and important when determining what variables have changed and when. Troubleshooting is more effective when you have on hand the following items:

- New backup fibers
- New backup column
- Pre-tested or control fiber with known performance
- Pre-tested or control column with known performance
- Spare injection port septa and liners
- Spare sampling vials and septa
- All associated product instruction sheets and instrument manuals

#### Isolating the Problem Source Establish a Systematic Approach

Carefully note the symptoms you are encountering (e.g., no peaks detected, extraneous peaks detected, etc.), then find these in the troubleshooting table in the guide (Figure 1). Next to each symptom is listed several possible causes for what is being observed. Review the possible causes and systematically address each one through the remedy listed. Start your elimination process with the most probable cause based upon your specific situation, then work through the remedies systematically. A

shotgun approach to applying the remedies, while seeming to be the fastest approach, is usually the least effective. Understanding the cause and effect relationship with the changes made and determining the actual root cause of the problem is the most beneficial and effective approach. The troubleshooting table contains most of the problems you will encounter with SPME, but we cannot anticipate every situation or application. If you experience a problem not covered in the table, you can still determine the cause and remedy by systematically isolating the problem into one of four areas: sampling, desorption, analysis, or product. The following is a general scheme for isolating the problem:

**Step 1:** Eliminate the sampling, desorption, and SPME fiber from your analytical process by directly injecting a known reference standard containing your compounds of interest. This will give you an analysis focused test that provides valuable data on the chromatographic system's performance. If the results show the problem persists, then focus your attention on the injection port, column, or detector (maintenance, replacement, etc.). If the problem disappears and your normal operating performance returns, then go on to step two.

**Step 2:** Next, eliminate the sample matrix from the process by sampling a clean matrix (reagent water or sand) spiked with a known reference standard. Sample this control with the identical sampling conditions used previously.

**Step 3:** Analyse the sampled fiber under the identical desorption and instrument conditions used previously. If the results show the problem persists, then proceed to step four. If the problem disappears, you have demonstrated that the SPME fiber and desorption process are not the likely causes of the problem. The most likely cause of the problem is the sample matrix and its effect on the equilibrium or fiber. Experiment with your sampling conditions (headspace vs. immersion, time, temperature, pH, salt, agitation, etc.) to determine the optimal parameters for that





# Exactly Why is Low Bleed Important for Capillary Columns?

Michael D. Buchanan Product Manager, Gas Chromatography

## Abstract

Capillary column manufacturers often use the term 'low bleed' to describe their columns. How exactly does 'low bleed' relate to the needs of end-users? Low detection limits, positive mass spectral identifications, and instruments that are not down for maintenance are all important daily goals for a GC analyst. Using a capillary column, such as Supelco SLB-5ms, that has a low bleed characteristic is an important step in achieving these goals.

## Low Bleed = Better Signal-to-Noise Ratio = Lower Detection Limits

Signal refers to the response from an analyte passing through a detector. It is the peak you see when looking at a chromatogram. System noise refers to everything else, other than the analyte, producing a response in the detector. It is the baseline on a chromatogram. The ratio of the peak height to the baseline variability is termed the signal-to-noise ratio.

High noise level is undesirable since it makes it difficult for the integration software to adequately measure all of the peak area. The better the signal-to-noise ratio, the more area counts are obtained, resulting in the ability to achieve lower detection limits. Figure 1 shows a graphic illustration of a peak when column bleed (a significant source of system noise) is low vs. high.

Analysts rely on GC-MS and other GC methods to provide highly sensitive, low-level detection. When measurement is required at the ppb or even ppt level, extreme care must be taken to ensure that nothing interferes with the analysis. For this reason, today's chemists require capillary columns, such as Supelco SLB-5ms, that exhibit a very low level of bleed in addition to being inert towards the various analytes in the method.

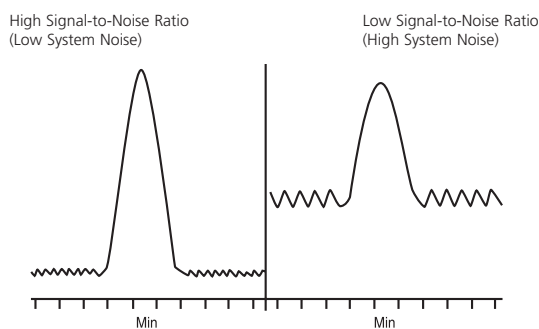
## Low Bleed = Cleaner Mass Spectra = Easier Mass Spectral Identifications

Analysts using MS detection have an additional concern: high column bleed that can interfere with proper mass spectral identification of analytes. In addition to the primary, or quant ion < in a mass spectrum, MS programs are set to measure the abundance of other ions that are characteristic to the analyte of interest. For the software to assign a high probability of positive identification, these so-called secondary, or qualifier ions must be within specific ratio ranges relative to the primary ion when the mass spectrum from a peak in a realworld sample extract is compared to a mass spectral library entry. If extraneous ions, such as those column bleed, are present in the mass spectrum, the software will assign a lower probability of positive identification.

Additionally, many US EPA methodologies require GC-MS analysts to assign tentative identification to non-target "unknown" compounds which may also be present in the sample extract. These are called Tentatively Identified Compounds, or TICs. In order for the software to assign a high probability of positive identification, the mass spectrum from the sample extract must compare well with the mass spectral library entry. High column bleed levels can interfere with this comparison, resulting in the reporting of TICs that are either poorly identified or misidentified.

Figures 2 and 3 show mass spectra of benzo(g,h,i)perylene at a 5 ng on-column level and an oven temperature of 325 °C. The major column bleed ion,  $m/z = 207$ , resulting from the formation

Figure 1. Illustration of Signal-to-Noise Ratio



of hexamethylcyclotrisiloxane (D3), should be present at a level lower than  $m/z = 138$  and  $m/z = 277$ , two secondary ions used to confirm the identity of the peak. Figure 2 is from a Supelco SLB-5ms column. The major bleed ion,  $m/z = 207$ , is lower than both of the secondary ions. Figure 3 is from a Competitor "A" column. The major bleed ion is actually larger in size than the two secondary ions.

What does all this mean? The MS software would assign a low probability of positive identification for benzo(g,h,i)perylene for analyses using the Competitor "A" column. For TICs, the situation would be less desirable since retention time data would not be available to assist with identification.

## Low Bleed = Reduced Detector Contamination = Less Instrument Downtime

High column bleed can foul the GC detector, reducing detector sensitivity. When the fouling becomes severe enough to warrant action, the detector must be dismantled and cleaned, a procedure that may remove an instrument from service for one or more days. MSD lenses can become coated and require polishing. The active foil in an ECD can become fouled to the point the entire detector needs to be sent out for refurbishing. Partially plugged FID jets need to be replaced. PID windows can become layered with contamination and require cleaning.

The more instrument downtime you experience, the fewer billable samples you will be able to run in a given period of time. When you have a backlog of analyses to perform, you simply cannot afford unnecessary instrument downtime.

## Conclusion

Low detection limits, positive mass spectral identifications, and instruments that are not down for maintenance are all important daily goals for a GC analyst. Using a capillary column, such as Supelco SLB-5ms, that has a low bleed characteristic is an important step in achieving these goals.

Figure 2. Mass Spectrum from Supelco SLB-5ms Column

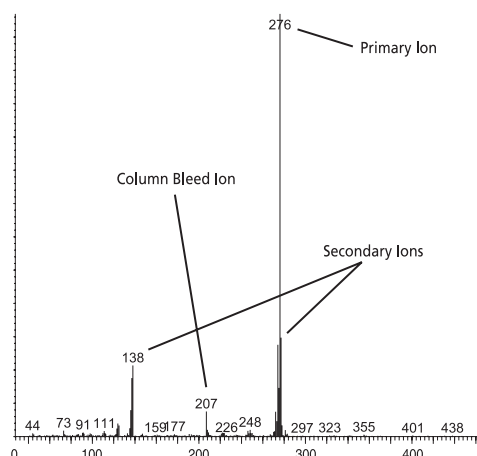
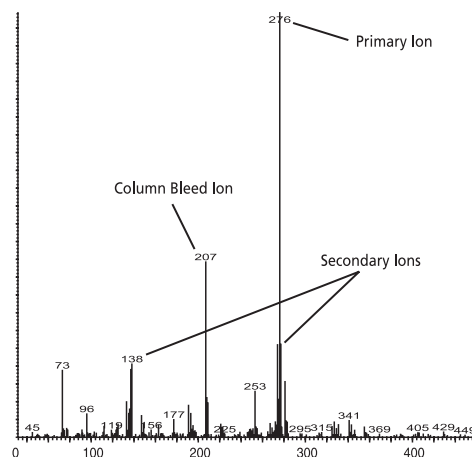


Figure 3. Mass Spectrum from Competitor "A" Column



### Special Purpose SLB-5ms Capillary GC Columns

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

Temp. Limits: 0.20 to 0.32 mm I.D. columns:  
-60 °C to 340 °C (isothermal) / 360 °C (programmable)

#### Ordering information

Prod No.	Description	Length (m)	D <sub>i</sub> (µm)	Beta
<b>0.20 mm ID</b>				
28513-U		30	0.2	250
<b>0.25 mm ID</b>				
28467-U		30	0.25	250
28469-U		15	0.25	250
28471-U		30	0.25	250
28472-U		60	0.25	250
28473-U		30	0.5	125
28476-U		30	1	63
<b>0.32 mm ID</b>				
28557-U		15	0.25	320
28482-U		30	0.25	320
28532-U		30	0.32	250
28484-U		30	0.5	160
28487-U		30	1	80

### Extend Column Life With Guard Columns

A decrease in peak shape quality in a capillary GC system can typically be traced to the inlet end of the column. Over time, the inlet end of the column becomes contaminated from an accumulation of non-volatile material. The phase can also be damaged from the continuous condensation and vaporisation of solvent and analytes. Inevitably, active analytes will adsorb to the contaminated / damaged section, leading to peak tailing, loss in resolution, and reduced response. When chromatography degrades to an unacceptable level, performance is restored by clipping the contaminated / damaged section off the inlet end of the column.

To extend the lifetime of capillary GC columns, Supelco recommends using a 3 m long guard column. A guard column is a short piece of uncoated deactivated fused silica tubing which is placed in-line between the GC injection port and the analytical column. The guard column will take the brunt of the contamination / damage. By clipping the guard column periodically to restore performance instead of the analytical column, the analytical column remains unaltered. Therefore, chromatography (retention times and resolution) is not affected.

### Fused Silica Guard Columns

For use as guard columns to protect your analytical column from damaging sample components. Match the deactivation of the tubing with the polarity of the injection solvent.

Deactivation	Injection Solvents	Max. Temp
Non-Polar	Alkanes, Carbon disulfide, Ethers	360 °C
Intermediate Polarity	Acetone, Methylene chloride, Toluene	360 °C
Polar	Acetonitrile, Methanol, Water	260 °C

#### Ordering information

Prod No.	Length (m)	ID
<b>Non-Polar Deactivation</b>		
25722	3	0.25
25742	5	0.25
25723	3	0.32
25743	5	0.32
<b>Intermediate Polarity Deactivation</b>		
25727	3	0.25
25747	5	0.25
25728	3	0.32
25748-U	5	0.32
<b>Polar Deactivation</b>		
25752-U	5	0.32

### Capillary Column Butt Connector

This device consists of a double-tapered ferrule and a stainless steel compression housing with a threaded cap. Small and light (2.3 cm x 0.6 cm, 4.4 g with ferrule), it provides a gas tight seal. This unit maintains inertness with no change in column efficiency.

#### Ordering information

Cat. No.	Description
23804	Capillary Column Butt Connector, body only
22453	Supeltex M-2B Ferrules, pack of 2 To connect 0.10/0.25 mm ID to 0.10/0.25 mm ID
22454	To connect 0.32 mm ID to 0.32 mm ID

For a complete listing of Supelco Low Bleed SLB-5ms capillary columns, visit our web site [sigma-aldrich.com/capillary-ms](http://sigma-aldrich.com/capillary-ms)

Information Request ..... 2107



SGT Super Clean™ Gas Purifiers Bob Wallace Product Specialist**Introduction**

Brief exposure to contaminated carrier gas can cause serious damage to GC capillary columns. Exposure to contaminants such as oxygen, moisture, and hydrocarbons can reduce performance and decrease column life. To prevent the irreversible degradation of the column, the GC system must include a gas management system designed to eliminate contaminants.

Supelco now offers the unique Click-On Inline Super Clean Gas Purifiers and Super Clean Gas Filters with Fast-Connect technology by SGT. When used as part of a comprehensive gas management system, these state-of-the-art purifiers eliminate costly instrument downtime.

**Click-On Inline Super Clean Gas Purifiers**

Changing out a traditional inline purifier allows the open gas line to be exposed to unpurified room air, requiring a lengthy and unproductive flushing process. Getting dirt in the threads or over-tightening of gas line fittings can prevent proper sealing, providing a source for gas contamination. The Click-On Inline Super Clean Gas Purifiers have none of those disadvantages. They are 100% diffusion-proof and are made from glass and metal. They can be used for purification of carrier gas and fuel gases for your GC or GC-MS system. Reduction of hydrocarbons, oxygen, and moisture to produce better than 6.0 gas quality (99.9999% purity) can be met independent of the input quality of the gas. Click-On Inline Super Clean Gas Purifiers are available with or without visual indicators.



E000937

In sharp contrast to traditional inline purifier replacement, change out of the SGT Click-On Inline Super Clean Gas Purifiers is simple and eliminates the risk of damaging compression fittings. Once the Click-On connectors are installed into the gas line, those connections never need to be broken. Subsequent replacement of purifiers can be done in seconds. The new purifier is snapped in place and the fittings are hand tightened. Each Click-On connector contains a needle valve that snaps shut the instant the purifier is removed. This prevents introduction of room air into the gas line and allows the change out without shutting down the GC system. Click-On connectors are available in either brass or stainless steel with either 1/8" or 1/4" compression fittings.

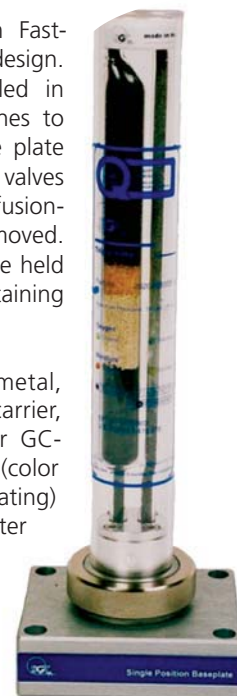


Fitting set for Click-On Inline Purifiers

**SGT Super Clean Gas Purifiers**

SGT Super Clean Gas Purifiers with Fast-Connect technology use a base plate design. Replacement purifiers can be installed in seconds without exposing the gas lines to room air. The specially designed base plate rests on the bench top and has needle valves that instantly close to provide a diffusion-proof seal when the gas purifier is removed. Replacement gas purifiers need only be held in place on the base plate while the retaining ring is hand tightened.

The unique point-of-use glass/metal, Super Clean Gas Purifiers purify carrier, fuel and other gases for your GC or GC-MS system. Hydrocarbons, oxygen (color indicating) and moisture (color indicating) are removed to produce gas that is better than 6.0 (99.9999%) gas quality. This purification is independent of the original gas quality.



E000938

**SGT Super Clean LC-MS Gas Purifiers**

Super Clean Gas LC-MS Purifiers, a revolution in clean gas purifiers, purify nitrogen from a nitrogen generator for LC-MS systems. These point-of-use glass/metal, diffusion-proof purifiers can remove hydrocarbons to produce nitrogen that is better than 6.0 gas quality, independent of the original gas quality.



E000939

The Super Clean Fast-Connect design avoids MS source damage and helps to eliminate LC-MS downtime. The two cartridge purifier systems, used in parallel (Figure 1), is the only fully glass/metal cartridge purifier capable of purifying LC-MS nitrogen carrier gas for all major hydrocarbon contaminants. The activated charcoal bed of the indicating hydrocarbon filter adsorbs organic compounds larger than ethane. The indicating material changes from bright yellow to dark green when the trap has reached saturation and needs to be replaced.

**For more information,** request SGT Super Clean LC-MS Gas Purifiers, T405137 (ILD), SGT Click-On Inline Super Clean Gas Purifiers, T405138 (ILE), and SGT Super Clean Gas Filters, T405139 (ILF).

This information is available in electronic form only. Be sure to include your email address on the request form.

**i** Information Request ..... 2108, 2109, 2110

SGT Super Clean Gas Purifier Specifications

Type of Purifier	H <sub>2</sub> O (g)	O <sub>2</sub> (mL)	Hydrocarbon (g)	Estim. Lifetime*
GC – Moisture	7.2	-	-	> 2 years
GC – Charcoal	-	1000	12 (as n-butane)	> 2 years
GC – Combi (moisture/charcoal)	-	-	6 (as n-butane)	> 1.5 years
GC-MS – Triple (moisture/oxygen/charcoal)	1.8	500	4 (as n-butane)	> 1 year
GC-MS – Triple : gas specific helium (moisture/oxygen/charcoal)	2.0	600	4 (as n-butane)	> 1 year

\* The specified lifetimes are strongly dependent on the quality of the incoming gas.

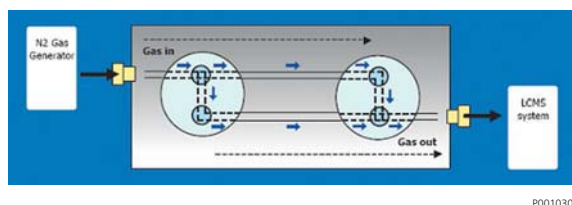
LC-MS Filter Adsorbents & Indicator Specifications

Compounds Removed: Hydrocarbons (> C2)  
 Capacity: 1.64 grams of Hexane  
 Efficiency: < 1ppb for the adsorbents;  
 < 3ppb for the indicator  
 Indicator Color: from Yellow to Green  
 Pressure Limit: 100 psig  
 Temperature Limit: 100 °C

Pressure Drop Specifications

At a flow of max. 20 L/min, the pressure drop is max. 2%

Figure 1. LC-MS Gas Purifier Flow Diagram



Ordering Information

Prod. No.	Description
<b>SGT Click-On Inline Super Clean Purifiers</b>	
28861-U	Click-On Moisture Inline Super Clean Trap
28862-U	Click-On Oxygen Inline Super Clean Trap
28863-U	Click-On Hydrocarbon Inline Super Clean Trap
28864-U	Click-On Triple O <sub>2</sub> /Moisture/Hydrocarbon Inline Super Clean Trap for Carrier Gas
28865-U	Click-On Gas Specific (He) Triple O <sub>2</sub> /Moisture/Hydrocarbon Inline Super Clean Trap
28866-U	Click-On Combi Moisture/Hydrocarbon Inline Super Clean Trap for Fuel Gas
28867-U	Click-On Indicating Triple O <sub>2</sub> / Moisture/Hydrocarbon Inline Super Clean Trap
<b>SGT Click-On Inline Super Clean Hardware</b>	
28868-U	Click-On Connector 1/4" Brass, Pk 2
28869-U	Click-On Connector 1/8" Brass, Pk 2
28872-U	Click-On Connector 1/4" Stainless Steel, Pk 2
28873-U	Click-On Connector 1/8" Stainless Steel, Pk 2
28874-U	Click-On Double Version Connector 1/8", Pk 2
28875-U	Click-On Replacement O-ring, Pk 10
28876-U	Wall Mounting Clip, Pk 4
<b>SGT Super Clean Gas Purifiers</b>	
SU861021	Ultra-high Capacity Moisture Super Clean Purifier with Indicator
SU861022	Ultra-high Capacity Oxygen Super Clean Purifier with Indicator
SU861023	Ultra-high Capacity Hydrocarbon (Charcoal) Super Clean Purifier w/o Indicator
SU861025	Combi GC Replacement (Charcoal/Moisture) Fuel Gas Super Clean Purifier w/o Indicator
SU861026	Triple GC Replacement (O <sub>2</sub> /Moisture/Charcoal) Super Clean Purifier
SU861027	Triple GC Replacement for Helium (O <sub>2</sub> /Moisture/Charcoal) Super Clean Purifier
SU861028	High Flow Special Moisture Super Clean Purifier, Pk of 2
SU861029	Charcoal Cartridges for LC, N <sub>2</sub> Purification Super Clean Purifier, Pk of 2
28877-U	High Flow Charcoal Super Clean Purifier for LC-MS
<b>SGT Super Clean Gas Kits</b>	
SU861040	GC-MS Super Clean Purifier Kit for Helium (Baseplate Plus One Triple Purifier)
SU861043	GC-FID Super Clean Purifier Kit, 3 Purifiers (1 Triple/Combi-Charcoal/Moisture)
SU861045	High Flow Special Moisture Super Clean Purifier Kit (2 Purifiers/Baseplate)
SU861046	2 Charcoal Purifiers, High Flow LC-MS Super Clean Kit, N <sub>2</sub> Purification w/o Indicator
28878-U	Triple GC Super Clean Purifier Kit for Carrier Gas, 1 position
<b>SGT Super Clean Gas Hardware</b>	
SU861011	Single Position Baseplate
SU861012	2-Position Baseplate for High Flow LC-MS
SU861013	3-Position Baseplate
SU861016	Wall Mounting Bracket
SU861050	Replacement O-rings for Super Clean Cartridges (10 Large/10 Small), Pk 20
28879-U	2-Position Baseplate for High Flow LC-MS

## STANDARDS &amp; REAGENTS ARTICLE

## GC Analysis of PBDE Flame-Retardant Compounds

By Roberto Ferrari, European Sales

Development Manager, and Katherine Stenerson, Senior Applications Chemist, Rainer Walz Ph.D., Product Manager Analytic Standards

**Supelco SLB-5ms low-bleed capillary columns and Fluka PBDE analytical standards combine for efficient, reliable analysis of these compounds that are of increasing environmental concern.**

Periodically, classes of compounds come under regulatory scrutiny: heavy metals, dioxins, pesticides and PCBs, for example. The attention is usually a result of published studies reporting toxicity, mass poisonings or wildlife kills, spills or site clean-up efforts. Gas chromatography (GC) is frequently used to monitor levels during the assessment and remediation processes, and in research aimed at developing safer alternatives.

Polybrominated diphenyl ethers (PBDEs) have recently become the focus of such scrutiny. These compounds are widely used as flame retardant additives in many consumer products including plastics, electronics, clothing, cushioning foams for furniture and automotive interiors. They slow ignition and rate of fire growth, allowing more time to escape from fires involving products to which they are added. According to the US EPA, world-wide demand in 2001 for two PBDE congeners, penta-BDE and octa-BDE, was estimated at 7,500,000 and 3,790,000 kg, respectively (US EPA, December 6, 2004 (69 FR 70404)).

Irrespective of the indisputable life- and property-saving value of these compounds, environmental studies indicate they exhibit persistence, toxicity and bioaccumulation. Because they are additives and not chemically bound within the product, PBDEs can and do leach into the environment (1, 2) and have been found everywhere, including household dust, landfill runoffs, ground and surface waters, sewage sludge, sediment, plants and animals, including humans. It is believed that their structural similarity to thyroid hormones is responsible for their wide spectrum of toxicity in laboratory animals, primarily developmental and neurological in nature (3, 4). As a result, the European community has changed its environmental regulations for the plastics industry, stating that the phase-out of all PBDEs in plastics, together with other toxic heavy metals such as lead, cadmium, mercury and hexavalent chromium, will commence on January 1, 2006. The US EPA proposed a rule in December of 2004 that as of Jan. 1, 2005, all manufacturers and importers would be required to notify the EPA at least 90 days in advance before starting the manufacture or import of certain PBDEs.

A well-defined analytical method is critical to the efficient monitoring of PBDEs and includes high quality PBDE standards and a separation and detection technique, which is most often GC-MS or GC-ECD. Sigma-Aldrich, as a leading supplier of chromatography consumables to the environmental market, answered this need by providing quantitative Fluka standards of the PBDE congeners and Supelco SLB™-5ms capillary GC columns. Figure 1 shows the separation of PBDEs on the SLB-5ms column. This mix contains fourteen of the most commonly found PBDE congeners.

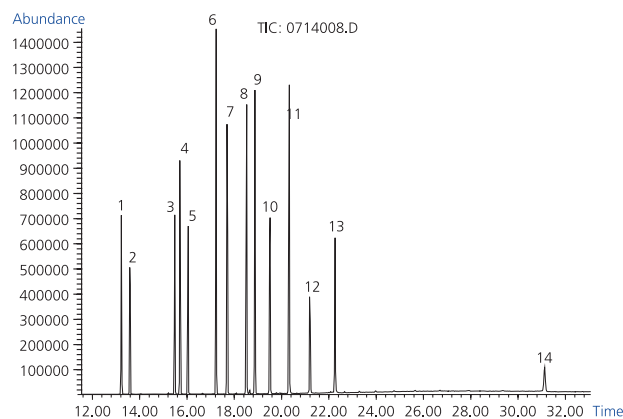
Two features of the separation are noteworthy. First, the absence of column bleed and subsequent high signal-to-noise ratio, even at the analysis temperature of 340 °C, is a direct result of the SLB-5ms silphenylene polymer chemistry and facilitates low level detection. Unlike traditional methyl silicone based polymers, silphenylene polymers incorporate a phenyl group into the

**Figure 1.** Polybrominated diphenyl ethers by GC-MS

column: Supelco SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)  
 oven: 125 °C (1 min.), 10 °C/min. to 340 °C (15 min.)  
 inj.: 300 °C  
 MSD interface: 340 °C  
 scan range: SIM  
 carrier gas: helium, 1.5 mL/min., constant  
 injection: 1 µL, splitless, pulsed (30 psi until 0.2 min.)  
 liner: 4 mm I.D. single taper  
 sample: PBDE standards, 2.5-10 µg/mL

Peak IDs

1. PBDE 17, 2.5 ppm	8. PBDE 85, 2.5 ppm
2. PBDE 28, 2.5 ppm	9. PBDE 154, 2.5 ppm
3. PBDE 71, 2.5 ppm	10. PBDE 153, 2.5 ppm
4. PBDE 47, 2.5 ppm	11. PBDE 138, 3.8 ppm
5. PBDE 66, 2.5 ppm	12. PBDE 183, 2.5 ppm
6. PBDE 100, 2.5 ppm	13. PBDE 190, 2.5 ppm
7. PBDE 99, 2.5 ppm	14. PBDE 209, 10 ppm



backbone structure. This increases the stability of the polymer by causing steric hindrance of the backbiting-scission reaction that leads to phase degradation and column bleed. Additionally, the polymers used in the SLB-5ms undergo extensive cross-linking, further reinforcing the stability of the bonded phase.

The second important feature is the excellent peak shape and response of the PBDE congeners tested on the SLB-5ms column. This is a critical requirement for trace level detection. The inertness of the SLB-5ms column is a result of the pre-treatment of the fused silica, which includes a proprietary deactivation technique.

Both the Fluka PBDE analytical standards and the Supelco SLB-5ms capillary GC column are examples of Sigma-Aldrich's commitment to offer practical, innovative and high-quality solutions for environmental applications. For more information on these products, please call or visit our website: [www.sigma-aldrich.com](http://www.sigma-aldrich.com)

#### Supelco SLB-5ms characteristics:

**Phase:** Bonded and highly cross-linked; silphenylene polymer virtually equivalent in polarity to 5% phenyl polymethylsiloxane

**Operating Conditions:** Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases. Stable to low levels of HCl in non-aqueous samples. Not damaged by organic acids or bases. Columns can be rinsed.

**Temperature Limits:** 0.20 to 0.32 mm I.D. columns: -60 °C to 340 °C (isothermal) / 360 °C (programmable)

**Guard Columns:** We recommend fused silica guard columns to protect the analytical column from damage by sample components. The deactivation should be chosen to match the polarity of the injection solvent.

This column meets USP G27 and G36 requirements.

**Ordering information**

Prod No.	Length (m)	df (µm)	Beta
<b>Supelco SLB-5ms Capillary GC Columns</b>			
<b>0.20 mm I.D. Fused Silica</b>			
28513-U	30	0.20	250
<b>0.25 mm I.D. Fused Silica</b>			
28467-U	30	0.10	625
28469-U	15	0.25	250
28471-U*	30	0.25	250
28472-U	60	0.25	250
28473-U	30	0.50	125
28476-U	30	1.0	63
<b>0.32 mm I.D. Fused Silica</b>			
28557-U	15	0.25	320
28482-U	30	0.25	320
28532-U	30	0.32	250
28484-U	30	0.50	160
28487-U	20	1.0	80

\* Dimension used in the PBDE separation described herein

**Ordering information**

Prod No.	Length (m)	I.D. (mm)	Max. Temp.
<b>Fused Silica Guard Column (Non-Polar Deactivation*)</b>			
25722	3	0.25	360°C
25742	5	0.25	360°C
25723	3	0.32	360°C
25743	5	0.32	360°C

\* Recommended for this application (alkane injection solvents)

**Ordering information**

Prod No.	Name	Description	Pack Size
<b>PBDE Analytical Standards</b>			
34120	PBDE 209	Decabromobiphenyl ether, 50 µg/mL in isooctane:toluene (9:1)	1 mL
34113	PBDE 207	2,2',3,3',4,4',5,6,6'-Nonabromobiphenyl ether, 10 µg/mL in nonane	1 mL
34122	PBDE 138	2,2',3,4,4',5'-Hexabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34121	PBDE 119	2,3',4,4',6-Pentabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34114	PBDE 85	2,2',3,4,4'-Pentabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34115	PBDE 77	3,3',4,4'-Tetrabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34116	PBDE 75	2,4,4',6-Tetrabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34118	PBDE 71	2,3',4,6-Tetrabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34119	PBDE 66	2,3',4,4'-Tetrabromobiphenyl ether, 50 µg/mL in isooctane	1 mL
34123	PBDE 37	3,4,4'-Tribromobiphenyl ether, 50 µg/mL in isooctane	1 mL

\*Additional PBDE standards are currently under development, please inquire

**References**

- (1) Kuriyama, S. N.; Talsness, C. E.; Grote, K.; Chahoud, I. *Environ. Health Perspect.* **2005**, 113(2), 149-54.
- (2) de Wit, C.A. *Chemosphere* 2002, 46, 583-624.
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- (4) Hardy, M. L.; Biesemeier, J.; Manor, O.; Gentit, W. *Environment International* **2003**, 29(6), 793-799.

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**i** Information Request ..... 2111

## STANDARDS &amp; REAGENTS ARTICLE

## New Sigma-Aldrich Analytical Standards Web Site and Search Engine

Ingo Haag, Ph.D. Analytical Marketing Manager

Recently released, our new and improved web marketing area for analytical standards includes Fluka, Riedel-de Haën, Sigma and Supelco standards and reference materials and features a Standards Explorer search engine, ready access to Certificate of Analysis (C of A) and MSDS information, custom quotations and many other benefits to the user in one convenient location.

The union of Fluka, Riedel-de Haën and Supelco with the rest of the Sigma-Aldrich family brought together their strengths as major suppliers of analytical chemical standards. With over eight thousands products representing all analytical markets and techniques (Table 1), the Sigma-Aldrich analytical standards offering ranges from individual compounds to complex mixtures containing dozens of chemicals. Our standards, test mixes and Certified Reference Materials cover many regulated methods from national and regional authorities from Europe and North America.

Table 1.

Markets	Analytical Techniques
Chemical & Polymer	Chromatography (HPLC, GC)
Clinical	Electrophoresis
Environmental	Ion Chromatography
Food & Beverage	Physical Properties
Forensic & Veterinary	Polymer Science
Life Science	Spectroscopy
Petroleum	Thermal Analysis
Pharmaceutical	Titrimetry

Although the vastness of the Sigma-Aldrich analytical standards offering is definitely a competitive advantage, navigating through it to find the right product could be a daunting task. To make the navigation easier, we began by combining Fluka, Riedel-de Haën and Supelco brand standards under one umbrella: "Analytical Standards." Next, realising the exponential growth of the internet for product information, especially through search engines, we turned our attention to designing and launching a new analytical standards website with facile search ability. Corporate-wide, the Sigma-Aldrich website is undergoing a facelift in 2006, so our timing was perfect.

Approaching our new **Analytical Portal**, users will see first-hand the breadth of our analytical products offering, which we have logically segmented into the following technology groups:

- Analytical Standards
- Analytical Reagents
- HPLC
- Gas Chromatography (GC)
- Sample Preparation and Purification
- Titration
- Spectroscopy
- Analytical Microbiology
- Thin Layer Chromatography (TLC)

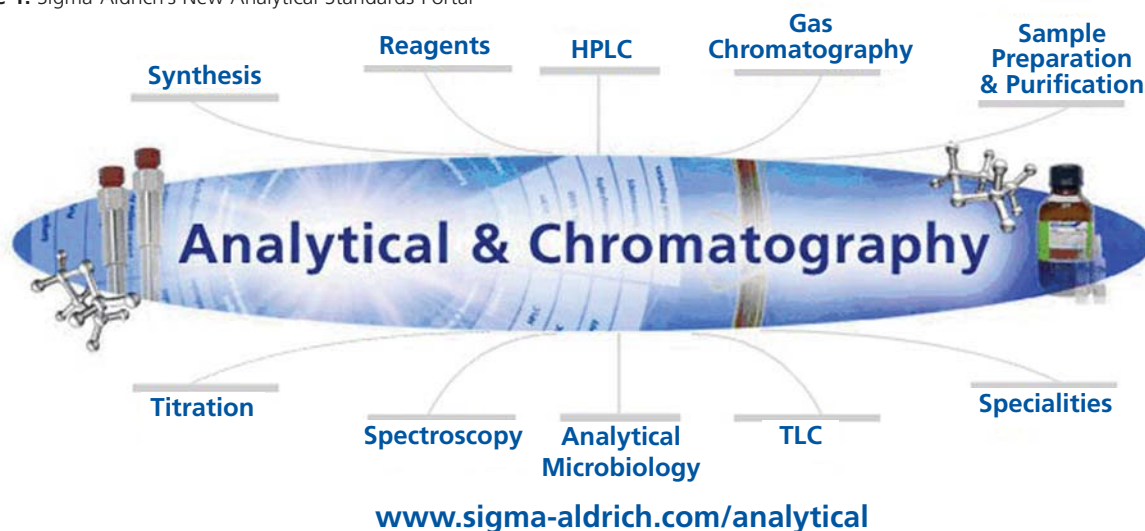
By selecting "Analytical Standards," the user is taken to the **Analytical Standards Portal** shown in Figure 1. Via this portal, users can search our collection of analytical standards and reference materials, get pricing and availability, obtain Certificates of Analysis (C of A) and MSDS data-packs, download literature and request quotations for custom standards. Our new Analytical Standards Catalogue and CD can be ordered on-line from this portal as well.

#### The Analytical Standards Portal

The Analytical Standards Portal gives access to information specific to our standards line. Here, the user can move between the various links, which include:

- Standards Search
- Certified Reference Materials
- Chromatography / CE
- Custom Services
- Certificates of Analysis (C of A) Datapacks and MSDS
- Literature
- Related Products
- Links

Figure 1. Sigma-Aldrich's New Analytical Standards Portal



### Standards Explorer Search Engine

We termed our standards search engine "Standards Explorer." Recognising that people have different approaches to searching for the target analytical standard and come into the search with different information about the standard they want, we developed a user interface for the Standards Explorer that allows fast yet flexible searching by:

- Product (Standard) name
- Compound (Component) name
- CAS number
- Molecular formula

The Standards Explorer allows multiple key word entries to help narrow searches to yield the most relevant products. For example, by entering "EPA 625" in Product Name (Figure 2a) the user is presented with our nine different test mixes for this method (Figure 2b).

Figure 2a. Standards Explore User Interface, Search Criteria "EPA 625"

Figure 2b. Results for Product Name = "EPA 625"

However, if the user is interested in only the EPA 625 mixes that contain naphthalene, entering "EPA 625" in Product Name and "naphthalene" in Component Search (Figure 3a) brings up our EPA 625 Base Neutral 2 test mix (Supelco, 48832) which meets both criteria (Figure 3b).

Figure 3a. Standards Explore User Interface, Search Criteria "EPA 625" and "naphthalene"

Figure 3b. Results for Product Name = "EPA 625" and Component = "naphthalene"

48832 EPA 625 Base Neutral 2	
Supelco 200 µg/mL each component in methylene chloride, ampule of 1 mL	
Expand/Collapse All	
Price and Availability	
Product Number	Your Price GBP
48832	29.70
Available to Ship See details details...	
Quantity <input type="text"/>	
Actions	
Properties	
composition	Acenaphthene
	Anthracene
	Benzo(a)anthracene
	Bis(2-chloroethoxy)methane
	Chrysene
	Dibenzo(a, h)anthracene
	1,2-Dichlorobenzene
	1,3-Dichlorobenzene
	Diethyl phthalate
	2,4-Dinitrotoluene
	Fluorene
	Hexachlorobenzene
	Hexachlorobutadiene
	Naphthalene
	Pyrene
suitability	suitable for 625 per US EPA
storage temp.	2-8°C

With one further click, the product can be ordered directly from this screen. If the search resulted in multiple hits, a "Refine Search" option is available where criteria such as brand, grades, purity and test methods can be applied.

### Custom Standards and New Product Ideas

Our customers are a valuable source of ideas for new analytical standards and they rely on Sigma-Aldrich for custom products – special formulations, testing and packaging. The Analytical Standards portal gives the user the convenience of web-based custom product request, and a means to enter ideas for new products.

We will continue to refine the Standards Explorer, adding new search features and other options to make browsing and choosing the right Sigma-Aldrich analytical standard faster and easier.

To see the Standards Explorer and take it for a test run, go to: [www.sigma-aldrich.com/standards](http://www.sigma-aldrich.com/standards)

# For Superior MS Identification of Trace Analytes...

## ...use Supelco SLB columns

health care   flavor/fragrance   pharmaceutical   food/beverage   material science   environmental



Today's chromatographers require GC columns with consistent low bleed characteristics. High column bleed may interfere with proper mass spectral identification of analytes and may also foul the MS source. For these reasons, capillary columns that exhibit very low levels of bleed are desirable.

With Supelco's new SLB capillary columns, analysts receive:

- Cleaner mass spectra
- Lower detection limits
- Less instrument downtime
- Less preventative maintenance
- Shorter analysis times
- Greater reproducibility, column-to-column

### Service before and after the sale

Sigma-Aldrich/Supelco provides consistent, customer-oriented solutions that ensure product quality and availability, competitive pricing, and award-winning customer service. Our Technical Service Department will be glad to assist you in the selection of the appropriate column for your application.

For more information, contact our technical experts at [EurTechServ@europe.sial.com](mailto:EurTechServ@europe.sial.com) or go to [sigma-aldrich.com/capillary-ms](http://sigma-aldrich.com/capillary-ms)

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