

A Technical Newsletter for Analytical & Chromatography

The Reporter

volume 25.3

Extraction of NNAL with SupelMIP™ SPE



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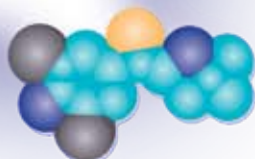
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Dear Colleague,



An Trinh
Product Manager, Sample Prep

In a recent LCGC Magazine survey conducted by Advanstar Communications, it was estimated that over 30% of errors and difficulties experienced during chromatographic analysis could be attributed to problems associated with sample preparation. The same survey also indicated that in terms of "time spent on the analytical process", over 60% of analysts' time is dedicated to sample preparation.

One of the main reasons behind this statistic is the growing mainstream popularity of mass-spectrometry. As more analytical laboratories are acquiring LC-MS-MS technology, analysts are able to achieve greater throughput through shorter analytical run times (1-5 min.) and higher levels of sensitivity that were previously difficult or impossible to achieve.

Even with such advances in analytical throughput and sensitivity, reduction in chromatographic run-time becomes a mute point if sample preparation takes ten times as long as the analysis itself. During the early stages of LC-MS growth, it was originally believed that mass-spectrometry was so selective, minimal sample preparation would be required. However, with the recent surge of journal publications related to the characterization and removal ion-suppression/enhancement, researchers are realizing that sample prep selectivity is still crucial for analytical success. This is especially true when trace levels are concerned.

Sample prep technology has definitely come a long way over the last two decades. The world of separatory funnels are eroding. Nowadays, analysts have a host of techniques available to them; and with the specific goal of providing researchers with chromatographic solutions to address their concerns regarding sample prep throughput, selectivity, and sensitivity, we have developed and commercialized an array of unique technologies. Listed below are a few examples of our most recent advances:

- SupelMIP™ SPE – molecularly imprinted polymers for trace analysis
- A line of all-metal SPME fibers more rugged/reproducible than previous generations
- Radiello® diffusive air samplers – used in an environmental VOC study that was recognized as one of the 24 best European LIFE projects in 2004/2005
- Two high purity carbons used to sample atmospheric gas around Saturn's moon Titan
- A Dioxin Prep System for dioxin, furan, and PCB analysis
- MiniTips™ SPE for micro-scale extraction and desalting of biomolecules
- Silver-ion SPE for the fractionation of cis/trans fatty acids
- Graphitized carbon, multi-layer SPE, and dispersive SPE for multi-residue pesticide analysis in food products

At this time, Supelco R&D is hard at work developing new sample prep tools, technology, and applications that will allow scientists to achieve their sample prep and analytical objectives more effectively and efficiently... so keep a look out for new Supelco sample prep developments in the months to come!

Best Regards,

An Trinh
Product Manager, Sample Prep

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Trace Level Analysis of NNAL in Urine Using SupelMIP™ SPE – NNAL and LC-MS-MS

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Introduction

Tobacco-specific nitrosamines (TSNAs) are created through the burning, curing, and fermentation of tobacco leaf. In 1989, the US Surgeon General provided a list of carcinogens found in tobacco products (1). Among that list were nine nitrosamines that can be found in chewing, smoking, and snuff tobacco. These TSNAs are considered to be highly carcinogenic and have been linked to tumors found in the lung, oral and esophageal cavity, cervix, and liver (1, 2). Because TSNAs are only found in tobacco products, their characterization is invaluable in the study of tobacco's cancerous nature (3).

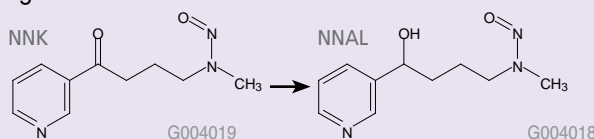
NNK (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone) is a TSA found in tobacco smoke at significant amounts. Upon inhalation, NNK rapidly metabolizes into NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol) (Figure 1). The extraction and quantitation of NNAL in urine is therefore a useful biomarker when assessing a subject's exposure to tobacco smoke. NNAL is not only found in smokers but in non-smokers (via second-hand smoke) as well. Because NNAL is detected in urine at very low concentrations (<1 ng/mL), a highly specific and sensitive assay is required. Although such extraction and analysis protocols have been developed, many of them require extensive and time-consuming (up to 2-3 days) sample preparation (4).

In an effort to develop a simple and highly sensitive assay for the extraction and LC-MS-MS analysis of NNAL in urine, Xia et al of the Center for Disease Control and Prevention, and MIP Technologies AB developed and validated a molecularly imprinted polymer phase and procedure specific for this application (5). In their study, they were able to achieve effective limits of detection of ~1.7 pg/mL. In this report, the utility of molecularly imprinted polymer technology is further demonstrated by comparing the SupelMIP SPE – NNAL procedure with a recently published procedure using a mixed-mode cation polymer SPE phase (6).

Molecularly Imprinted Polymers

Molecularly imprinted polymers (MIPs) are a class of highly cross-linked polymer-based molecular recognition elements engineered to bind one target compound or a class of structurally related compounds with high selectivity. Selectivity is introduced during MIP synthesis in which a

Figure 1. The Metabolism of NNK to NNAL



template molecule, designed to mimic the analyte, guides the formation of specific cavities that are sterically and chemically complementary to the target analyte(s). As a result, multiple interactions (e.g., hydrogen bonding, ionic, Van der Waals, hydrophobic) can take place between the MIP cavity and analyte functional groups (Figure 2). The strong retention offered between an MIP phase and its target analyte(s) allows for the use of exhaustive wash procedures during solid phase extraction that results in superior sample cleanup prior to analysis.

The Extraction of NNAL from Urine

In this study, NNAL was extracted from human urine using SupelMIP SPE – NNAL prior to subsequent LC-MS-MS analysis. The SupelMIP procedure was compared against a method adapted from a recently published study using mixed-mode cation polymer SPE sample prep approach (6). The protocols for both extraction methods are described in Table 1 (page 4).

Improved Selectivity Using SupelMIP SPE – NNAL

NNAL was extracted from urine using both the SupelMIP (molecularly imprinted polymer) and mixed-mode cation-

(continued on page 4)

Figure 2. Example of an MIP Binding Site

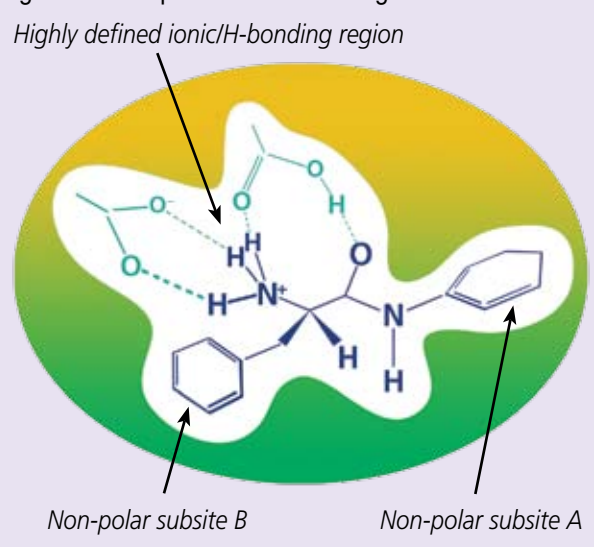


Table 1. Description of SupelMIP SPE – NNAL and Mixed-Mode Cation-Exchange SPE Method for the Extraction of NNAL from Urine

SupelMIP SPE – NNAL Method

Sample Pre-Treatment:

Human urine samples were centrifuged at 3000 rpm. The resulting supernatant was spiked at the levels 0.0 (blank), 60, and 100 pg/mL NNAL and acidified to pH 6 with acetic acid.

SPE Procedure:

SupelMIP SPE – NNAL, 25 mg/10 mL (LRC) (53206-U)

1. Condition and equilibrate MIP phase with 1 mL dichloromethane, 1 mL methanol, and 1 mL DI water.
2. Load pre-treated urine sample onto the cartridge.
 - 2 mL sample volume used for 60 pg/mL spike level
 - 5 mL sample volume used for 100 pg/mL spike level
3. Wash (elute interferences) using the following wash scheme:
 - 2 x 1 mL DI water followed by 10 min. vacuum
 - 1 mL toluene
 - 1 mL toluene:dichloromethane (9:1, v/v)
 - 1 mL toluene:dichloromethane (4:1, v/v) followed by 2 min. vacuum to remove residual solvent
4. Elute NNAL with 2 x 1 mL 10% methanol in dichloromethane. Apply gentle vacuum between each fraction.
5. Evaporate under nitrogen and reconstitute with 0.15-0.25 mL LC mobile phase prior to LC-MS-MS analysis

Published NNAL Method Using Conventional Mixed-Mode Cation-Exchange Polymer Phase (6)

Sample Pre-Treatment:

Human urine samples were centrifuged at 3000 rpm. The resulting supernatant was spiked at the levels 0.0 (blank), 50 pg/mL NNAL and acidified to pH 3-4 by adding 0.15 mL 1 M HCl per 10 mL urine.

SPE Procedure:

Conventional Mixed-Mode Cation-Exchange Polymer Phase (60 mg/10 mL)

1. Condition and equilibrate SPE phase with 4 mL methanol and 4 mL DI water
2. Load 10 mL pre-treated sample onto the cartridge.
3. Wash (elute interferences) with:
 - 2 mL 0.1 M HCl
 - 5 mL methanol
 - 2 mL DI water:methanol:ammonium hydroxide (90:5:5, v/v)
4. Elute NNAL with 5% ammonium hydroxide in methanol
5. Evaporate under nitrogen and reconstitute with 0.25 mL LC mobile phase prior to LC-MS-MS analysis

(continued from page 3)

exchange procedures described in Table 1. Figure 3 is a representative ion-chromatogram (MRM 210.2/180.2) of a 1 ng/mL external standard injection of NNAL. The ion-chromatograms (MRM transitions 210.2/180.2 and 210.2/93.2) for blank urine samples extracted from both procedures are detailed in Figure 4. Of the two MRM transitions, 210/93.2 was more intense; however, high level matrix impurities had co-eluted in the LC elution area of NNAL (0-2 min.) when extracting urine using the mixed-mode cation-exchange procedure. Such impurities can potentially result in signal-suppression of NNAL. Therefore, the MRM transition of 210.2/180.2 was used for quantitation. In contrast, the SupelMIP approach provided a much cleaner extract with a signal-to-noise ratio of at least 10 at the lowest spike concentration tested. This is further demonstrated by spiking NNAL into the SPE eluate of blank urine samples extracted with SupelMIP SPE – NNAL and comparing the response with NNAL standards diluted in LC-mobile phase. In Figure 5, we see that when overlapping the response of NNAL calibration curves diluted in both blank urine extracts (spiked post-extraction) and LC mobile phase, the curves overlapped almost perfectly, signifying almost no (less than 4%) signal suppression from co-extracted matrix impurities (data provided by MIP Technologies AB).

Improved Recovery Using SupelMIP NNAL

From Table 2, we see that at the 50-60 pg/mL spike level, absolute recovery using the SupelMIP protocol was

higher than Mixed-Mode Cation-Exchange procedure. Note that MIP Technologies AB, the manufacturers of SupelMIP SPE – NNAL, recommends quantitating relative recovery against NNAL-d₃ internal standards that are spiked into urine samples prior to SupelMIP extraction. Under such conditions analysts can readily achieve relative recovery values > 90% and limits of detection and limits of quantitation values of 5 pg/mL and 13 pg/mL, respectively (data not shown).

Figure 3. Ion-Chromatogram (MRM 210.2/180.2) of 1 ng/mL NNAL standard

column: Ascentis Express C18, 5 cm x 2.1 mm I.D., 2.7 μm (581307-U)
 instrument: Applied Biosystems 3200 Q-TRAP®
 mobile phase: (A) 10 mM ammonium acetate;
 (B) 10 mM ammonium acetate in acetonitrile

Time (min.)	% A	% B
0.0	90	10
1.5	70	30
2.5	70	30
2.6	90	10
6.0	90	10

temp.: 35 °C
 flow rate: 0.3 mL/min.
 detection: MS/MS, MRM transitions
 (210.2/180.2 and 210.2/93.2 m/z)
 ion mode: Positive
 ion source: Turbospray
 ion spray voltage: 4500 V
 source temp.: 450 °C
 collision gas: 35 psi
 injection: 20 μL

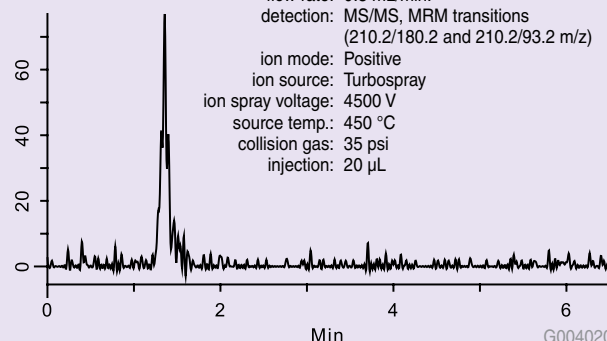


Figure 4. Blank and Spiked NNAL Urine Samples Extracted with SupelMIP SPE – NNAL and Mixed-Mode Cation Exchange Polymer SPE

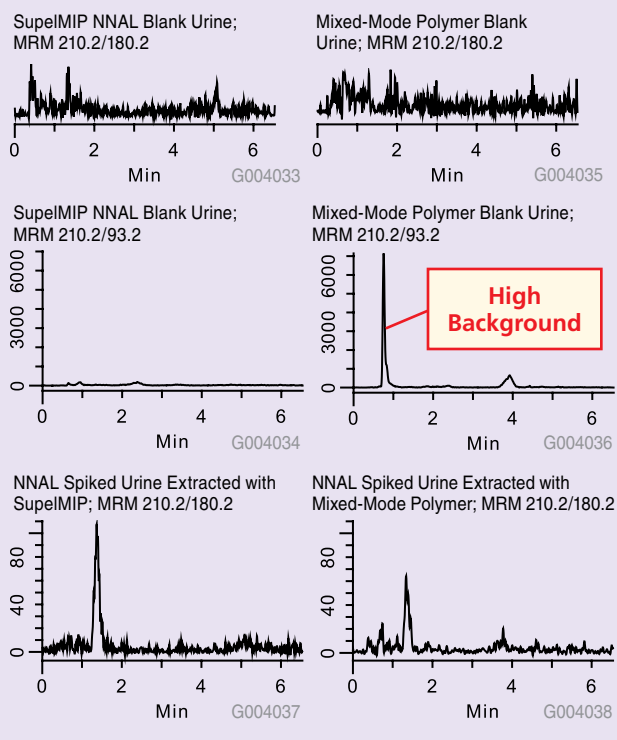


Figure 5. Response Comparison of NNAL Calibration Curve Generated from SupelMIP SPE – NNAL Urine Extract (post-SPE spike) vs. External Standards

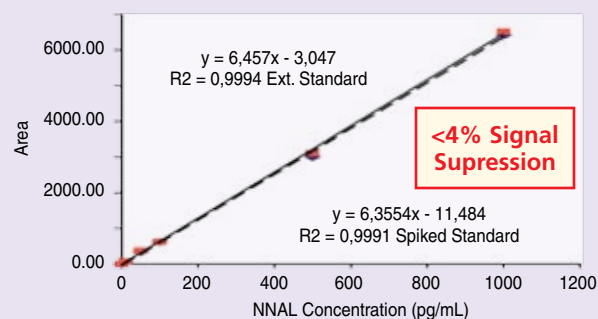


Table 2. Absolute Recovery of SupelMIP SPE – NNAL vs. Mixed-Mode Cation Exchange Polymer SPE

SPE Procedure	% Recovery		
	50 pg/mL Spike	60 pg/mL Spike	100 pg/mL Spike
SupelMIP SPE – NNAL	*	67%	87%
Mixed-Mode Polymer SPE	47%	*	*

* Not analyzed

Conclusion

In this report, we demonstrated the extraction of NNAL from urine using SupelMIP SPE – NNAL method against a published mixed-mode cation exchange polymer phase. Because selectivity is introduced during the development of the MIP itself, it allows for a binding site that is sterically and chemically complementary to the target analyte(s). The multiple interactions that take place between the imprint binding site and analyte(s) of interest offer strong interactions enabling the use of exhaustive wash conditions during the SPE process to provide cleaner extracts prior to analysis. The SupelMIP approach offered improved selectivity over the mixed-mode approach. This was particularly evident when analyzing blank urine sample extracts at MRM transitions 210.2/93.2. In addition, absolute recovery was also higher for the SupelMIP method relative to the mixed-mode procedure. Higher recovery is essential when analyzing NNAL at trace levels.

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6. "Liquid chromatographic/tandem mass spectrometric method for the determination of the tobacco-specific nitrosamine metabolite NNAL in smokers' urine", G.D.Byrd, M.W.Ogden in J. Mass Spectrom. 2003 (38) 98-107

Featured Products

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

Improving HPLC Sample Throughput Using Ascentis™ Express Fused-Core Technology Columns

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Introduction

The demand for increased sample throughput and speed of results has driven HPLC users to search for breakthroughs in HPLC instruments and column technology. Although improvements have been realized, setbacks have been encountered. Reduction in column ruggedness, costly replacements of existing instrumentation, and difficulties in transferring methods to new systems have often made these past improvements unappealing to analysts.

Ascentis Express: Breakthrough HPLC Technology

The Fused-Core™ HPLC particle technology behind Ascentis Express permits 4- to 6-fold reduction in analysis time, with a subsequent increase in sample throughput compared to conventional HPLC columns, without sacrificing resolution or column ruggedness and without the need to change systems or sample prep procedures.

High Column Efficiency via the Kinetic Advantage

Highly efficient columns deliver more plates per meter, which means shorter columns can be used to generate the same number of plates as a longer, less efficient column. Compared to 5 µm totally porous particles, the 2.7 µm Ascentis Express provides about three times the efficiency. Therefore, a 10 cm Ascentis Express column

will provide the same efficiency as a 25 cm column packed with 5 µm particles.

Figure 1 compares the resolution of a five-component sample on 25 cm, 5 µm C18 and 10 cm Ascentis Express C18 columns. Each column has approximately the same number of theoretical plates and hence the same resolving power. However the shorter Ascentis Express column delivers this separation in a much shorter time, in this case less than one-fourth the time as the 25 cm column.

Therefore, a 10 cm Ascentis Express column will provide the same efficiency as a 25 cm column packed with 5 µm particles.

The improved kinetics from the physical structure of the Ascentis Express leads to efficiencies that are higher than predicted by particle size alone. Called "Fused-Core," the 2.7 µm Ascentis Express particle comprises a solid 1.7 µm silica core surrounded by a 0.5 µm porous silica layer (Figure 2). The solid core prevents solutes from diffusing as deeply into the Ascentis Express particle as they can in a totally porous particle. This reduced diffusion lessens band broadening and gives higher efficiency. The Fused-Core particle technology inside Ascentis Express columns provides efficiencies up to 240,000 N/m, which rivals that available from sub-2 µm particles.

Figure 1. Increase Sample Throughput by Using Ascentis Express

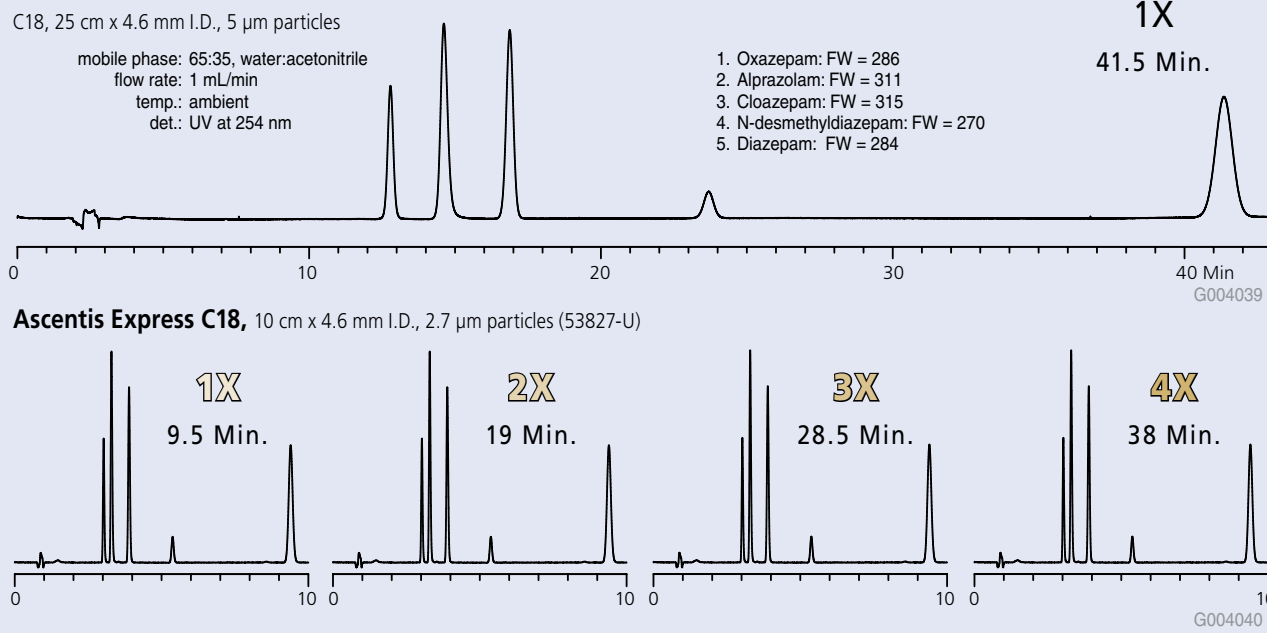
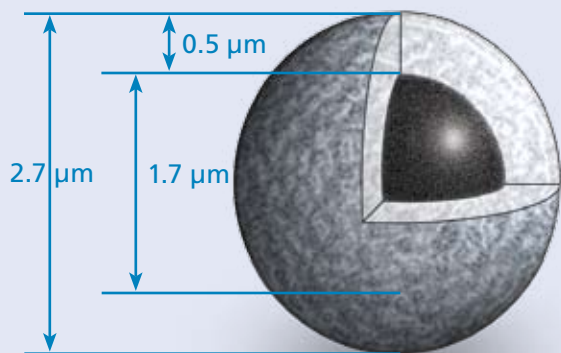


Figure 2. Ascentis Express Particle



Another advantage of Ascentis Express HPLC columns is the very narrow particle size distribution. This allows for 2 µm pore size frits to be used in Ascentis Express columns. These larger pore size frits do not foul as easily as the 0.5 µm frits that are necessary to retain the sub-2 µm and conventional 3 µm particles.

Do More Work in Less Time Without Changing Your Method

The current high resolution column for traditional HPLC methods is a 25 cm column packed with 5 µm particles. Until now, this dimension provided the most efficiency within the pressure limit of a conventional HPLC system. With the high efficiency Ascentis Express, one can now achieve the same number of plates as a 25 cm column packed with 5 µm particles with a 10 cm column or even more efficiency and resolution with a 15 cm Ascentis Express

column. Therefore, by simply changing columns and keeping all other conditions the same, you can reduce the runtime and increase the resolution of your method.

Conclusion

Ascentis Express is the ideal choice for HPLC analysts interested in increasing sample throughput while maintaining or even improving resolution. By reducing solute dispersion, the unique Fused-Core technology gives Ascentis Express a kinetic advantage over conventional particles. Its higher column permeability compared to sub-2 µm particles means that Ascentis Express can achieve UHPLC-like performance on conventional HPLC systems. Under UHPLC conditions, Ascentis Express can exceed the efficiency possible on sub-2 µm columns because longer columns can be used. There are several strategies for improving LC performance by using Ascentis Express. Table 1 outlines the strategies.

+ Featured Products

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

Table 1. Strategies for Improving LC Performance by Using Ascentis Express

Desired Improvement	Current Particle	Change in Column Dimension with Ascentis Express	Change in Flow Rate with Ascentis Express	Resulting Improvement
Speed	Sub-2 µm	None	Double	Double the speed with equivalent plates
Efficiency	Sub-2 µm	Double the length	None	Double the plates
Efficiency	3 µm	None	None	Double the plates
Speed & Efficiency	5 µm	Half the length	None	4 times the speed & 1.3 times the plates

TRADEMARKS: Ascentis, ATIS, Carbo-pack, Carbotrap, Carboxen, CHIROBIOTIC, CHROMASOLV, CYCLOBOND, Fluka, MiniTips, P-CAP, P-CAP-DP, Riedel-de Haën, SGT, Sigma-Aldrich, SLB, SPB, Supelco, SupelMIP, Thermogreen, TraceCERT - Sigma-Aldrich Biotechnology LP; CombiPAL - CTC Analytics; Fused-Core - Advanced Materials Technology, Inc.; Mininert - Valco Instruments Co., Inc.; Q-TRAP - Applied Biosystems; Radiello - Fondazione Salvatore Maugeri; Super Clean - Scientific Glass Technology (SGT); Tenax - Enka Research Institute Arnhem

SPME - Technology licensed exclusively to Supelco. US patent #5,691,206, European patent #523,092.

P-CAP and P-CAP-DP are patent pending and manufactured under license from La Sapienza, Università degli Studi di Roma.

Astec CYCLOBOND™ Chiral HPLC Columns Offer Broad-Based Chiral Selection

Ric Cone

ric.cone@sial.com

Astec CYCLOBOND chiral stationary phases (CSPs) are 5 μm spherical silica covalently bonded with cyclodextrins (CDs) or derivatized CDs. CYCLOBOND I 2000 CSPs are bonded β -CD or derivatives with 0.78 nm cavities. CYCLOBOND II CSPs are bonded γ -CD or derivatives with 0.95 nm cavities. A CYCLOBOND Methods Development Kit (20005AST) consisting of 4 CYCLOBOND CSPs offers complementary selectivity to the Astec CHIROBIOTIC™ Methods Development Kit (10305AST).

Separations on CD-bonded phases involve inclusion complexing, based on the insertion of a polar aromatic or cycloalkyl ring(s) of the analyte into the hydrophobic CD cavity. In general, substituted phenyl, naphthyl, and biphenyl rings can be separated on CYCLOBOND I 2000. Analytes with 3-5 rings, including steroids are best separated on CYCLOBOND II. The affinity for the CD cavity is influenced by functional groups such as halogens, nitrates, sulfates, phosphates and phenols on the analyte's aromatic rings, or preferred hydrogen groups on the rest of the molecule, including carboxyl, carbonyl, amide, hydroxyl, and amines. Chiral selectivity is also based on hydrogen bonding, and dipole-dipole interactions with the CD cavity.

Chiral separations can be carried out using reversed-phase, polar-organic phase, or normal phase modes, with the reversed-phase mode generally providing for inclusion complexing, and normal phase mode favoring hydrogen bonding, or π - π interaction for some derivatized CDs. The polar-organic mode consists of acetonitrile and/or methanol with varying amounts of acetic acid and/or triethylamine, added for control of hydrogen bonding or peak tailing. This mode enhances interactions with secondary hydroxyl groups across the CD opening, as well as some functional groups found on derivatized CDs.

Derivatized CYCLOBOND CSPs such as CYCLOBOND I 2000 DNP (dinitrophenyl), and CYCLOBOND I 2000 DMP (3,5-dimethylphenyl carbamate) provide additional chiral selection based on π -acid and π -base interactions, respectively. CYCLOBOND I 2000 HP-RSP (high performance R,S-hydroxypropyl ether) extends hydrogen bonding capabilities, and is ideal for screening and methods development, offering broader selectivity, shorter retention time, higher reproducibility, MS compatibility, and twice the stability of other cyclodextrin CSPs. Together with

Figure 1. Warfarin

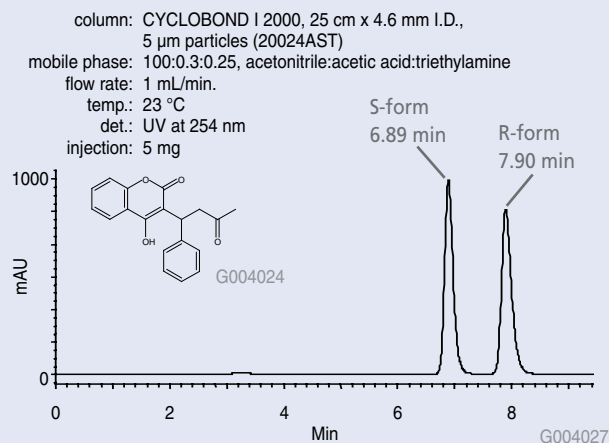


Figure 2. 1,1'-Binaphthol

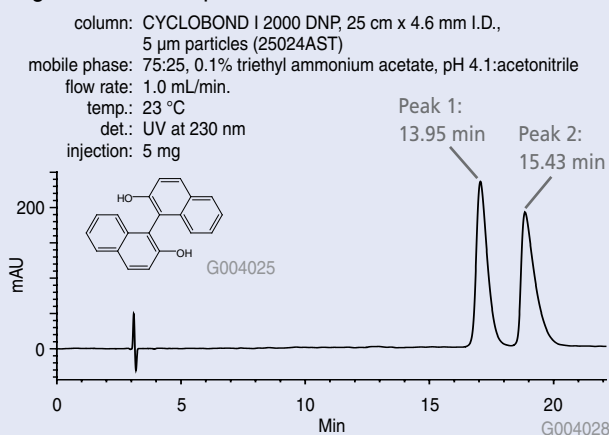
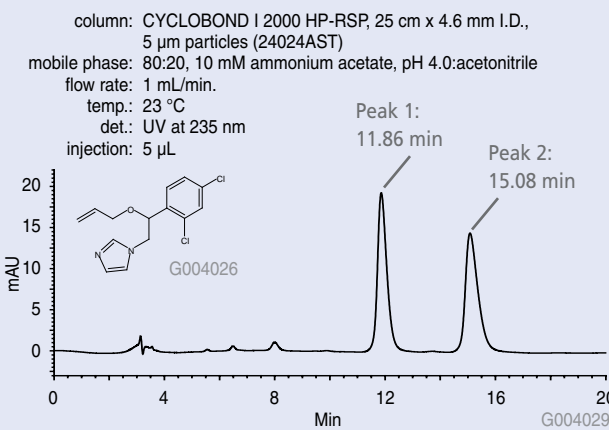


Figure 3. Enilconazole



CYCLOBOND I 2000, these CSPs form the CYCLOBOND Method Development Kit (20005AST).

CYCLOBOND I 2000 RN (R-naphthylethyl carbamate) and CYCLOBOND I 2000 SN (S-naphthylethyl carbamate)

are additional derivatized CDs that also provide chiral selection based on π - π interactions. CYCLOBOND I 2000 RSP (R,S-hydroxypropyl ether) offers broad selectivity including the ability to separate non-aromatic structures such as *t*-Boc amino acids. CYCLOBOND I 2000 AC (acetyl) is used primarily for aromatic alcohols or amines with chirality on the alpha or beta carbon. CYCLOBOND I 2000 DM (dimethyl) separates structural and geometric isomers

and some enantiomers not separated using CYCLOBOND I 2000 (reversed phase mode only).

Figures 1 – 3 are enantiomer separations on some CYCLOBOND columns: (1) Warfarin separated on CYCLOBOND I 2000; (2) 1,1-bi-2-naphthol separated on CYCLOBOND I 2000 DNP; and (3) enilconazole, a fungicide, separated on CYCLOBOND I 2000 HP-RSP.

Astec P-CAP™ and P-CAP-DP™ Chiral HPLC Columns

Durable Design and High Load Capacity for Normal Phase and Analytical-to-Preparative Applications

Ric Cone

ric.cone@sial.com

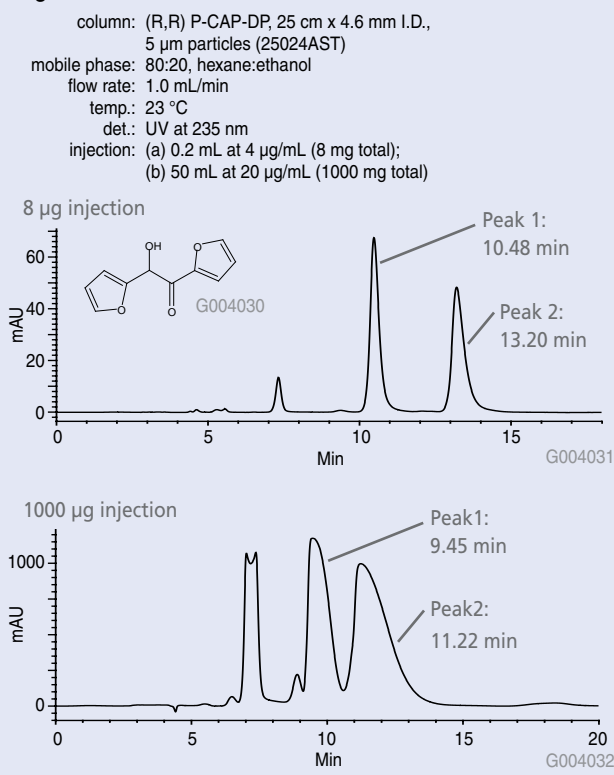
Astec P-CAP and P-CAP-DP are polymer-based chiral stationary phases (CSP) covalently bound to either 5 μ m or 3.5 μ m spherical silica. Chiral separations are typically carried out either in normal phase (heptane/IPA or ethanol) or polar organic (acetonitrile/methanol) mode, with chiral selection either through hydrogen bonding for P-CAP, or through both hydrogen bonding and π - π interaction for P-CAP-DP. The high loading capacity for these CSPs makes them excellent for both analytical and preparative scale separations. For screening, each CSP offers different and sometimes complimentary selectivity, and use of normal phase versus polar organic mode also offers different and sometimes complimentary selectivity. Each phase is available in both R, R and S, S enantiomeric forms, allowing the analyst full control of the order of elution for successful separations.

P-CAP and P-CAP-DP are rugged, experience no memory effect, and can be run in a wide variety of solvents. Volatile acids and buffers can be used to enhance peak efficiency when needed or to enhance ion detection for MS platforms.

For P-CAP-DP, additional racemates resolved with high selectivity include hydroxycarboxylic acids, alcohols, sulfoxides, esters, amides and lactones and N-blocked amino acids. There are no known limitations on the kind of solvents that can be used with these phases including halogenated compounds.

The separation of furoin enantiomers is shown in Figure 1 using a 25 cm x 4.6 mm (R,R) P-CAP-DP analytical column and mobile phase of 20/80 EtOH/Hexane. Excellent separation is demonstrated with a load of 8 μ g of furoin racemate. The ability of this analytical column to separate up to 1 mg of furoin demonstrates the high loading capacity of this phase.

Figure 1. Furoin



Featured Products

Description	Length (cm)	I.D. (mm)	Cat. No.
CYCLOBOND - 250 Kit	25	4.6	20005AST
CYCLOBOND I 2000, 5 μ m	15	2.1	20019AST
CYCLOBOND I 2000, 5 μ m	25	4.6	20024AST
CYCLOBOND I 2000 DMP, 5 μ m	15	2.1	20719AST
CYCLOBOND I 2000 DMP, 5 μ m	25	4.6	20724AST
CYCLOBOND I 2000 HP-RSP, 5 μ m	15	2.1	24019AST
CYCLOBOND I 2000 HP-RSP, 5 μ m	25	4.6	24024AST
CYCLOBOND I 2000 DNP, 5 μ m	15	2.1	25019AST
CYCLOBOND I 2000 DNP, 5 μ m	25	4.6	25024AST

Description	Length (cm)	I.D. (mm)	Cat. No.
(R,R) P-CAP, 5 μ m	15	2.1	31019AST
(R,R) P-CAP, 5 μ m	25	4.6	31024AST
(S,S) P-CAP, 5 μ m	15	2.1	33019AST
(S,S) P-CAP, 5 μ m	25	4.6	33024AST
(R,R) P-CAP-DP, 5 μ m	15	2.1	35019AST
(R,R) P-CAP-DP, 5 μ m	25	4.6	35024AST
(S,S) P-CAP-DP, 5 μ m	15	2.1	37019AST
(S,S) P-CAP-DP, 5 μ m	25	4.6	37024AST

Novel Carbon-Based Adsorbents For Thermal Desorption Applications

Jamie L. Brown

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Introduction

Adsorbents used for sampling volatile organic compounds (VOCs) in the air are typically hydrophobic, have high thermal stability, and provide low background levels. VOC atmospheres typically contain a significant amount of moisture. Because the condensation of water on the adsorbent surface can compete with the organic analytes of interest, hydrophobic adsorbents are required. Many consider thermal desorption as the preferred method of VOC extraction (over solvent desorption) because the sample is concentrated and can be analyzed without dilution of the sample. Therefore, adsorbents with high thermal stability and low background levels are required. In the past decade, the use of carbon-based adsorbents has increased due to their superior performance.

The Problem With Porous Polymers

Porous polymers are commonly used for sampling VOCs. However, these materials are plagued with variable background levels. If a porous polymer becomes exposed to oxygen, even at trace levels, during the conditioning and/or desorption processes, oxidation of the polymer framework can produce high background levels. This is caused by the release of residual monomers left in the adsorbent during manufacturing. In addition to high background, small voids could be created as the residual monomers are baked out of the adsorbent. If these voids become large enough, they can lead to channeling during sampling and/or desorption.

The Solution: Carbon-Based Adsorbents

Carbon-based adsorbents offer more choices for sampling atmospheres containing VOCs. These graphitized carbons and carbon molecular sieves are hydrophobic, efficiently release the compounds of interest during the desorption process, allow a broader spectrum of analytes to be collected, and exhibit lower background levels than porous polymers. Carbon-based adsorbents do not exhibit high baseline problems because they are manufactured at temperatures well above 500 °C. This is hot enough to ensure complete pyrolysis of the polymeric precursor material so that no residual monomers remain. Therefore, there is nothing left to be oxidized, no voids can be created, and channeling is not a concern.

Hydrophobicity Test

To create a dynamic humid atmosphere, a stream of “dry” air was mixed with a stream of “wet” air. The stream of “wet” air was made by bubbling clean air through deionized water. By mixing the two gas streams in the correct proportion, a humidified stream with a relative humidity level of 85% RH was created. The total flow was 0.05 L/min. A pre-conditioned tube was weighed to get its tare weight and then attached to the outlet of the humidified gas stream. After 1 liter of humidified air passed through the tube, it was removed and weighed. This was repeated four more times until 5 liters passed through the tube. As shown in Table 1, the carbon-based adsorbents did not retain significant amounts of water. The amount of water exposed to the tubes is shown for comparison.

Table 1. Hydrophobic Characteristics of Three Carbon-Based Adsorbents (weight gain in grams)

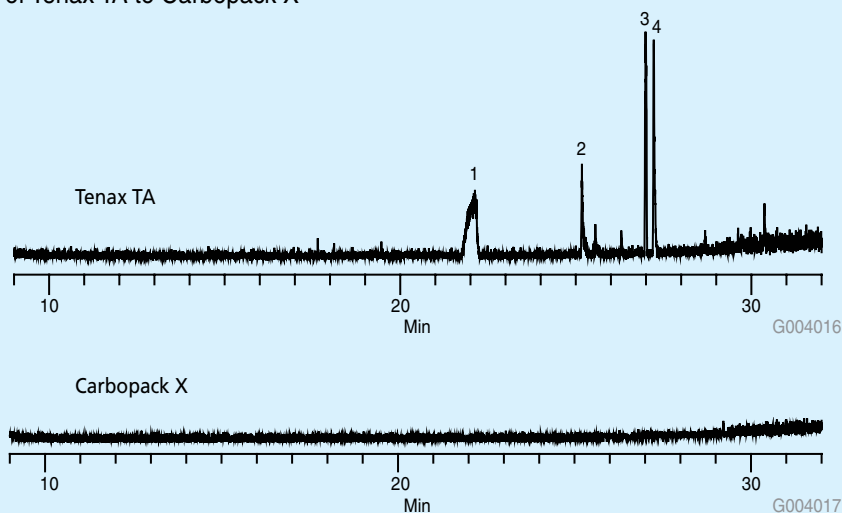
Sample Volume (L)	0	1	2	3	4	5
Carbopack™ X	0	1	1	1	2	1
Carbopack Z	0	0	0	0	0	0
Carboxen™ 1016	0	0	1	1	0	1
Mass of water tube exposed to	0	19	38	57	76	95

Background Test

In Figure 1, a comparison of the background level is shown for both Tenax® TA and Carbopack X. Before the analysis, each tube was conditioned at 50 mL/min. at 320 °C for 1 hour. After the tubes reached room temperature, they were thermally desorbed to determine their background. The chromatograms were generated using the conditions specified in Figure 1. As shown, the porous polymer Tenax TA exhibits background of some residual benzene ring compounds that are breakdown products of the polymer. In comparison, the carbon-based adsorbent Carbopack X does not exhibit any background peaks, allowing for a better quality analysis.

Figure 1. Comparing Background Levels of Tenax TA to Carbpacck X

sample/matrix: blank runs
 adsorbent tube: 11.5 cm x 6 mm O.D. x 4 mm I.D.
 glass TDU tubes with adsorbent packed into a 6 cm bed at the center of the tube
 desorption temp.: 300 °C for 5 min.
 desorption flow: 25 mL/min.
 transfer line/
 valve temp.: 200 °C
 column: SPB-1, 60 m x 0.25 mm I.D., 1.0 µm (24031)
 oven: 35 °C (8 min.), 5 °C/min. to 100 °C, 15 °C/min. to 230 °C (hold 4 min.)
 inj.: 200 °C
 MSD interface: 230 °C
 scan range: m/z = 35-260
 carrier gas: nitrogen, 2 mL/min. constant
 injection: split 10:1



1. Siloxane
2. Benzaldehyde
3. Limonene
4. Acetophenone

Conclusion

Today there are novel carbon-based adsorbents that can be used with, or in place of, porous polymers. These carbon-based adsorbents have low background, retain minimal water while sampling humid environments, and can be used to sample a broader spectrum of analytes. Presented in this article is just a small sampling of Supelco's continued research and development on the most state-of-the-art carbon-based adsorbents.

Featured Products

Description	Qty.	Cat. No.
Carbpacck X, 60-80 mesh	10 g	10437-U
Carbpacck Z, 60-80 mesh	10 g	11051-U
Carboxen 1016, 60-80 mesh	10 g	11021-U
Tenax TA, 60-80 mesh	10 g	11982
ATIS (Adsorbent Tube Injector System)	1	28520-U
ATIS Purge and Trap / Humidifier Module	1	28522-U
SPB-1, 60 m x 0.25 mm I.D., 1.0 µm	1	24031

Related Products

Description	Qty.	Cat. No.
Carbotrap™ X, 20-40 mesh	10 g	10435-U
Carbpacck X, 40-60 mesh	10 g	10436
Carbotrap Y, 20-40 mesh	10 g	10460-U
Carbpacck Y, 40-60 mesh	10 g	10461-U
Carbpacck Y, 60-80 mesh	10 g	10462

Related Information

For more information on carbon-based adsorbents, request *New Carbon-Based Adsorbents for Thermal Desorption Applications*, T407014 (JLQ) (Available in electronic form only. Please provide email address.), *A Tool for Selecting an Adsorbent for Thermal Desorption Applications*, T402025 (HTA), *Carbon Adsorbent Kits*, T406044 (IPS), or contact Supelco Technical Service at 800-359-3041 (US and Canada

Did you know...?

The Adsorbent Tube Injector System (ATIS™) is a sample preparation device that provides a quick and efficient way to transfer calibration standards or samples onto an adsorbent tube. The ATIS employs the technique of flash vaporization to vaporize the sample into a continuous flow of an inert gas. The inert gas carries the sample to the tube. After enough time has elapsed, typically less than 5 minutes, the tube is removed from the ATIS and analyzed using an appropriate technique. The ATIS Purge & Trap / Humidifier Module is designed to be used in conjunction with the ATIS. In addition to expanding the ATIS capabilities to perform purge & trap type sampling, this same module can also be used to create a humidified gas stream, mimicking conditions the adsorbent tube could be exposed to in the field.



Selecting the Appropriate SPME Fiber Coating – Effect of Analyte Molecular Weight and Polarity

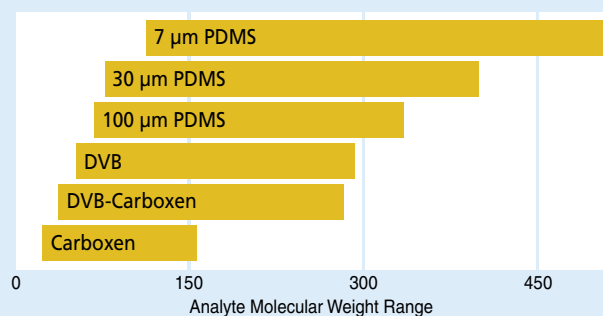
Robert Shirey

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Previously in the Reporter (Vol. 24.3) we discussed how to select the appropriate fiber assembly, focusing on fiber core, needle gauge and assembly design. This article focuses on how to select the appropriate fiber coating based upon analyte size and polarity. There are 4 major criteria that are commonly used in selecting the proper fiber coating. These are molecular weight/size of an analyte, the polarity, the analyte concentration level, and the complexity of the sample matrix. This article will focus on the molecular weight or size and polarity of the analyte in relation to fiber coatings. A future article will focus more on the relationship between analyte concentration and fiber capacity.

The molecular weight of an analyte determines how rapidly it can move in and out of the fiber phase coating and through the sample. A smaller analyte will move faster and is not as well retained; whereas, the larger analytes migrate through the coating and sample more slowly and take a much longer time to reach equilibrium. The coating thickness plays a major role in determining the equilibrium time and the desorption efficiency of the analyte from the fiber coating (Figure 1).

Figure 1. Molecular Weight Extraction Range for SPME Fiber Coatings

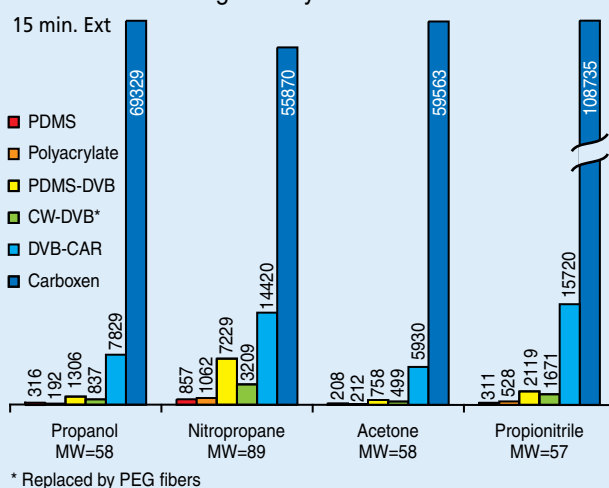


The size and shape of a molecule is also important. A flat planar structure with no substitution groups can interact with the phase coating or adsorbent usually through π - π interactions. Examples of these types of analytes are PAHs, and long chained molecules with multiple double bonds that make them more rigid. Because of increased interactions, these analytes effectively act like analytes with molecular weights 30-50 atomic mass units (AMU) higher. Conversely, if the analyte is highly branched, or is aromatic with substitution groups, such as chlorine, or bromine, this reduces the interaction with adsorbents, and their effective size is actually smaller if the substitution group has high electronegativity. Since the size of the molecule is smaller

than an all hydrocarbon structure of the same MW, it has the extraction efficiency similar to a molecule that is approximately 30 amu less in molecular weight. This number will vary depending upon the degree of substitution and is just a rough guideline.

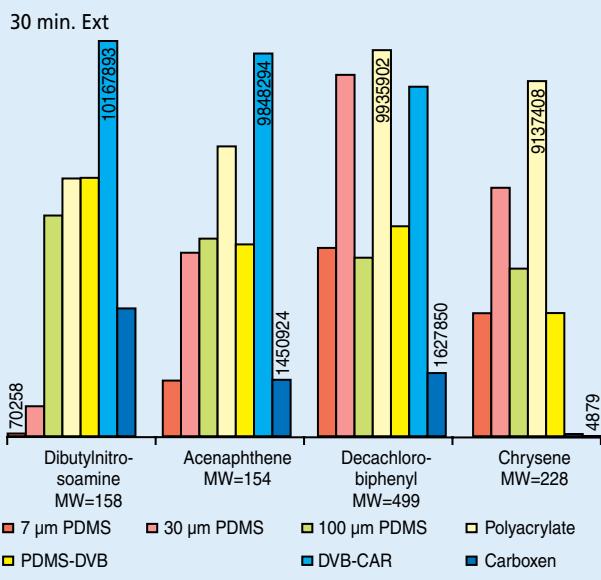
Generally for smaller analytes with a MW less than 150, an adsorbent fiber coating that can retain these fast moving analytes is recommended. Carboxen fibers are recommended for these analytes especially at trace concentration levels. Other fiber coatings containing DVB and those with thicker phase coatings may be suitable especially if the analytes are in higher concentration levels or the samples contain multiple analytes (Figure 2). In this figure the area counts are labeled above the peaks. The Carboxen-PDMS fiber coating on average extracted 200 times more of these analytes than the 100 µm PDMS fiber.

Figure 2. Fiber Coating Comparison - Response of Low Molecular Weight Analytes



For larger analytes, adsorbent fibers (those without adsorbents) may be the better option, but divinylbenzene (DVB) containing fibers with larger pores are suitable for many of these analytes. Figure 3 shows the extraction efficiency of some larger analytes. The area responses for the fiber coatings yielding the highest and lowest responses have been noted with the actual area counts. The fiber least effective for the extraction of chrysene is Carboxen-PDMS. This 4-ringed PAH has strong interaction with the carbon surface and is not efficiently desorbed. The response is so low that it does not show on the graph. Even though decachlorobiphenyl has a higher molecular weight than chrysene, it is desorbed more efficiently by the Carboxen-PDMS fiber because the chlorine molecules reduce the π - π interactions between Carboxen and DCBP.

Figure 3. Comparison of Fiber Coatings – Response of Semi-volatile Compounds



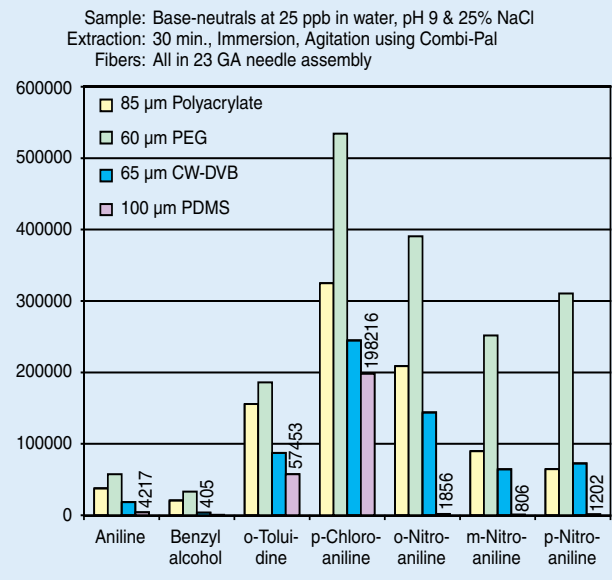
Most of the other fibers extract these large non-polar analytes fairly well. The extraction time plays a major role. The extraction time for the analytes shown in Figure 2 was 30 min. The larger analytes are more efficiently analyzed by the 30 µm PDMS fiber than the 100 µm, PDMS fiber. It takes much longer for large analytes to reach equilibration in thicker fiber coatings. Also, desorption is less efficient from thicker coatings. In general the absorbent fiber coatings, those not containing DVB or Carboxen, are the better choice for these larger analytes.

Dibutylnitrosoamine is the only analyte in this graph that has mixed polarity. It has 2 bulky non-polar butyl groups, but it contains a polar nitrosamine grouping. Because of the increased polarity, the non-polar PDMS fibers were not efficient for extracting these analytes. The polyacrylate fiber and the adsorbent containing fibers were better choices for these more polar analytes. Another alternative is the newly developed PEG fiber.

The DVB-Carboxen fiber was designed to extract a wider molecular weight range. This fiber coating consists of a layer of DVB suspended in PDMS coated over a layer of Carboxen-PDMS. The concept is that the larger analytes will be retained by DVB whereas, the smaller analytes will migrate into the Carboxen adsorbent. This effectively increases the molecular weight range that the fiber can extract. As shown in Figures 2 and 3, this fiber was a good compromise over a very wide molecular weight range. The only limitation is that this fiber will have less sample capacity due to the thinner coatings of each layer.

The second criteria to look for is the polarity of the analyte. For volatile, low molecular weight compounds,

Figure 4. Comparison of SPME Fiber Coatings – Extraction of Base-Neutrals, Polar Fraction

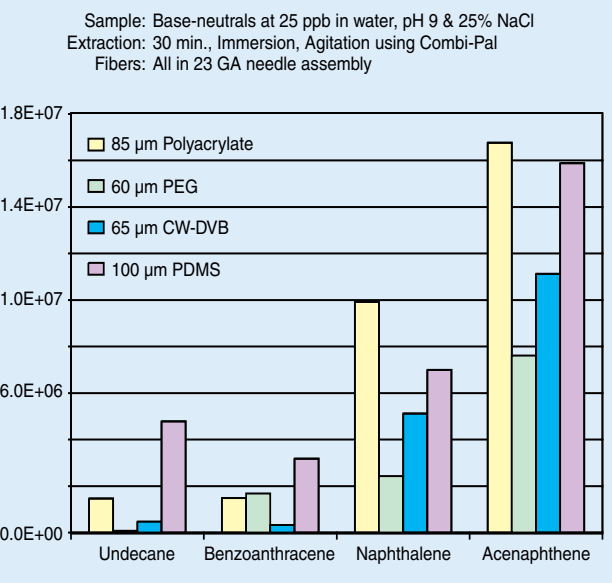


Carboxen-PDMS coated fibers are still the best choice. Once the molecular weight increases above 80 amu, the effect of fiber polarity becomes more evident for the extraction of polar analytes.

There are only 2 polar fiber coatings, however, there are some cases where other fibers will extract polar analytes. The 2 polar fibers are the 60 µm polyethylene glycol (PEG) phase and the 85 µm polyacrylate (PA) phase. Ideally you want polar fibers to be more selective, not only to extract polar analytes but also to repel non-polar analytes. Figures 4 and 5 show the comparison of 4 different SPME coatings for the extraction of

(continued on page 14)

Figure 5. Comparison of SPME Fiber Coatings – Extraction of Base-Neutrals, Neutral Fraction



(continued from page 13)

base-neutral analytes. Figure 4 shows the polar fraction of the analytes. In all cases the 60 μm PEG fiber was the best choice for these analytes. The PA fiber coating, slightly less polar than the PEG coating, is also suitable for the extraction of these analytes. The 100 μm PDMS fiber poorly extracted these polar analytes. Because counts were so low, in some cases the area response did not show on the graph, so the area counts are listed above the bar.

Figure 5 shows that in the neutral fraction, the analytes are extracted efficiently by all of the fibers as indicated by the higher area counts. Interestingly, the PA fiber, that likes aromatic compounds, was the best fiber for some of the neutral compounds. The PEG fiber as expected, being the most polar, extracted these analytes less efficiently than the other fibers. This indicates that the fiber does selectively extract polar analytes compared to the other SPME fibers.

+ Featured Products

Description	Cat. No.
SPME Fiber w/ 60 μm PEG Coating, 23 GA	
Autosampler, pack of 3	57354-U
Manual, pack of 3	57355-U

NEW! SPME Metal Fiber Assemblies

A flexible metal alloy in the needle, plunger, and fiber core gives this new design a thicker, flexible plunger that is much less likely to kink or break, and helps to reinforce the needle especially when used in conjunction with a sample agitator. Since the needle is more flexible and has a thinner wall than the standard stainless steel needle, a bevel has been placed on the needle to help it pierce septa materials more easily. The metal fiber assemblies increase fiber assembly life up to 10 times and improve fiber-to-fiber reproducibility.

For more information request T405058 (HXS) or visit sigma-aldrich.com/supelco-spme

SPME Metal Fiber Assemblies

Description	Cat. No.
7 μm PDMS	57919-U
30 μm PDMS	57922-U
100 μm PDMS	57928-U
65 μm PDMS/DVB	57902-U
85 μm Carboxen/PDMS	57906-U
50/30 μm DVB/Carboxen PDMS (1 cm)	57912-U
50/30 μm DVB/Carboxen PDMS (2 cm)	57914-U

The New 6th Edition SPME Applications CD (with Video Demonstrations)

SPME is a popular sample prep technique used by researchers worldwide for over 10 years. It has been one of the most referenced sample prep techniques in analytical chemistry winning an R&D 100 award from R&D Magazine. Many years ago we recognized that researchers would be interested in and might benefit from a comprehensive list of the SPME applications and we created the first SPME applications CD. Most researchers new to SPME feel the most useful part of the SPME Applications CD is this

applications reference list. Five updates later, the 6th edition CD has been updated to add over 600 new SPME application references (from approximately 18 months) bringing the total number of references to over 2200. Almost half of the new SPME references are in the area of food and flavor analysis with many in water analysis, biologicals and forensic analysis among many others. SPME is a very versatile technique, ideal for difficult sample matrices and remote site sampling.

Researchers unfamiliar with SPME are always curious how SPME works. What's new and unique about the 6th edition SPME CD is that we have added short video demonstrations on SPME that serve as a basic introduction for those unfamiliar with the technique. The demonstrations vary from "SPME use with an Autosampler" to "Getting Started with SPME". The demonstrations are not intended to be an in-depth overview of the technique as much as a basic introduction to what SPME is and how it works.

For your free copy of the 6th edition SPME CD please complete the attached postcard or visit us on the web at sigma-aldrich.com/supelco-spme



NEW! Molded Thermogreen™ LB-2 Septa

Michael D. Buchanan
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Introduction

Injection port septa used in gas chromatographic analyses should exhibit low bleed, resist leaks, and be easy to penetrate. For years, Thermogreen LB-2 septa have been considered by many as the benchmark GC septa for these parameters due to several performance advantages over other commercially available septa. A new generation septum, molded Thermogreen LB-2, continues the Supelco tradition of setting the benchmark in high performance.

Features and Benefits

Molded Thermogreen LB-2 septa are manufactured from high quality, low bleed material using the same exclusive rubber formulation as the popular Thermogreen LB-2 septa. The difference is that molded septa, unlike traditional die cut septa, offer easier installation and also provide a better seal inside the injection port. With a liquid injection molding process, every septum conforms to the same mold shape with crisp, clean sides.

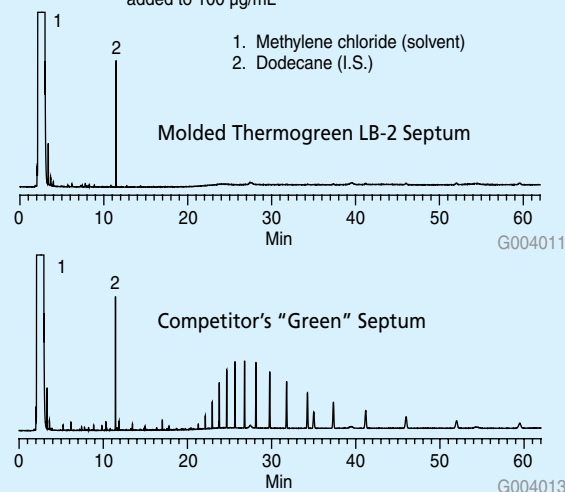
A liquid injection molding process allows injection holes to be incorporated into the septa. An injection hole allows needle penetration through the same location, time after time. This helps reduce septum coring, and prevents septum fragments from entering the injection port. The high puncture tolerance makes these septa ideal for autosamplers as well as users of solid phase microextraction (SPME).

Low Bleed Profile

A molded Thermogreen LB-2 septum and a popular molded septum from a competitor were solvent extracted. Both molded septa tested were a standard design without injection hole. The resulting chromatograms are shown in Figure 1. As seen, the competitor's "green" septum performed very poorly when subjected to this bleed test. The unlabelled peaks represent silicone oils that were extracted from the septum. When placed on an injection port and heated, these silicone oils would inevitably bleed off, collect on the head of the column, and appear as contamination peaks in chromatograms.

Figure 1. Bleed Profiles of 'High Performance' Septa

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 μ m (28471-U)
oven: 40 °C (3 min.), 15 °C/min. to 325 °C (15 min.)
inj.: 250 °C
det.: FID, 325 °C
carrier gas: helium, 25 cm/sec constant
injection: 1 μ L, splitless (1 min.)
liner: 4 mm I.D., single taper, unpacked
sample: solvent extracts of septa, dodecane (as an internal standard) added to 100 μ g/mL



Conclusion

The strict tolerances resulting from the constant dimensions of the mold itself result in septa that are easier to install and consistently fit better. Using a rubber formulation exclusive to Supelco, molded Thermogreen LB-2 septa exhibit an ultra low bleed profile, are very resistant to both slivering and coring, and have a high puncture tolerance when used in autosampler applications.

+ Featured Products

Diam. (mm)	Description	Qty.	Cat. No.
9.5 (3/8 in.)		50	28670-U
9.5 (3/8 in.)		250	28671-U
9.5 (3/8 in.)	with injection hole	50	28331-U
9.5 (3/8 in.)	with injection hole	250	28332-U
10 (13/32 in.)		50	28673-U
10 (13/32 in.)		250	28675-U
10 (13/32 in.)	with injection hole	50	28333-U
10 (13/32 in.)	with injection hole	250	28334-U
11 (7/16 in.)		50	28676-U
11 (7/16 in.)		250	28678-U
11 (7/16 in.)	with injection hole	50	28336-U
11 (7/16 in.)	with injection hole	250	28338-U

! Related Information

For more information on molded Thermogreen LB-2 septa, request T407082 (JQV). To request a no-charge sample pack, call Account Development at 877-787-4437.



Photoionization Detector (PID) Lamp Improvements

Carl Carelli¹ and Robert F. Wallace²

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2. Supelco, Bellefonte, Pennsylvania, USA, e-mail: bob.wallace@sial.com

Introduction

The photoionization detector (PID) lamp is a valuable tool for chromatographers and others investigating the presence of volatile organic compounds (VOCs). Major applications include laboratory analyses of air, water, and soil samples; on-site field monitoring of air, water, and soil; and personnel safety monitoring in confined spaces. In fact, it continues to be the preferred choice for detecting VOCs after nearly 50 years of use due to its fast response time and sensitivity; the PID can detect volatiles as low as 1 ppb. Recent improvement in both manufacturing processes and packaging should allow the PID to remain a top detector choice well into the future.

How the PID Lamp Works

The PID is a glow-discharge lamp, which when excited, releases photons into an ionization chamber. The combination of krypton gas and a magnesium fluoride lens (typical configuration) operates at wavelengths 116.6 nm and 123.6 nm, or the equivalent of 10.0 and 10.6 eV. An eV is an electron volt, which is the energy gained when a particle of one electronic charge is accelerated through a potential of one volt. The term eV is used as a convenient way to express the radiant strength of a PID lamp.

When the "lamp is lit" it radiates into the ionization chamber and passes between a pair of electrode plates. As the test gas moves into the radiated field between the electrodes, it is ionized and the free electrons are collected at the electrodes. This produces a current flow whose magnitude is in direct proportion to the gas concentration.

Each gas has its own ionization potential (IP), also expressed as an eV. Gases with an IP at or below the eV output of the lamp will be detected. The most popular PID is the 10.0/10.6 eV lamp, because it detects most VOCs, lasts longer, is easier to clean, and is less expensive.

Recent Lamp Improvements

Andrews Glass Co., a recognized leader in specialty glass products, has acquired the PID lamp product line from Scientific Services Co., pioneers of the PID lamp. Coupling the innovativeness and proprietary fabrication processes of Scientific Services Co. with the craftsmanship of Andrews Glass Co. allows the continued supply of the first, the best, and the broadest range of PID lamps.

Andrews Glass Co. now manufactures PID lamps in their ISO-compliant facility, providing the highest possible quality at critical points in manufacturing. Every step in the manufactur-

ing process has precise documentation to ensure the highest standards of quality in every PID lamp produced. Precision cutting, glass-to-metal sealing, and glass sealing all provide excellent protection against leaks. The use of a state-of-the-art plasma welder to achieve the critical metal-to-metal seal is inherent in lamp construction. This helps to eliminate any possibility of microscopic voids in the seal that would result in the slow leak of the krypton or xenon gas from the lamp. Precise control of dimensions also ensures a proper fit in the GC instrument.



With stringent quality control standards each lamp is tested and inspected prior to packaging to ensure they are in excellent operating condition. Quality and longevity tests are continually conducted to ensure the level of performance is maintained. The longevity tests are conducted under conditions simulating normal PID operation.

In addition to the changes in manufacturing, the way the lamps are packaged has been changed. PID lamps are now placed into plastic bags that contain a desiccant. The bag is then packaged into a cardboard form instead of a foam insert. Both these changes are designed to minimize exposure to moisture during shipment and storage to help extend the longevity of the PID lamp

Conclusion

Sensitivity and stability are the two primary areas of PID lamp performance that are most important to GC users. Recent changes in PID lamp production and packaging have been done to improve performance in these areas. PID lamps now being produced are very good and very consistent, from one lamp to the next.

+ Featured Products

Description	Chamber Gas	Cat. No.
Model 108-10.0/10.6 eV	Krypton	22626
Model 103C-10.0/10.6 eV	Krypton	22631
Model 108-BTEX-10.0/10.6 eV	Krypton	23129-U
Model 107-8.4 eV	Xenon	23132-U
Model 108-9.6 eV	Xenon	23133-U

+ Related Product

Description	Cat. No.
PID Window Cleaning Kit	22627

! Related Information

For more information on PID lamps, request T496088 (JTF). Available in electronic form only. Please provide email address on the attached postcard.

The Derivatization and Analysis of Amino Acids by GC-MS

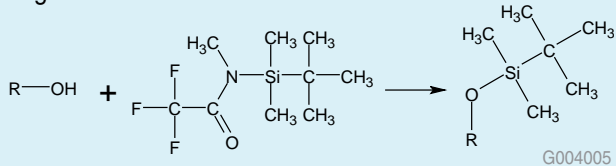
Katherine K. Stenerson
katherine.stenerson@sial.com

Introduction

Typically, high performance liquid chromatography (HPLC) is used for the analysis of amino acids. However, GC can also be used, and in some cases availability of instrumentation or operation costs can make it a better choice. The polar nature of amino acids requires derivatization prior to GC analysis. The goal of derivatization is to make an analyte more volatile, less reactive, and thus improve its chromatographic behavior. In the case of amino acids, derivatization replaces active hydrogens on OH, NH₂, and SH polar functional groups with a nonpolar moiety.

Silylation is a very common derivatization technique, and is useful for a wide variety of compounds. The main disadvantage of this method is its sensitivity to moisture. The presence of moisture results in poor reaction yield and instability of the derivatized analytes. For this study, we evaluated the use of the silylation reagent N-tert-butyltrimethylsilyl-N-methyltrifluoroacetamide (MTBSTFA) for the derivatization of amino acids. MTBSTFA, forms tert-butyl dimethylsilyl (TBDMS) derivatives when reacted with polar functional groups containing an active hydrogen:

Figure 1. Structure of MTBSTFA



MTBSTFA derivatives are more stable and less moisture sensitive than those formed using lower molecular weight reagents such as N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) (1).

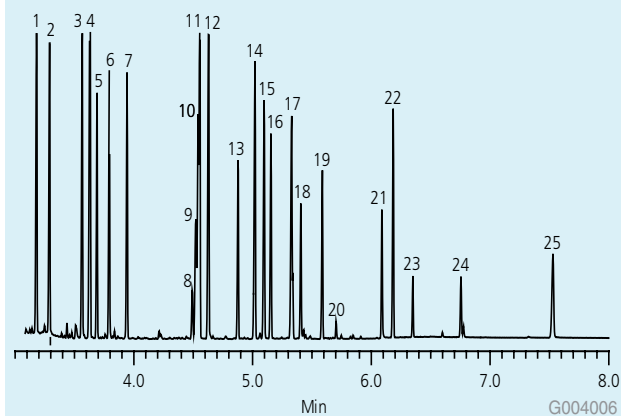
Experimental

A 50 μ L aliquot of a solution containing a mix of L-amino acids at 91 μ g/mL in 0.1 N HCl was dried, and 100 μ L of neat MTBSTFA, followed by 100 μ L of acetonitrile, were added. The mixture was heated at 100 $^{\circ}$ C for 4 hours. The sample was then neutralized with sodium bicarbonate and subjected to GC-MS analysis on a 20 m x 0.18 mm I.D. x 0.18 μ m SLB™-5ms capillary column.

Figure 2. GC-MS Analysis of Amino Acid Derivatives on the SLB-5ms

column: SLB-5ms, 20 m x 0.18 mm I.D., 0.18 μ m (28564-U)
oven: 100 $^{\circ}$ C (1 min.), 35 $^{\circ}$ C/min. to 290 $^{\circ}$ C (3 min.),
40 $^{\circ}$ C/min. to 360 $^{\circ}$ C
inj.: 250 $^{\circ}$ C
MSD interface: 325 $^{\circ}$ C
scan range: m/z = 40-450
carrier gas: helium, 1 mL/min., constant
injection: 0.5 μ L, splitless (1.0 min.)
liner: 2 mm I.D., straight
sample: TBDMS derivatives of amino acids,
each approximately 23 μ g/mL

- | | |
|--|--|
| 1. Alanine; m/z=260, 232, 158 | 14. Aspartic acid; m/z= 418, 390, 316 |
| 2. Glycine; m/z=246, 218 | 15. Hydroxyproline; m/z= 416, 388, 314 |
| 3. Valine; m/z= 288, 260, 186 | 16. Cysteine; m/z= 406, 378 |
| 4. artifact from derivatization | 17. Glutamic acid; m/z= 432, 330, 272 |
| 5. Leucine; m/z=302, 274, 200 | 18. Asparagine; m/z= 417, 302 |
| 6. Isoleucine; m/z=302, 274, 200 | 19. Lysine; m/z= 431, 329, 300 |
| 7. Proline; m/z= 286, 258, 184 | 20. Glutamine; m/z= 431, 357, 329, 299 |
| 8. Asparagine, extra derivative;
m/z= 327, 285, 243 | 21. Histidine; m/z= 459, 440, 338, 196 |
| 9. Glutamine, extra derivative;
m/z= 342, 300, 272 | 22. Tyrosine; m/z= 466, 438, 364, 302 |
| 10. Methionine; m/z= 320, 292, 218 | 23. Tryptophan, extra derivative
m/z= 417, 375, 347, 302, 273 |
| 11. Serine; m/z= 390, 362 | 24. Tryptophan; m/z= 489, 302, 244 |
| 12. Threonine; m/z= 404, 376, 303 | 25. Cystine; m/z=639, 589, 537, 348 |
| 13. Phenylalanine; m/z= 336, 302, 234 | |



Results

A chromatogram of the TBDMS derivatives of the amino acids is presented in Figure 2. Spectral data obtained from the peaks helped in identification of the amino acid derivatives. Replacement of an active hydrogen with a TBDMS group adds 114 to the molecular weight. Electron impact spectra (2) of these derivatives contains typical fragments corresponding to the molecular weight of the derivative less CH₃ (M-15), C₄H₉ (M-57), C₄H₉ + CO (M-85), and CO-O-TBDMS (M-159). Figure 3 shows an example of this fragmentation pattern in the spectrum of TBDMS-valine.

(continued on page 18)

(continued from page 17)

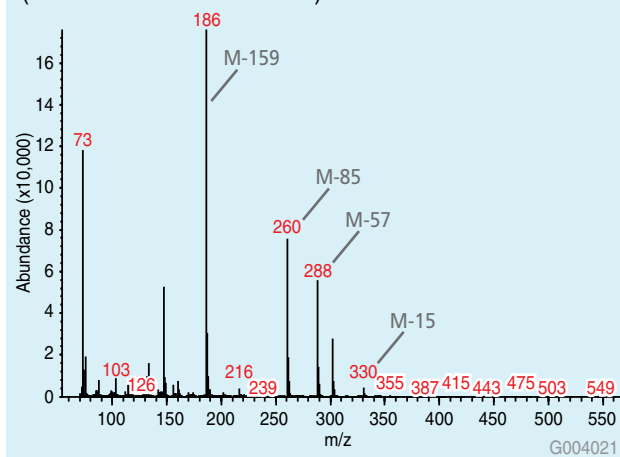
Under the reaction conditions used, the majority of the amino acids produced one derivative, with active hydrogens on hydroxyl, amine, and thiol groups (in the case of cysteine) replaced by TBDMS. Some amino acids produced multiple derivatives, specifically asparagine, glutamine, and tryptophan. In the case of these amino acids, a modification in the reaction conditions, such as, lowering the temperature or changing the reaction time, may prevent this from occurring (3). For example, increasing the reaction time from 2 to 4 hours resulted in an increased response of the fully derivatized form of tryptophan.

While TBDMS derivatives are more stable than traditional TMS derivatives, their higher molecular weights result in longer elution times during GC analysis. To balance this, the separation was done on a short, narrow bore capillary column. A starting temperature no higher than 100 °C was necessary to maintain resolution of the glycine derivative peak from the solvent. A quick ramp to 360 °C after the elution of the cystine derivative was performed to ensure the column was clean for subsequent analyses.

Conclusions

This study demonstrates that with the proper use of derivatization reagents such as MTBSTFA, amino acids can be analyzed by GC-MS. The reaction conditions may have to be "tweaked" to produce maximum response of the derivatives of interest. The derivatives produce characteristic fragments, allowing for easy identification by MS. To reduce the overall GC analysis time of these derivatives, a short, narrow bore column such as the 20 m x 0.18 mm I.D. x 0.18 µm SLB-5ms is recommended.

Figure 3. Mass Spectrum of TBDMS Derivative of Valine (MW of the derivative = 345)



References

1. T. G. Sobolevsky, A. I. Revelsky; Barbara Miller, Vincent Oriedo, E. S. Chernetsova, I.A. Revelsky, *J. Sep. Sci.* 2003, 26, 1474-1478.
2. F. G. Kitson, B. S. Larsen, C. N. McEwen, *Gas Chromatography and Mass Spectrometry, A Practical Guide*; Academic Press: San Diego, 1996; Chapter 9.
3. I. Molnar-Perl, Zs. F. Katona, GC-MS of amino acids as their trimethylsilyl/t-butyl-dimethylsilyl derivatives: in model solutions III. *Chromatographia Supplement*, 51, 2000, S228-S236.

Featured Products

Description	Cat. No.
SLB-5ms Capillary Column, 20 m x 0.18 mm I.D. x 0.18 µm	28564-U
N-tert-butyltrimethylsilyl-N-methyltrifluoroacetamide (MTBSTFA)	394882

Related Products

Description	Cat. No.
21 L-Amino acids plus glycine	LAA21-1kt
N-tert-butyltrimethylsilyl-N-methyltrifluoroacetamide with 1% tert-butyltrimethylchlorosilane, >95% (MTBSTFA plus TBDMCS)	375934

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NEW! 50 Component Indoor Air Monitoring Standard

Carpeting, cabinetry and furniture manufactured from pressed woods, paints, and household cleaning products are examples of the many materials known to off gas volatile organic compounds (VOCs) inside office buildings and homes, raising the indoor air pollution level to as much as 2 to 5 times that found in the outdoors. Exposure to these VOCs has been associated with eye, ears, and throat irritation, headaches, dizziness, and nausea. Some of the chemicals are also suspected or known to cause cancer in

humans. Consequently, both short- and long-term health effects continue to be studied.

Sigma-Aldrich is pleased to introduce a new indoor air standard for monitoring the presence of VOCs in the air and studying their impact on health. The mixtures are gravimetrically prepared and quantitatively analyzed by gas chromatography. A certificate of analysis accompanies each standard.

Description	Concentration	Pkg. Size	Cat. No.
50 Component Indoor Air Standard	100 µg/mL in methanol:water (95:5) 100 µg/mL in methanol:water (95:5) 1000 µg/mL in methanol:water (97:3)	1 x 1 mL 3 x 1 mL 1 x 1 mL	49148-U 4M9148-U 49149-U
Acetone	Ethyl acetate	2-Propanol	
Benzene	Ethylbenzene	Styrene	
Bromodichloromethane	2-Ethyltoluene	Tetrachloroethylene	
1-Butanol	3-Ethyltoluene	Tetradecane	
2-Butanol	4-Ethyltoluene	1,2,4,5-Tetramethylbenzene	
Butyl acetate	Heptane	Toluene	
Chloroform	Hexadecane	Trichloroethylene	
Decanal	Hexane	Tridecane	
Decane	(R)-(+)-Limonene	1,2,3-Trimethylbenzene	
Dibromochloromethane	4-Methyl-2-pentanone	1,2,4-Trimethylbenzene	
1,4-Dichlorobenzene	Nonane	1,3,5-Trimethylbenzene (mesitylene)	
1,2-Dichloroethane	1-Nonanol	2,2,4-Trimethylpentane	
Dichloromethane	Octane	Undecane	
1,2-Dichloropropane	Pentadecane	m-Xylene	
2,4-Dimethylpentane	(1S)-(-)-α-Pinene	o-Xylene	
Dodecane	(-)-β-Pinene	p-Xylene	
Ethanol	1-Propanol		

NEW! Solvent Standards for Evaluating Food Packaging

Residual solvents from inks and dyes used in packaging graphics can impart off flavors to food. In some cases, the residual solvents will penetrate the packaging material and be absorbed by the food product. To ensure consumer safety and customer satisfaction, the United States and the European Union have implemented regulations to address the use of inks and dyes in food packaging.

For compliance with these regulations, food manufacturers and food processors must investigate the barrier properties of new, innovative packaging with current food products, as well as the use of existing packaging

materials for new food products. Equally important is the adoption of an on-going in-house quality control program to monitor approved packaging for unexpected changes in barrier properties that could result in undesirable levels of residual solvent contamination.

Sigma-Aldrich has introduced two Supelco brand residual solvent standard mixtures to aid food chemists with these evaluations. All solvents have been screened for identity and purity. A certificate of composition accompanies each standard.

Description	Concentration	Pkg. Size	Cat. No.
Residual Solvents in Pkg Material Mix # 1	7.14% each (v/v)	1 x 1 mL	48994-U
n-Butyl acetate	Cyclohexanone	Methanol	
n-Butanol	Ethanol	Methyl acetate	
sec-Butanol	2-Ethoxyethanol	Methyl cellosolve acetate	
2-Butanone	Ethyl acetate	Toluene	
Cyclohexane	Isobutyl acetate		
Residual Solvents in Pkg Material Mix # 2	9.09% each (v/v)	1 x 1 mL	48995-U
Acetone	n-Propyl acetate	1-Propanol	
2-Ethoxyethyl acetate	2-Methoxyethanol	2-Propanol	
Isobutanol	1-Methoxy-2-propanol	Tetrahydrofuran	
Isopropyl acetate	4-Methyl-2-pentanone		

For a more complete list of analytical standards for air monitoring, visit our website sigma-aldrich.com/standards

NEW! Analytical Standards for Environmental Monitoring

Sigma-Aldrich is continuously adding new analytical standards for environmental monitoring through our Fluka, Riedel-de Haën, and Supelco brands. The decision on which new standards to introduce is based on customer input and changes in governmental regulatory methodologies.

Below are just a few of the recent Supelco brand additions. All raw materials and solvents have been screened for identity and purity. The mixtures are gravimetrically prepared and quantitatively analyzed by gas chromatography. A certificate of analysis accompanies each standard. Free data packets are available upon request.

Description	Concentration	Pkg. Size	Cat. No.
EPA 504.1 Calibration Solution	2000 µg/mL each in methanol	1 x 1 mL	49119-U
<i>1,2-Dibromo-3-chloropropane</i>	<i>1,2-Dibromoethane</i>	<i>1,2,3-Trichloropropane</i>	
EPA 522.2 Haloacetic Acids Mix	2000 µg/mL each in methyl-tert-butylether	1 x 1 mL	49107-U
<i>Bromoacetic acid</i>	<i>Chloroacetic acid</i>	<i>Dichloroacetic acid</i>	
<i>Bromochloroacetic acid</i>	<i>Chlorodibromoacetic acid</i>	<i>Tribromoacetic acid</i>	
<i>Bromodichloroacetic acid</i>	<i>Dibromoacetic acid</i>	<i>Trichloroacetic acid</i>	
EPA 8150B 2,4-Dichlorophenylacetic Acid Spike Mix	100 µg/mL each in acetone	1 x 5 mL 2 x 5 mL	49344-U 49343-U

Did you know...?

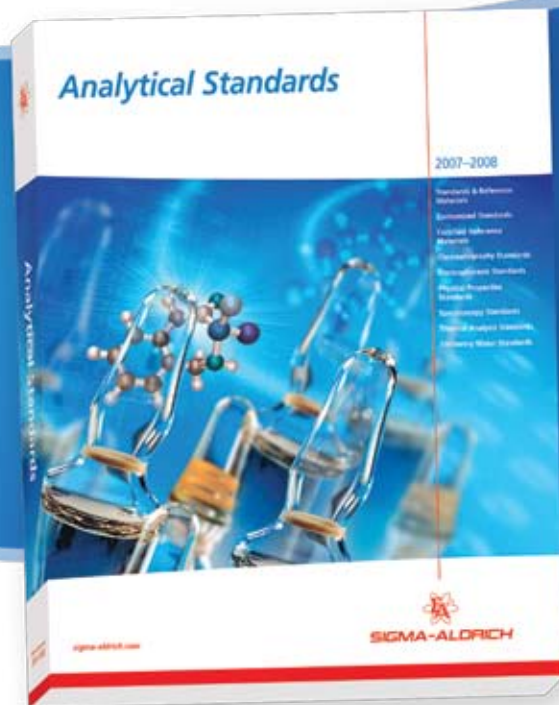
Supelco chemical standards are often shipped in flame-sealed ampuls. Once the ampul is open, the chemical is no longer properly stored. Immediately transfer the contents to a vial that can be properly sealed, and place the compound under specified storage conditions. Sigma-Aldrich offers a variety of vials, caps, and septa for properly storing a wide range of compounds. Consider using Mininert® push-button valves to allow for repeated entry into the vial, while maintaining a leak-tight seal. To learn more, visit sigma-aldrich.com/vials or email our Technical Services department at techservice@sial.com

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Super Clean™ Purifiers, Base-Plate Design

Robert F. Wallace
bob.wallace@sial.com

Introduction

Contaminants such as water and oxygen can enter carrier or other gas streams, adversely affecting the performance of a gas chromatographic system. UHP-grade gas normally contains less than 10 ppm of moisture and oxygen. Even though these concentration levels are normal, they can be detrimental to a GC system (especially the chromatographic column). To prevent damage and interference caused by contaminants, most manufacturers of gas chromatographs and ultra sensitive detectors recommend purification of all gases.

Super Clean Purifiers, Base-Plate Design



Super Clean purifiers' fast-connect technology allows today's chemist to change purifiers within seconds without disrupting the analytical performance of the gas chromatographic system. These purifiers do not connect directly into the gas line like a typical inline purifier, but onto a specially designed base-plate, allowing installation without the use of tools. The unique diffusion-proof glass/metal purifiers will purify delicate carrier, fuel, and other gases for GC and GC-MS systems.

E000938

Super Clean High Flow Purifiers



P001029

Super Clean high flow purifiers, a new revolution in clean gas purifiers, allow purification of gas at flows up to 20 L/min. The two-position base-plate incorporates 1/4 inch fittings to handle these high flows. An integrated particle filter on the upstream fitting ensures that the incoming gas stream is free of damaging dust. The diffusion-proof, fully glass/metal purifiers are arranged in parallel on the base-plate, doubling the adsorbent material

the gas must pass through. Two kits are available. The hydrocarbon kit is recommended for the purification of nitrogen going to the MS component in LC-MS systems. The moisture kit is recommended for the purification of hydrogen or zero air going to FIDs.

Advantages of Super Clean Base-Plate Purifiers

Purifier replacement with the Super Clean base-plate design is quick and easy when compared to traditional inline purifiers. The base-plate can be wall or table mounted for convenient positioning near the chromatograph. Regardless of how the base-plate is mounted, it requires far less valuable bench space than an inline design purifier. Features/benefits include:

- Specially designed base-plate has needle valves that instantly close to provide a diffusion-proof seal when the purifier is removed during change out
- Purifier replacement can be performed in seconds without exposing the gas lines to room air
- At 2 L/min. or less, these purifiers remove contaminants to produce better than 6.0 gas quality (99.9999% pure), independent of the purity of the incoming gas

Conclusion

Carrier gas purifiers are one of the safest ways to help prevent the lowest level of carrier gas contamination from entering the system. Super Clean purifiers, with their single- or multi-bed configurations, help to ensure the maximum protection for the system and longer lasting column life.



Related Information

For more information on Super Clean Purifiers, request T405139 (ILF) (base-plate design), T405137 (ILD) (high flow base-plate design), T405138 (ILE) (inline design), or visit our website: sigma-aldrich.com/sgtpurifier



Featured Products

Description	Fittings	Cat. No.
Super Clean Gas Purifiers, Base-Plate Kits		
Carrier (SU861026, SU861011)	1/8 inch	28878-U
Helium Carrier (SU861027, SU861011)	1/8 inch	SU861040
High Flow Hydrocarbon (SU861029, 28879-U)	1/4 inch	SU861046
High Flow Moisture (SU861028, 28879-U)	1/4 inch	SU861045
Replacement Single Bed Purifiers, Indicating		
Moisture	n/a	SU861021
Moisture, pack of 2	n/a	SU861028
Oxygen	n/a	SU861022
Replacement Single Bed Purifiers, Non-Indicating		
Hydrocarbon	n/a	SU861023
Hydrocarbon, pack of 2	n/a	SU861029
Replacement Multi-Bed Purifiers, Indicating		
Triple (hydrocarbon, moisture, oxygen)	n/a	SU861026
Triple, helium specific (hydrocarbon, moisture, oxygen)	n/a	SU861027
Dual (hydrocarbon, moisture)	n/a	SU861025
GC-FID (SU861026, two SU861025)	n/a	SU861043
Replacement Super Clean Base-Plates and Accessories		
Single position, brass fittings	1/8 inch	SU861011
Two position, brass fittings	1/8 inch	SU861012
High Flow, two position, brass fittings	1/4 inch	28879-U
Three position, brass fittings	1/8 inch	SU861013
Wall Mounting Bracket	n/a	SU861016
O-Rings (10 large, 10 small)	n/a	SU861050

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 2-22 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.



P000893

Capacity	Dimensions	PTFE/Silicone Septa Thickness In. (mm)	Pk Size	Cat. No.
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



P000376

Crimpers/Decappers

The Supelco hand-held crimper provides a consistent and dependable seal that allows secure vial closure each and every time. The high-quality construction is engineered for durability and long life to provide a smooth and simple operation. The adjustable stop accommodates a wide range of cap and septa thickness.

- Quality construction for durability and long life
- Consistent, dependable performance
- Smooth, simple operation
- Painted, plated or coated for maximum corrosion resistance
- High quality at a reasonable price



Description	Pack Size	Cat. No.
8 mm		
Hand crimper 8.0 mm	1	33272-U
Decapper, pliers-type, 8.0 mm cap	1	33284
Decapper for 8.0 mm caps	1	33180-U
11 mm		
Hand crimper, 1.0 mL, 11 mm cap	1	33195
Adjustable hand crimper, 1 mL, 11 mm cap	1	22313-U
Decapper, pliers-type, 11 mm cap	1	33281
Decapper for 11 mm caps	1	33181-U
13 mm		
Hand crimper, 2.0 mL, 13 mm cap	1	33279-U
Decapper, pliers-type, 13 mm cap	1	33282
Decapper for 13 mm cap	1	33182-U
20 mm		
Hand crimper, 5-100 mL, 20 mm cap	1	33280-U
Adjustable hand crimper, 5-100 mL, 20 mm cap	1	22316-U
Decapper, pliers-type, 20 mm cap	1	33283
Decapper for 20 mm caps	1	33183

An Inappropriate Crimp Can Be Recognized By:

	Undercrimped	Overcrimped	Overcrimped	Overcrimped	Overcrimped
Correct Crimp Flat cap surface Flat septa surface 					
Appearance	Loose aluminium edge	Upward bulge of the crimp cap	Deformation of the crimp cap sides	Convex looking liner	Rounded edges/upward bulge of the cap/liner
Solutions	Adjust crimping pressure with the screw in the handle + Adjust crimping height with the hexagon key	Adjust crimping pressure with the screw in the handle + Adjust crimping height with the hexagon key	Adjust crimping height with the hexagon key	Adjust crimping pressure with the screw in the handle	Especially with headspace caps it is important not to overcrimp them. If the aluminium is stretched too much under the crimp neck, the bridges of the scorelines suffer too much stress and can break open at even low pressure (below 3 bars) or - in worst case - can even tear apart beside the scorelines
Tight fitting of the aluminium edge Plain + undeformed cap sides					

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Glass Magnet Products

Our glass magnet sheet prevents your glass apparatus from being knocked over accidentally. This 2' x 2' sheet (61 cm x 61 cm), with its tacky surface, effectively secures the vessels in place. It can be cut with a razor blade or utility knife to fit strategically on counter tops or in drawers containing fragile glassware. Add it to carts when transporting glass to another lab or into the field.

The glass magnet vial holder keeps individual vials, ampuls, and other vessels in place.



Description	Cat. No.
Glass magnet sheet, 2' x 2' (61 x 61 cm)	57269
Glass magnet vial holder, 4" (10 cm) dia., pk. of 2	57270

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Economical and convenient storage for samples in your screw cap and crimp cap vials. Can be stacked when empty or full. Autoclavable, 120 °C. Each case contains 5 racks.



Vial Diam (mm)	No. Vials Per Rack	Rack Dimensions L x W x H (mm)	Cat. No.
6-8	96	183 x 125 x 23	23204-U
11-12	50	192 x 100 x 23	23207
15-16	48	268 x 95 x 30	23205-U
17	90	320 x 170 x 30	23202
22	36	320 x 90 x 30	23201
29	50 [▲]	336 x 175 x 30	23206

[▲] Will accommodate 40 mL EPA vial (Cat. No. 23189)

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