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2008.01

Focus on Performance

- Accurately quantify PAHs down to 5pg on-column using SIM analysis.
- Quantify benzodiazepines by LC/MS/MS at 10ng/mL in matrix in less than 10 minutes.
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- and much more inside.



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1 (of 32) 2008.1

IN THIS ISSUE

Editorial

Using Guard Columns and Retention Gaps in GC (Part 2) 2

Environmental

Accurately Quantify PAHs
Down to 5pg On-Column 3

13 Minute Chlorophenoxyacid
Herbicides Analysis 6

Enhancing Air Monitoring Methods
with Thermal Desorption 8

Chemical/Petrochemical

Selecting a GC Column for
Glycerin in Biodiesel 10

Stable Sulfur & Mercury
Sampling in Refineries 12

Foods, Flavors & Fragrances

High Sensitivity Melamine
|GC/MS Analysis of Cat Food 14

Sample Preparation

Fast, Simple Sample Cleanup 16

Pharmaceutical

Multi-task with an Ultra IBD Column 18

Clinical/Forensics

Fast, Sensitive Analysis of
Benzodiazepines by LC/MS/MS 20

Accurate, Reproducible
Amphetamines Analysis 22

Tech Tip

Selecting the Right HPLC
Guard Column 26

HPLC Accessories

Waste Overflow Indicator
for HPLC Systems 27

GC Accessories

The Forgotten Septum 24

Peak Performers
FID Detector Replacement Parts 28

Electron Multipliers for
Mass Spectrometry 30

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Using Guard Columns and Retention Gaps in GC (Part 2)

Jaap de Zeeuw, International GC Consumables Specialist, Restek Corporation



Guard columns and retention gaps are used widely in gas chromatography (GC). Many users have difficulty understanding the difference between these two products, even though there is a significant difference in application. In Part 1 of this article we reviewed retention gaps, which mainly are used for focusing the sample components when introducing a large (liquid) sample directly onto the column. In contrast, guard columns are used to protect the analytical column from contamination. Guard columns and retention gaps both must be coupled to the analytical column, and this connection introduces a potential point of risk. A new approach is to integrate the retention gap directly into the analytical column tubing. By applying a "segment" coating technology the stationary phase can be deposited only in a certain part of the column allowing a deactivated section at the beginning. Column coupling is not required and maintenance is greatly simplified. Here we will review guard columns and discuss the new segment coating technology.

Use of guard columns

The purpose of using guard columns is to protect the analytical column from contamination since the sample that is introduced is not always pure. Although the best chromatography is obtained with "clean" samples, the practical situation is that sample clean-up procedures are minimized and relative "dirty" samples are introduced onto the column. Samples can contain particulates, heavy components, derivatization reagents, ionic residues, acids, bases... all these compounds can interfere with the stationary phase and they will influence the separation process. Usually the degradation of column performance is a slow process but it will happen.

Most of the time the impurities accumulate in the first meter(s) of the column and by cutting off this section adequate separation is restored. Many users choose to connect a guard column in front of the analytical column. Such a guard column is deactivated and can be trimmed when contaminated and eventually replaced. Depending on the application, guard columns have a lifetime of 1 week up to 6 months. One has different choices for guard columns; a guard column can consist only of deactivated capillary, or it can be a coated capillary.

Deactivated capillary tubing: Deactivated fused silica tubing can be purchased by the meter and then a defined length can be coupled in front of the analytical column. Upon contamination, a section of the guard column is removed. When the whole guard is "consumed" a new guard column can be coupled. The disadvantage of cutting parts off of the guard column is that the column becomes shorter and this may affect retention times. However, if a similar length is always cut from the guard column, the change in retention time becomes very predictable. A deactivated guard column will also result in band focusing. If the injection is not optimal, there will be a focusing effect similar to that of a retention gap.

Coated capillary tubing: As the guard column needs to prevent contamination of the analytical column, a coated guard column can help as it has both the surface deactivation and also the stationary phase layer. The easiest and most economical way of using coated guard columns (or precolumns) is to buy two analytical columns. One we will use as a separation column and the second one will be used to make coated guard columns. From this second column we will cut 2m sections and couple a section in front of the analytical separation column. We can run our samples until contamination affects peak shape/response and then we can replace the guard with a new 2m section.

The system we have created will produce reproducible retention times as we always will replace the entire 2m coated guard column. Since the stationary phase is the same on the guard as on the analytical column, there will be no surprises. The coated guard column also will allow more aggressive samples/more contamination before it will give up. Lastly, we are able to cut 15 coated guard columns from a full 30m analytical column...that's also economical! However, if using a coated guard column, there will be no focusing effects.

Continued on page 31.

Accurately Quantify PAHs Down to 5pg On-Column

GC/MS SIM Analysis with the **New** Rxi®-5Sil MS Column

By Robert Freeman, Environmental Innovations Chemist

- Excellent linearity across a broad calibration range.
- Ideal for trace level analyses.
- Low bleed at high temperatures, for better overall response and lower detection limits.

Polycyclic aromatic hydrocarbons (PAHs) are common environmental pollutants, affecting air, water, and soil quality. Although naturally occurring, human impact has created a steady increase in environmental levels of PAHs and their byproducts. PAHs are typically formed through the incomplete combustion of organic materials, such as wood, coal, and oil, but are also used in manufacturing of some medicines, plastics, and pesticides. Many chromatographic methods are available to analyze these pollutants. Laboratories performing low-level PAH analyses often utilize the single ion monitoring (SIM) function of GC/MS because of the sensitivity required to achieve typical regulatory or monitoring levels.

Continued on page 4.

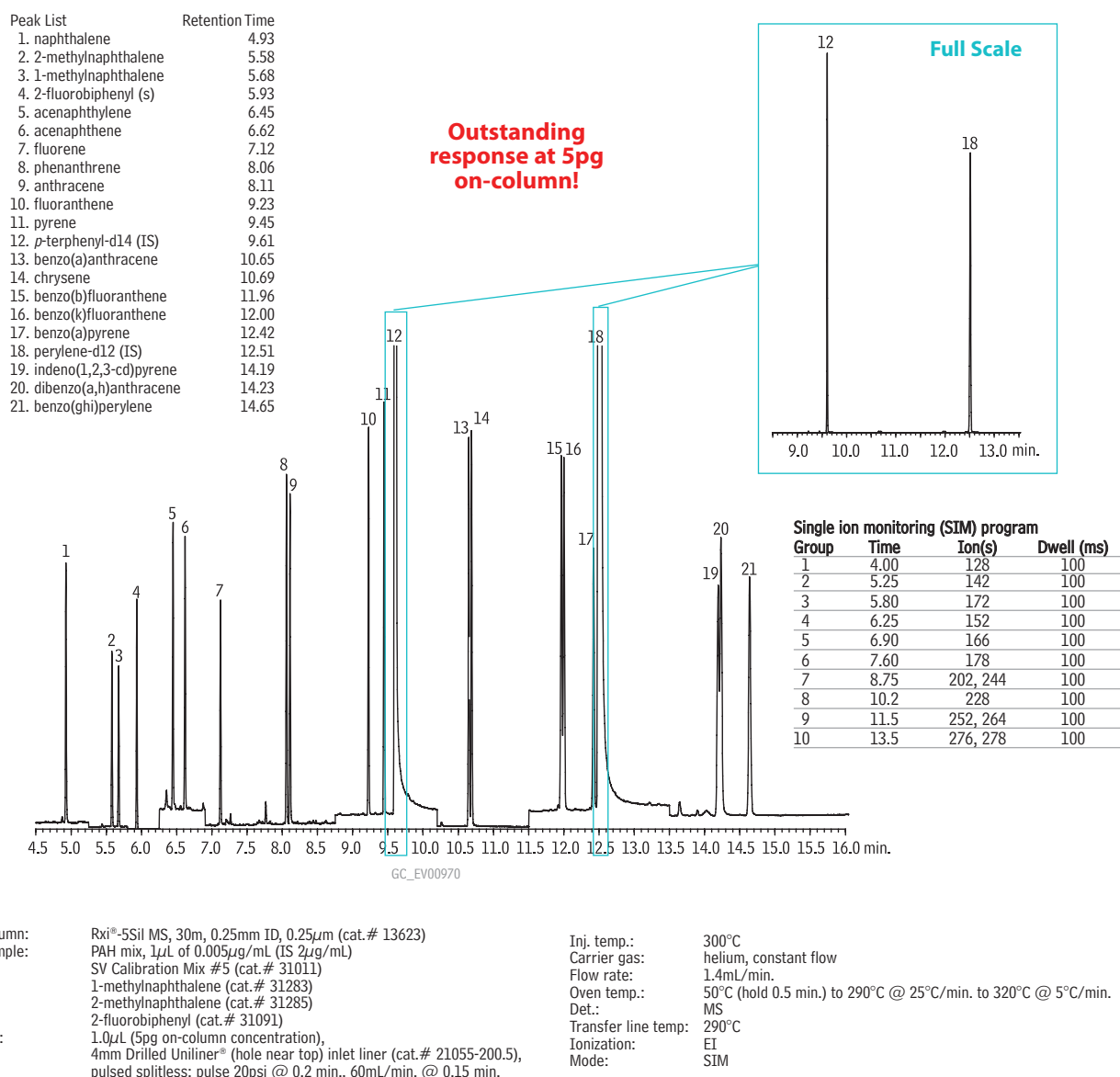
Accurately Quantify PAHs Down to 5pg On-Column *Continued from page 3.*

Method Parameters

For our SIM method we chose to use the new Rxi®-5Sil MS column. This stationary phase incorporates phenyl rings in the polymer backbone, which strengthens the siloxane chain, preventing thermal breakdown. This low bleed column is similar in selectivity to 5% diphenyl/95% dimethyl phases, but offers improved signal-to-noise ratios, resulting in increased sensitivity and subsequently lower detection limits. The silarylene polymer not only exhibits improved thermal stability and reduced bleed, but it also shows improved separation for aromatic compounds, such as PAHs.

Analytical conditions were set to optimize resolution of critical pairs and reduce discrimination of high molecular weight analytes. We chose a 4mm Drilled Uniliner® inlet liner with wool, since direct injection using this liner provides near complete transfer of sample analytes to the column. To improve the quantification of high molecular weight compounds we chose a thin film thickness (0.25µm) and set the injection port temperature to 300°C. A pulsed splitless injection technique was used to maximize the transfer of analytes onto the column. The pressure pulse is an effective injection technique for trace level analyses and also helps minimize discrimination against the high molecular weight components. Finally, the ion source and quadrupole temperatures were set at 290°C and 180°C, respectively. This increase in detector temperatures, from the defaults of 230°C and 150°C, yields better peak shapes and responses for the PAHs.

Figure 1 Excellent response and resolution of PAHs at 5pg on column in SIM mode



Results

These run conditions produced excellent resolution and response for all of the target analytes in a run time of less than 16 minutes. Figure 1 shows the SIM trace at 0.005µg/mL (5pg on column). The system was calibrated at eight levels, from 0.005 to 10µg/mL in single ion monitoring mode. The SIM acquisition program used for this analysis is shown in Figure 1. Each calibration standard contained eighteen target PAHs, two internal standards (*p*-terphenyl-d14 and perylene-d12), and the surrogate (2-fluorobiphenyl). At each level, the relative response factor (RRF) was calculated for all compounds and linearity was determined by calculating the percent relative standard deviation (%RSD) for all response factors, as shown in Table II. The %RSDs for all compounds are in the low single digits with an average for all compounds of 4.7%.

The Rxi®-5Sil MS column allows for a very broad calibration range, in this case 2000-fold from 5pg to 10ng while maintaining exceptional linearity. Using the Rxi®-5Sil MS column and an optimized temperature program is an excellent solution to the challenges posed by SIM PAH analyses.

Table I Relative response factors and %RSD for calibration standards (0.005-10µg/mL).

Compound	Relative Response Factor								Avg	%RSD
	0.005	0.01	0.05	0.1	0.5	1	5	10		
<i>p</i>-Terphenyl-d14 (IS)	-	-	-	-	-	-	-	-	-	-
Naphthalene	0.825	0.778	0.822	0.785	0.760	0.774	0.771	0.721	0.779	4.28
2-Methylnaphthalene	0.539	0.518	0.556	0.525	0.512	0.524	0.521	0.495	0.524	3.42
1-Methylnaphthalene	0.503	0.478	0.518	0.483	0.470	0.481	0.476	0.455	0.483	4.05
2-Fluorobiphenyl (SS)	0.689	0.664	0.691	0.680	0.664	0.679	0.669	0.608	0.668	3.93
Acenaphthylene	0.879	0.838	0.917	0.887	0.868	0.899	0.904	0.856	0.881	3.00
Acenaphthene	0.541	0.508	0.544	0.522	0.508	0.522	0.514	0.482	0.518	3.80
Fluorene	0.700	0.662	0.709	0.677	0.659	0.679	0.668	0.627	0.673	3.80
Phenanthrene	1.108	1.049	1.119	1.068	1.028	1.050	1.022	0.953	1.050	4.97
Anthracene	1.052	0.962	1.043	1.003	0.981	1.013	0.993	0.921	0.996	4.27
Fluoranthene	1.239	1.161	1.254	1.206	1.166	1.195	1.171	1.093	1.185	4.25
Pyrene	1.364	1.254	1.355	1.295	1.256	1.284	1.247	1.155	1.276	5.20
Perylene-d12 (IS)	-	-	-	-	-	-	-	-	-	-
Benzo(a)anthracene	1.111	0.980	1.086	1.054	1.048	1.087	1.090	1.017	1.059	4.12
Chrysene	1.153	1.041	1.116	1.073	1.057	1.078	1.043	0.951	1.064	5.59
Benzo(b)fluoranthene	1.282	1.039	1.183	1.146	1.139	1.185	1.204	1.144	1.165	5.92
Benzo(k)fluoranthene	1.327	1.119	1.223	1.189	1.183	1.229	1.225	1.136	1.204	5.35
Benzo(a)pyrene	1.037	0.967	1.146	1.083	1.038	1.089	1.134	1.080	1.072	5.36
Indeno(1,2,3-cd)pyrene	1.457	1.224	1.379	1.366	1.333	1.387	1.471	1.424	1.380	5.69
Dibenzo(a,h)anthracene	1.195	1.027	1.150	1.180	1.094	1.164	1.233	1.173	1.152	5.56
Benzo(ghi)perylene	1.331	1.118	1.238	1.263	1.140	1.192	1.244	1.190	1.215	5.68

SV Calibration Mix #5 / 610 PAH Mix

(16 components)

acenaphthene	chrysene
acenaphthylene	dibenzo(a,h)anthracene
anthracene	fluoranthene
benzo(a)anthracene	fluorene
benzo(a)pyrene	indeno(1,2,3-cd)pyrene
benzo(b)fluoranthene	naphthalene
benzo(k)fluoranthene	phenanthrene
benzo(ghi)perylene	pyrene

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31011

1-Methylnaphthalene

1,000µg/mL in methanol, 1mL/ampul
cat. # 31283

2-Methylnaphthalene

1,000µg/mL in methylene chloride, 1mL/ampul
cat. # 31285

2-Fluorobiphenyl

2,000µg/mL in methylene chloride, 1mL/ampul
cat. # 31091

Rxi®-5Sil MS Columns (fused silica)

(Crossbond®, selectivity close to 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	13623

Direct Injection Liners for Agilent GCs (For 0.25/0.32/0.53mm ID Columns)

ID* x OD & Length (mm)	qty.	cat.#
Drilled Uniliner® (hole near top)		
4.0 ID x 6.3 OD x 78.5	ea.	21054
4.0 ID x 6.3 OD x 78.5	5-pk.	21055
4.0 ID x 6.3 OD x 78.5	25-pk.	20998

Drilled Uniliner® (hole near top) w/ Wool

4.0 ID x 6.3 OD x 78.5	ea.	21054-200.1
4.0 ID x 6.3 OD x 78.5	5-pk.	21055-200.5
4.0 ID x 6.3 OD x 78.5	25-pk.	20998-214.25

*Nominal ID at syringe needle expulsion point.



NEW—
Optimized Film
Thicknesses!

13 Minute Chlorophenoxyacid Herbicides Analysis

On New Rtx®-CLPesticides & Rtx®-CLPesticides2 Columns

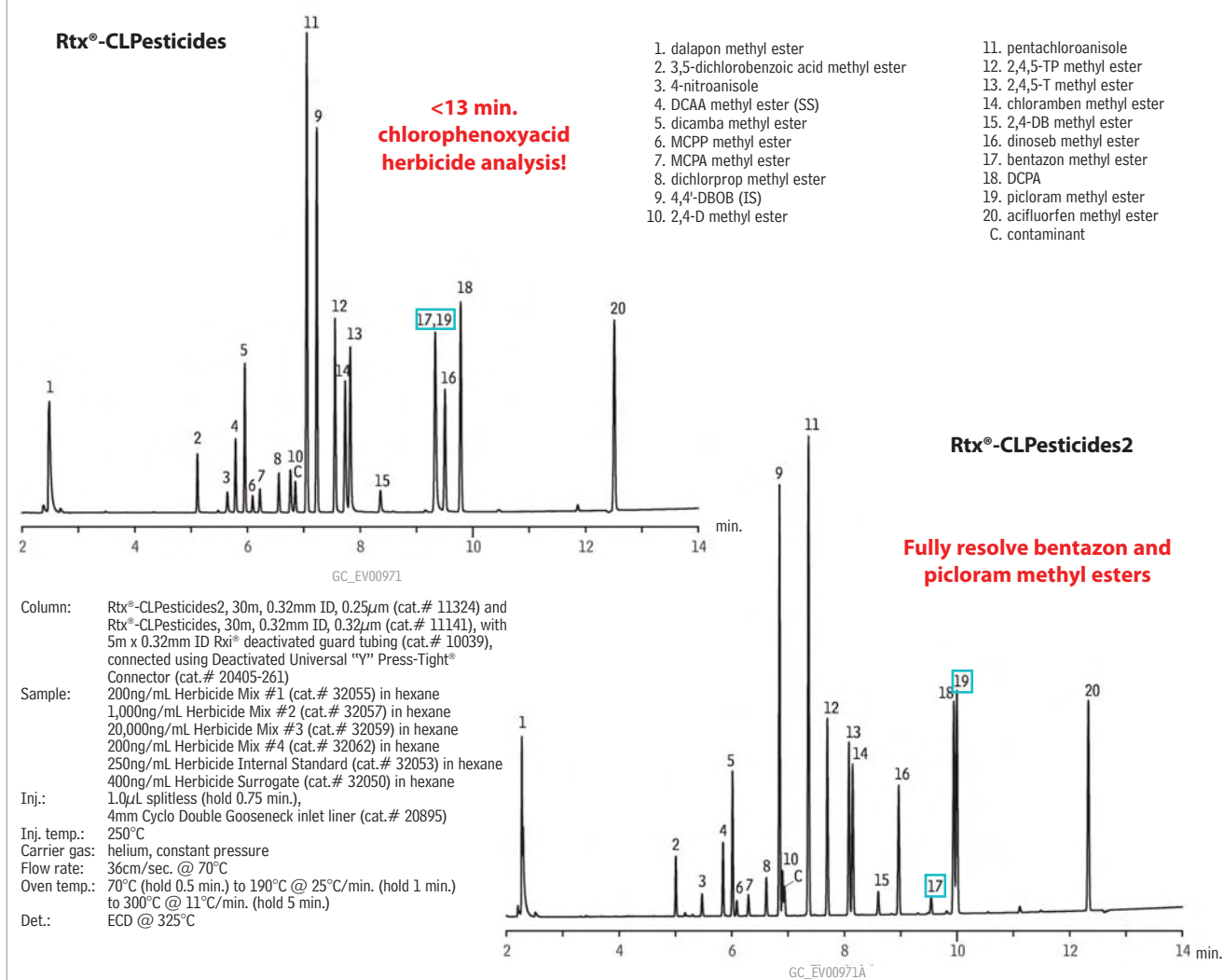
By Jason Thomas, Environmental Innovations Chemist

- Higher throughput compared to typical methods of 20 minutes or more.
- Use one column pair for multiple dual column ECD methods.
- Versatility and durability to harsh samples lead to longer life and less down time.

The analysis of chlorophenoxyacid herbicides is a very common assay performed routinely in most environmental laboratories today. Chlorophenoxyacid herbicides, as a group, are used to prevent the growth of broadleaf plants in agricultural fields. EPA Method 8151A is commonly used for chlorophenoxyacid herbicide analysis and involves extraction and derivatization to methyl ester form. GC analysis using an electron capture detector (ECD) is the analytical procedure of choice, although mass spectrometry is also used. ECD detection requires the use of second column confirmation for quantification of target analytes.

The Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair is an excellent choice for chlorophenoxyacid analysis. Now, with an optimized film thickness for the 0.32mm ID version, this difficult analysis can be made in less than 13 minutes on both the primary and confirmation columns. Near baseline resolution is achieved for all analytes except for bentazon/picloram on the Rtx®-CLPesticides column; however, this pair is fully resolved on the Rtx®-CLPesticides2 column (Figure 1). The Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair is an excellent choice for chlorophenoxyacid herbicide analysis due to the unique selectivity, low bleed, and durability of the columns. The Rtx®-CLPesticides column pair can also be used for other environmental ECD methods, including chorinated pesticide analysis.

Figure 1 Excellent resolution of chlorophenoxyacid herbicides on the Rtx®-CLPesticides column pair.



Rtx®-CLPesticides Columns (fused silica)

ID	df (μm)	temp. limits	length	cat. #
0.32mm	0.32	-60 to 320/340°C	30-Meter	11141

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (μm)	temp. limits	length	cat. #
0.32mm	0.25	-60 to 320/340°C	30-Meter	11324

Rxi® Guard/Retention Gap Columns (fused silica)

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter	10-Meter/6-pk.
0.32mm	0.45 ± 0.04mm	10039	10039-600	10064	10064-600

Universal "Y" Press-Tight® Connectors

An alternative method of performing dual-column confirmational analyses!

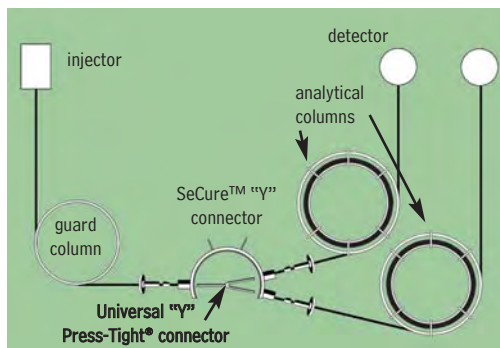
Description	ea.	3-pk.
Universal "Y" Press-Tight® Connector	20405	20406
Deactivated Universal "Y" Press-Tight® Connector	20405-261	20406-261
Siltek® Treated Universal "Y" Press-Tight® Connector	20485	20486



Dual-column confirmational analysis with a single injection—one of the SeCure™ "Y" connector's many uses.

for **more** info

For more information on Restek's Secure™ "Y" connector, download a free copy of lit. cat. #598788A from www.restek.com.

**Herbicide Mix #1** (7 components)**Derivatized Form:**

2,4-D methyl ester	dicamba methyl ester
2,4-DB methyl ester	dichlorprop methyl ester
2,4,5-T methyl ester	dinoseb methyl ether
2,4,5-TP methyl ester	

200μg/mL each in hexane, 1mL/ampul
cat. # 32055

Herbicide Mix #2**Derivatized Form:**

dalapon methyl ester
2,000μg/mL in hexane, 1mL/ampul cat. # 32057
1,000μg/mL in methanol, 1mL/ampul cat. # 32254

Herbicide Mix #3**Derivatized Form:**

MCPA methyl ester	MCCP methyl ester
20,000μg/mL each in hexane, 1mL/ampul cat. # 32059	

Herbicide Mix #4 (8 components)**Derivatized Form:**

acifluorfen methyl ester	methyl ester
bentazon methyl ester	4-nitroanisoole
chloramben methyl ester	pentachloroanisoole
DCPA (Dacthal®)	picloram methyl ester
3,5-dichlorobenzoic acid	
200μg/mL each in hexane, 1mL/ampul cat. # 32062	

Splitless Liners for Agilent GCs

ID* x OD & Length (mm)	qty.	cat. #
Cyclo Double Gooseneck (4mm)		
4.0 ID x 6.5 OD x 78.5	ea.	20895
4.0 ID x 6.5 OD x 78.5	5-pk.	20896
4.0 ID x 6.5 OD x 78.5	25-pk.	20997

*Nominal ID at syringe needle expulsion point..

Herbicide Internal Standard

4,4'-dibromooctafluorobiphenyl
250μg/mL in hexane, 1mL/ampul cat. # 32053
2,000μg/mL in methylene chloride, 1mL/ampul cat. # 31040
2,000μg/mL in methyl <i>tert</i> -butyl ether, 1mL/ampul cat. # 31856

Herbicide Surrogate**Derivatized Form:**

2,4-dichlorophenyl acetic acid methyl ester (DCAA methyl ester)
200μg/mL in hexane, 1mL/ampul cat. # 32050

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Pesticides"

"Choosing a Liner for
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Enhancing Air Monitoring Methods with Thermal Desorption

Advantages Over Solvent Extraction Tubes

By Liz Woolfenden, Director, Markes International, UK, and Irene DeGraff, Product Marketing Manager

- Accurately monitor down to ppb/ppt levels.
- Use thermal desorption tubes for either active or passive sampling, without modification.
- Compliant with air sampling methods.

The use of active sampling onto glass tubes packed with charcoal, followed by carbon disulfide (CS₂) extraction and gas chromatography (GC) analysis, was developed as an air monitoring method for vapor-phase organic compounds (VOCs) in the 1970s. The approach is still used today for some personal exposure assessment (occupational hygiene) applications and stack emission testing, but is fundamentally limited with respect to detection limits. Thermal desorption (TD) is a complementary gas extraction technique whereby sorbent tubes (Figure 1) are heated in a flow of carrier gas. Trapped vapors desorb from the sample tubes into the gas stream and are transferred into the GC/MS for analysis. Here, we summarize the key advantages of thermal desorption versus solvent extraction.

Sensitivity & Reproducibility

Solvent extraction of charcoal tubes requires at least 1 or 2ml of CS₂ followed by injection of only 1-2µl of extract into the GC/MS, resulting in a 1000-fold dilution of the sample right at the start of the process. Conversely, thermal desorption allows complete transfer of all target analytes to the analytical system, with no dilution or solvent interference. Detection limits offered by thermal desorption methods facilitate ambient monitoring at ppt/ppb levels as well as higher ppm (and %-level) concentrations. In addition to high sensitivity, thermal desorption is highly reproducible, offering efficiency greater than 95%, regardless of ambient conditions and the nature of the target analytes. By comparison, results from solvent desorption tubes may be highly variable.

Passive Sampling Option

While thermal desorption tubes are used extensively for active air sampling, they are also compatible with low-cost passive sampling. Passive samplers eliminate the requirement for personal monitoring pumps making them much less heavy/intrusive. Instead of a pump, each tube is simply fitted with a diffusion cap at the sampling end.

Repeat Analysis & Method Compliance

The historical advantages of solvent desorption tubes over thermal desorption, such as multiple sample injection and method compliance, no longer hold true. Since the advent of the SecureTD-Q™ thermal desorption unit, quantitative re-collection of split flow during both tube and trap desorption is possible. The utility of quantitative sample re-collection for repeat TD-GC/MS analysis has recently been recognized in standard methods as an aid to TD method/data validation.¹ Well-validated thermal desorption methods for many applications are now available from all the major international standards agencies. Key examples include: EN ISO 16017, ISO 16000-6, ASTM D-6196, US EPA Method TO-17, NIOSH 2549, MDHS 72, 80, etc. (UK) and EN 14662.

Conclusion

Thermal desorption technology offers several significant advantages over conventional solvent extraction. TD systems offer better sensitivity, desorption efficiency, and reproducibility compared to charcoal/CS₂ systems. Additionally, tubes may be used for both passive and active sampling without modification. These benefits, in combination with SecureTD-Q™ technology, which allows repeat analysis, make thermal desorption an excellent choice for many air monitoring applications.

References

1. ASTM D6196-03

Figure 1 A selection of thermal desorption air sampling tubes from Restek's new line.



Thermal Desorption Unit (TDU) Tubes

- Variety of sorbents to collect a wide range of VOCs.
- Use glass tubes for maximum inertness in active sampling.
- Choose stainless steel tubes for either active or passive sampling. No sampling pump necessary for passive sampling with diffusion caps!
- Individually etched with unique serial number for convenient sample identification.
- Available unconditioned or preconditioned and ready to sample. Tubes are Reusable after thermal desorption.

High-quality thermal desorption tubes by Markes International are now available from Restek. These sorbent tubes are suitable for ppt to ppm concentrations of volatile organic compounds (VOCs) in ambient, indoor, and industrial hygiene environments. Available in both stainless steel and glass (for thermally labile VOCs), they fit Markes ULTRA-UNITY, PerkinElmer, and Shimadzu thermal desorbers. Packed tubes come with a report detailing the total mass of sorbent in the tube; conditioned tubes also include a blank chromatogram.

Thermal Desorption Tube Sorbent	Applications
Tenax TA	Vapour phase organics from C6/7 to C26
Graphitized Carbon	Vapour phase organics from C5/6 to C14
Tenax GR/Carbopack™ B	Vapour phase organics from <i>n</i> -C5/6 to <i>n</i> -C20 (EPA Methods TO-14/TO-15/TO-17)
Carbopack™ B/Carbosieve™ SIII	Vapour phase organics from <i>n</i> -C2/3 to <i>n</i> -C12/14 (EPA Methods TO-14/TO-15/TO-17)
Tenax TA/Graphitized Carbon/Carboxen™ 1000	Vapour phase organics from C2/3 to C20
Carbopack™ C/Carbopack™ B/Carbosieve™ SIII	Vapour phase organics from <i>n</i> -C2/3 to <i>n</i> -C16/20 (EPA Methods TO-14/TO-15/TO-17)



method applications

Method	Application
US EPA	TO-17
ASTM	D-6196
NIOSH	2549
DIN EN ISO	16017

Specifications

Dimensions: 1/4" OD x 3-1/2" Long
 Low sampling rates:
 0.01-0.20 L/min.
 (<10L total volume)
 Long-term storage caps are supplied with conditioned tubes

Thermal Desorption Unit Tubes, Unconditioned and Conditioned & Capped

Description	qty.	Unconditioned		Conditioned & Capped	
		Stainless Steel	Glass	Stainless Steel	Glass
		cat. #	cat. #	cat. #	cat. #
TDU Tubes, Tenax TA	10-pk.	24056	24062	24080	24086
TDU Tubes, Graphitized Carbon	10-pk.	24057	24063	24081	24087
TDU Tubes, Tenax GR/Carbopack™ B	10-pk.	24058	24064	24082	24088
TDU Tubes, Carbopack™ B/Carbosieve™ SIII	10-pk.	24059	24065	24083	24089
TDU Tubes, Tenax TA/Graphitized Carbon/Carboxen™ 1000	10-pk.	24060	24066	24084	24090
TDU Tubes, Carbopack™ C/Carbopack™ B/Carbosieve™ SIII	10-pk.	24061	24067	24085	24091



Stainless Steel,
Conditioned and Capped

Thermal Desorption Unit Tubes, Empty

Description	qty.	Stainless Steel	Glass
		cat. #	cat. #
TDU Tubes, Empty	10-pk.	24054	24055



Glass, Unconditioned

Thermal Desorption Unit Tubes, Calibration

Description	qty.	Stainless Steel	Glass
		cat. #	cat. #
TDU Tubes, Calibration, Tenax TA 1cm Bed	10-pk.	24075	24076
Calibration Solution Loading Rig	ea.		24077
Calibration Solution Loading Rig 9.5mm Replacement Septa	10-pk.		24078
Certified Reference Standard, 100ng BTX on Tenax TA	10-pk.		24079



Stainless Steel,
Unconditioned

Thermal Desorption Unit Tubes, Accessories

Description	Benefits/Uses	qty.	cat.
1/4" Brass Cap and PTFE Ferrules	Use for long-term storage of blank/sampled tubes.	20-pk.	24068
1/4" PTFE Ferrules	Long-term storage caps.	20-pk.	24069
CapLok Tool	Use for tightening long-term storage caps.	ea.	24070
Pen Clip		10-pk.	24071
TubeMate Tool	Assists with tube packing.	ea.	24072
1/4" Stainless Steel Union and PTFE Ferrules	Use for connecting tubes in series.	10-pk.	24073
Diffusion Caps	Required for diffusive sampling with stainless steel tubes.	10-pk.	24074



CapLok Tool



Diffusion Caps

Selecting a GC Column for Glycerin in Biodiesel

By Barry Burger, Petroleum Innovations Chemist, and Gary Stidsen, Product Marketing Manager

- Choose metal MXT®-Biodiesel TG columns for high temp. conditions; low bleed and leak-proof for more accurate results.
- Use Rtx®-Biodiesel TG columns up to 380°C when fused silica is desired; reliable, low bleed performance.
- Innovative Alumaseal™ and Integra-Gap™ technology; choose Restek for leak-proof retention gap options.

Comparing Fused Silica and Metal Columns

Fused silica columns traditionally have been used for GC biodiesel analysis, but metal columns offer significant performance advantages. How can analysts determine which column is best for their lab? Here we compare fused silica and metal column performance for total glycerin analysis of biodiesel and offer guidelines for column selection.

Excellent chromatography can be obtained using Rtx®-Biodiesel TG fused silica columns. However, for high temperature work (>380°C) metal columns are much more rugged because the polyimide resin used to make fused silica hardens at high temperatures, making columns brittle and producing active sites in the column. To maximize column lifetime, tubing choice should be based on the maximum temperature setting in the GC temperature program. If the temperature program will be 400°C or lower, high temperature fused silica tubing is an acceptable choice; for GC temperatures that will exceed 400°C, metal tubing should be used.

Rtx®-Biodiesel TG Fused Silica Columns

Two fused silica GC column dimensions are available for the analysis of total glycerin: 10m x 0.32mm ID or 15m x 0.32mm ID, both of which are connected to a 2m x 0.53mm ID retention gap for cool on-column injection. The retention gap is factory coupled using Restek's unique Alumaseal™ connector (Figure 1). This innovative connector is leak-tight and low dead volume, making it advantageous for high temperature work.

Metal Column Solutions: Two Options for Increased Stability and Performance

- 0.32mm MXT®-Biodiesel TG columns with factory-connected retention gaps.
- 0.53mm MXT®-Biodiesel TG columns with built-in retention gaps.

The primary advantage of using metal MXT® columns is that they are more stable at high temperatures than fused silica columns. This means they will exhibit lower bleed, improving analytical performance, and have longer lifetimes, making them a cost-effective option. High temperature tolerance also means these columns can be brought to high temperatures (430°C) allowing nonvolatile material to be baked off of the column. MXT®-Biodiesel TG columns are available in the same dimensions as their fused silica counterparts:

Figure 1 The Alumaseal™ connector

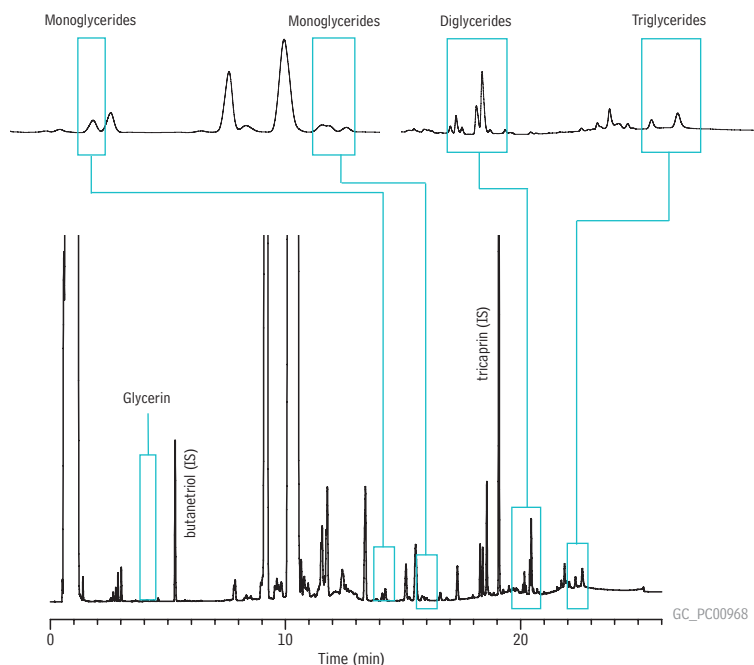
The Alumaseal™ connector is the best column connector for coupling fused silica and metal columns, even columns of different internal diameters. Made of aluminum, it is designed for high temperature performance. These connectors have been factory-coupled and tested using temperature programmed mass spectrometry and have shown no signs of leaks, even at 430°C.

The Alumaseal™ connector offers:

- A leak-tight connection.
- Low dead volume.
- Low thermal mass.
- High inertness.



Figure 2 Derivatized B100 samples resolve well on the 15m x 0.32mm MXT®-Biodiesel TG column, which is factory coupled to a 0.53mm retention gap using an Alumaseal™ connector.



Column: MXT®-Biodiesel TG, 15m, 0.32mm ID, 0.10µm (cat.# 70291) with a 2m x 0.53mm MXT® retention gap connected with an Alumaseal™ connector (17m total length)

Sample: biodiesel (B100), derivatized

Inj.: cool on-column injection

Inj. temp.: 1µL in heptane

Carrier gas: oven track

Flow rate: hydrogen, constant flow

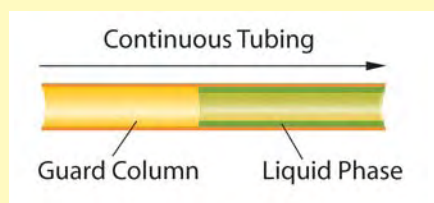
Oven temp.: 3mL/min.

Det.: 50°C (hold 1 min.) to 180°C @ 15°C/min. to 230°C @ 7°C/min. to 380°C @ 30°C/min. (hold 5 min.)

FID @ 380°C

Figure 3 The Ultimate Biodiesel Solution: MXT®-Biodiesel TG column with Integra-Gap™ integrated retention gap.

The 0.53mm MXT®-Biodiesel TG columns are an innovative alternative to using a 0.32mm column coupled to a 0.53mm retention gap. Restek applied the Integra-Gap™ integrated retention gap technology to the 0.53mm MXT®-Biodiesel TG columns, eliminating the column coupling. These 100% leak-proof columns feature a built-in retention gap, reducing the risk of peak broadening and tailing, and guaranteeing the user many analyses without downtime.



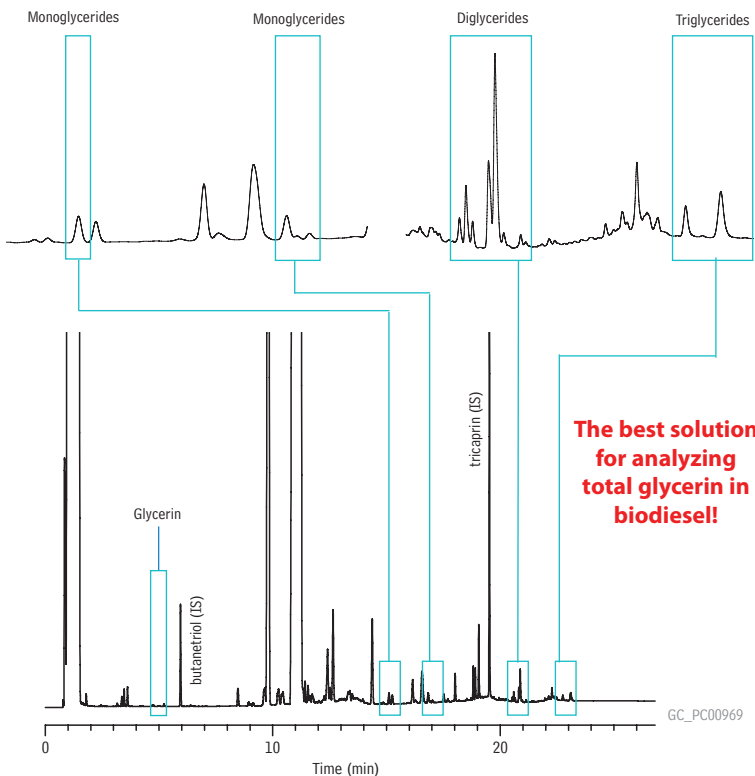
10 x 0.32mm ID and 15m x 0.32mm ID, both of which are factory coupled to a 2m x 0.53mm retention gap using an Alumaseal™ connector. Excellent resolution of all glycerides is achieved, as shown in Figure 2.

Restek has also developed an innovative column where the analytical column includes a built-in retention gap in a continuous section of tubing, requiring no connectors. This column, the MXT®-Biodiesel TG column, is 14m x 0.53mm ID, and features a 2m x 0.53mm ID Integra-Gap™ integrated retention gap (Figure 3). This product eliminates any need for connections because the column and retention gap are one piece of continuous tubing. Target analytes resolve exceptionally well and the solvent and triglyceride peaks show excellent symmetry (Figure 4). Peak shape for butanetriol is very good, demonstrating inertness, and the resolution and response of the glycerides is also excellent.

Conclusion

There are several column options available for analyzing total glycerin in biodiesel fuels. The best chromatographic solution for this analysis is the 14m x 0.53mm ID MXT®-Biodiesel TG column with the 2m x 0.53mm ID Integra-Gap™ integrated retention gap. This column eliminates the column connection and can be used to 430°C allowing for faster analysis times and higher sample throughput.

Figure 4 Excellent chromatographic quality and resolution on the 0.53mm MXT®-Biodiesel TG column, with the Integra-Gap™ integrated retention gap.



Column: MXT®-Biodiesel TG, 14m, 0.53mm ID, 0.16µm (cat.# 70289) with a 2m x 0.53mm Integra-Gap™ retention gap (16m total length)
 Sample: biodiesel (B100), derivatized
 Inj.: cool on-column injection
 Inj.: 1µL in heptane
 Inj. temp.: oven track
 Carrier gas: hydrogen, constant flow
 Flow rate: 4mL/min.
 Oven temp.: 50°C (hold 1 min.) to 180°C @ 15°C/min. to 230°C @ 7°C/min. to 380°C @ 30°C/min. (hold 5 min.)
 Det.: FID @ 380°C

MXT®-Biodiesel TG Columns (Siltek® treated stainless steel)

Description	temp. limits	cat.#
14m, 0.53mm ID, 0.16 w/2m Integra-Gap™	-60 to 380/430°C	70289
10m, 0.32mm ID, 0.10	-60 to 380/430°C	70292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70290
15m, 0.32mm ID, 0.10	-60 to 380/430°C	70293
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70291

**Connected with low-dead-volume Alumaseal™ connector.

Rtx®-Biodiesel TG Columns (fused silica)

Description	temp. limits	cat.#
10m, 0.32mm ID, 0.10	to 330/380°C	10292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10291
15m, 0.32mm ID, 0.10	to 330/380°C	10294
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10293

**Connected with low-dead-volume Alumaseal™ connector.



Stable Sulfur & Mercury Sampling in Refineries

Using Siltek® and Sulfinert® Surface Treated Components

By Gary Barone, Restek Performance Coatings, and Irene DeGraff, Product Marketing Manager

- Reliably sample sulfur and mercury compounds at ppb levels.
- Reduce lab costs—obtain accurate results the first time.
- Detect costly process upsets, improving product yield.

Refinery and natural gas samples often contain trace amounts of sulfur- and mercury-containing compounds, which can interfere with reactions, poison catalysts in petrochemical processes, and damage equipment. Because these compounds quickly react with stainless steel surfaces, accurate determination of these compounds is impossible when samples are collected and stored in untreated sample cylinders. Restek's Siltek® and Sulfinert® passivation techniques bond an inert layer into the surface of stainless steel, preventing active compounds from reacting with or adsorbing to the steel.

Accurate sulfur sampling

To characterize Sulfinert® surfaces, we tested the stability of 17ppbv standards of sulfur compounds in three Sulfinert® sample cylinders over a 54-hour period. Dimethyl sulfide, which is not adsorbed by stainless steel, was used as an internal standard. The Sulfinert®-treated cylinders were inert to the reactive sulfur compounds over the 54-hour test period (Figure 1). Hydrogen sulfide exhibited greater than 85% recovery; methyl mercaptan, ethyl mercaptan, carbonyl sulfide, and dimethyl disulfide exhibited greater than 90% recovery.

Sulfinert®-treated gas sampling equipment is ideal for collecting and storing samples containing ppb levels of sulfur compounds, such as natural gas or beverage-grade carbon dioxide. Sulfinert® treatment ensures that sulfur compounds or other highly active compounds remain stable during transport from the field to the laboratory.

Stable Mercury Results

Siltek® surface treatment has been used in a wide variety of applications in which an inert surface is of paramount importance. To measure the impact of Siltek® treatment on adsorption of mercury during storage, we compared the performances of 304 grade stainless steel gas sampling cylinders (Swagelok®, Solon OH) with and without Siltek® treatment.

We filled each cylinder with $8\mu\text{g}/\text{m}^3$ of elemental mercury (approximately 1 part per billion) (Spectra Gases, Alpha NJ) and assessed the mercury concentration in each cylinder over time to determine changes in mercury concentration. Detection was achieved by direct interface gas sampling to an atomic absorption detector. The sample pathway regulator and tubing were Siltek® treated to ensure accurate transfer.

The data in Figure 2 demonstrate that Siltek® treatment provides a stable surface for elemental mercury, and untreated stainless steel does not. Based on these results, we conclude that Siltek® surface treatment for steel or stainless steel components and tubing in CMMS and sorbent tube mercury sampling systems will improve analytical reliability.

Siltek® and Sulfinert® surface treated cylinders and sampling components provide an inert sample path, which prevents adsorption of active compounds and ensures accurate sampling. For more information about these treatments, visit us at www.restekcoatings.com.

Acknowledgement

The authors wish to acknowledge Ted Neeme and Steve Mandel from Spectra Gases for their contributions to this work.

Figure 1 Stability of sulfur compounds is remarkable in Sulfinert®-treated cylinders.

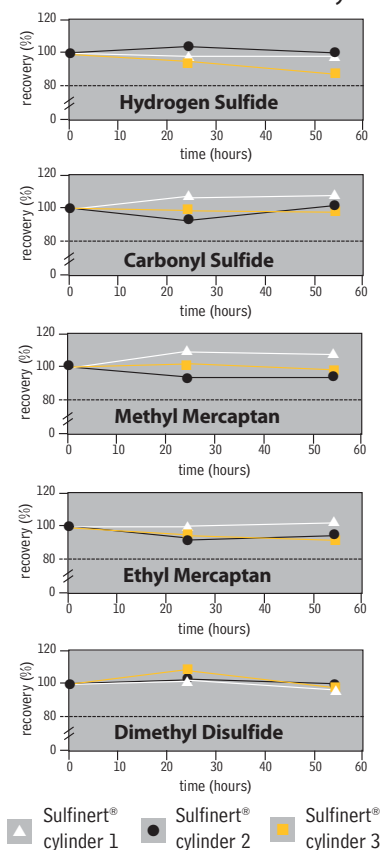
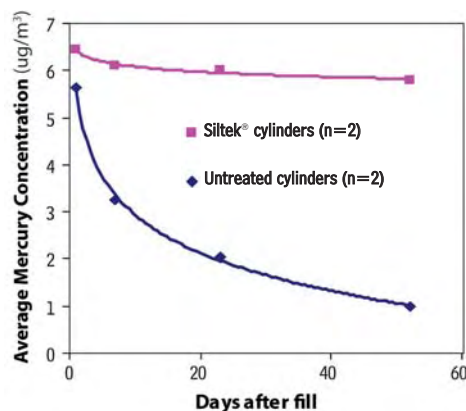


Figure 2 Siltek® treated gas sampling cylinders show very good inertness toward mercury.



Sulfinert® Treated Swagelok® Sample Cylinders

- Stable storage of samples containing ppb levels of sulfur compounds.
- Manufactured by Swagelok®; U.S. D.O.T. rated to 1,800psi (12,411kPa) at room temperature.
- 304 grade stainless steel with 1/4" female NPT threads on both ends.

Description	Size	qty.	cat.#
Sulfinert® Sample Cylinder	75cc	ea.	24130
Sulfinert® Sample Cylinder	150cc	ea.	24131
Sulfinert® Sample Cylinder	300cc	ea.	24132
Sulfinert® Sample Cylinder	500cc	ea.	24133
Sulfinert® Sample Cylinder	1000cc	ea.	24134
Sulfinert® Sample Cylinder	2250cc	ea.	21394

**Sulfinert® Treated Alta-Robbins Sample Cylinder Valves**

- All wetted parts are Sulfinert® treated for inertness.
- Compatible with Sulfinert® treated Swagelok® sample cylinders.
- Large, durable, Kel-F® seat ensures leak-free operation; temperature range: -40°C to 120°C.

Description	qty.	cat.#
1/4" NPT Exit	ea.	21400
1/4" Compression Exit	ea.	21401
1/4" NPT with Dip Tube*	ea.	21402
1/4" NPT with 2850psi Rupture Disc	ea.	21403
1/4" NPT Male Inlet x 1/4" Female Outlet with 2850psi Rupture Disc	ea.	21404

*To order catalog #21402 (Sulfinert Alta-Robbins Sample Cylinder Valve, 1/4" NPT with Dip Tube), please call Customer Service at 800-356-1688, ext. 3, or contact your Restek representative. Specify dip tube length or % outage when ordering (maximum length = 5.25" / 13.3cm). Note: End of part will not be treated after cutting tube to length.

**Siltek®/Sulfinert® Treated Coiled Electropolished 316L Grade Stainless Steel Tubing**

Recommended for:

- high temperatures
- demanding/corrosive environments
- ultimate inertness

OD	ID	cat.#	5-24 ft.	25-99 ft.	100-299 ft.	> 300 ft.
1/8" (3.18mm)*	0.085" (2.16mm)	22538				
1/4" (6.35mm)**	0.180" (4.57mm)	22539				

Siltek®/Sulfinert® Treated Coiled 316L Grade Stainless Steel Tubing

Recommended for:

- inert applications
- high temperatures
- high pressures
- corrosive environments

OD	ID	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	> 400 ft.
1/8" (3.18mm)**	0.055" (1.40mm)	22508				
1/4" (6.35mm)**	0.180" (4.57mm)	22509				
3/8" (9.52mm)***	0.277" (7.04mm)	22914				

Siltek®/Sulfinert® Treated Straight Seamless 316L Grade Stainless Steel Tubing

- Individual 6-foot pieces.

6 foot Length

OD	ID	qty.	cat.#
1/8" (3.18mm)**	0.055" (1.40mm)	ea.	22901
1/4" (6.35mm)**	0.180" (4.57mm)	ea.	22902
3/8" (9.52mm)***	0.277" (7.04mm)	ea.	22903

1/8" OD: 5 ft. to 100 ft. in one continuous coil; 1/4" OD: 5 ft. to 300 ft. in one continuous coil. Longer lengths will be more than one coil.
Note: required length in meters x 3.2808 = length in feet.

*0.020" wall thickness

**0.035" wall thickness

***0.049" wall thickness

ordering note

An extra charge is applied for cutting Siltek®/Sulfinert® or Silcosteel®-CR tubing. The charge is calculated from the total number of pieces produced for each line item

High Sensitivity Melamine GC/MS Analysis of Cat Food

Modified Conditions Save Costs and Reduce Maintenance

By Michelle Long, Innovations Chemist and Julie Kowalski, Ph.D., Food Flavor and Fragrance Innovations Chemist

- Excellent results in pet food matrix; lower pyridine background for better sensitivity.
- Easy sample preparation; reduced derivatization reagent volume lowers costs and keeps inlet and column clean.
- Modified conditions reduce maintenance and extend filament lifetime.

A large pet food recall occurred in 2007 when animals became ill or died after eating food contaminated with melamine and related compounds. Melamine is an industrial chemical used in the production of plastics, adhesives, flame retardants, fabrics and other materials. It is not a food ingredient, but since melamine and related compounds are high in nitrogen content—and protein testing methods are based on nitrogen levels—these compounds were used as additives to generate artificially high label values for protein content.

Procedure

The procedure for this experiment was adapted from the U.S Food and Drug Administration (FDA), GC/MS Method for Screening and Confirmation of Melamine and Related Analogs, Version 2, May 7, 2007. Standards were diluted to 10µg/mL and 1µg/mL with 10:40:50 diethylamine:water:acetonitrile. Three 0.5g matrix samples (dry cat food) were prepared: one control, one spiked at 50µg/g and one at 10µg/g.

Two modifications were made to the derivatization procedure in the FDA method. The amount of derivatizing reagent was reduced from 200µL to 50µL of BSTFA with 1% TMCS (which is still a molar excess of 50:1). Incubation time was subsequently increased from 45 min. to 120 min.

Analyses were performed on a Shimadzu QP-2010 Plus gas chromatograph mass spectrometer (GC/MS) using a 30m x 0.25mm ID x 0.25µm Rtx®-5MS column. The mass spectrometer data was acquired in SIM acquisition mode with selected ions for each analyte of interest (Table I).

Results

The original method conditions resulted in a significant initial baseline elevation due to the presence of pyridine, which is necessary for the derivatization reaction (Figure 2). Pyridine can increase ion signal background over a long period of time. To combat this, pyridine can be evaporated and the remaining analytes can be dissolved in a more GC amenable solvent, but this is time consuming and can result in analyte loss. A simpler solution is to eliminate the pyridine ion signal by changing the mass range to be scanned. All of the analytes have characteristic ions of interest well above m/z 79 which is associated with pyridine. Therefore,

Figure 1 Melamine and related compounds are nitrogen-rich and can artificially raise labeled protein content when used as an additive.

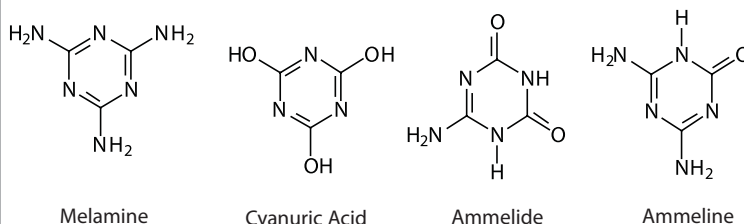
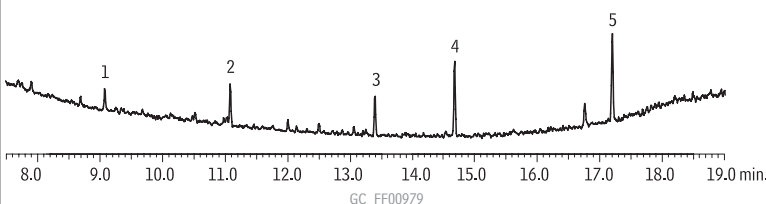


Figure 2 Original method produces an elevated baseline, compromising integration and reducing sensitivity (10µg/mL standard).



1. cyanuric acid
2. ammelide
3. ammeline
4. melamine
5. benzoguanamine

Column: Rtx®-5MS, 30m, 0.25mm ID, 0.25µm (cat.# 12623)
 Sample: melamine, cyanuric acid, ammelide, ammeline, benzoguanamine (10µg/mL prederivatized)
 Inj.: 1µL, splitless (hold 1 min.), 3.5mm splitless inlet liner (cat.# 22286)
 Inj. temp.: 280°C
 Carrier gas: helium, constant flow
 Flow rate: 1mL/min.
 Oven temp.: 75°C (hold 1 min.) to 320°C (hold 4 min.) @ 15°C/min.
 Det: MS
 Transfer line temp.: 290°C
 Scan range: 50-450 m/z
 Ionization: EI
 Mode: scan

Table I MS conditions (SIM mode).

Compound	Retention Time (min.)	Target Ions	Reference Ions	Reference Ions	Reference Ions
Cyanuric Acid	8.97	345 (100)*	330 (36)	346 (30)	347 (15)
Ammelide	9.79	344 (100)	329 (30)	345 (58)	330 (16)
Ammeline	10.44	328 (100)	343 (79)	329 (29)	344 (24)
Melamine	10.97	327 (100)	342 (53)	328 (30)	343 (17)
Benzoguanamine	13.18	316 (100)	331 (68)	332 (20)	330 (9)

Figure 3 Excellent separation of melamine and related compound using modified conditions (10µg/mL standard).

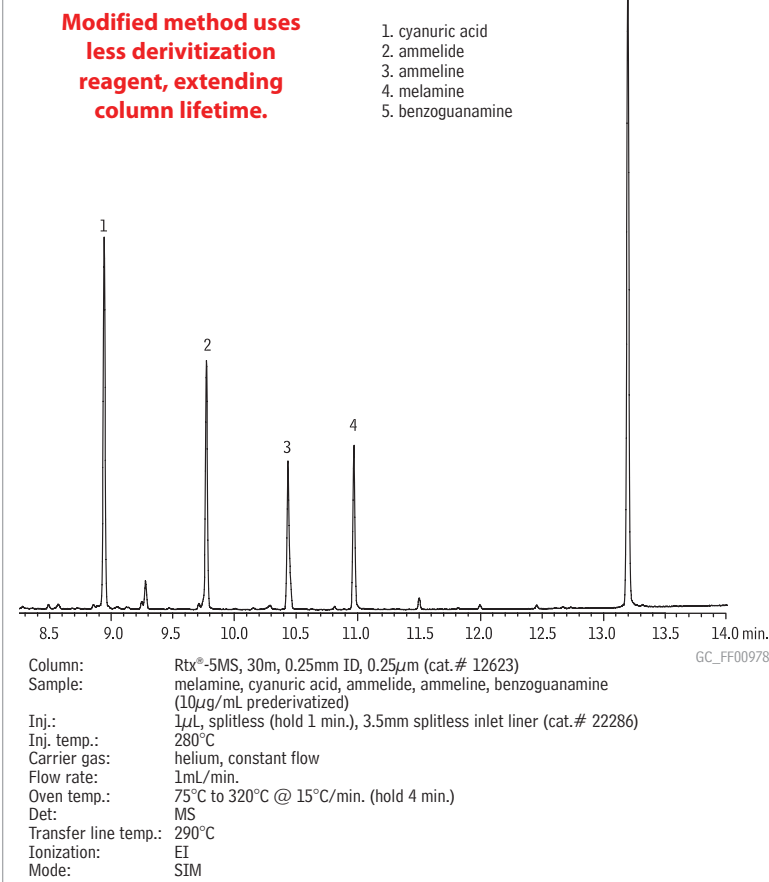
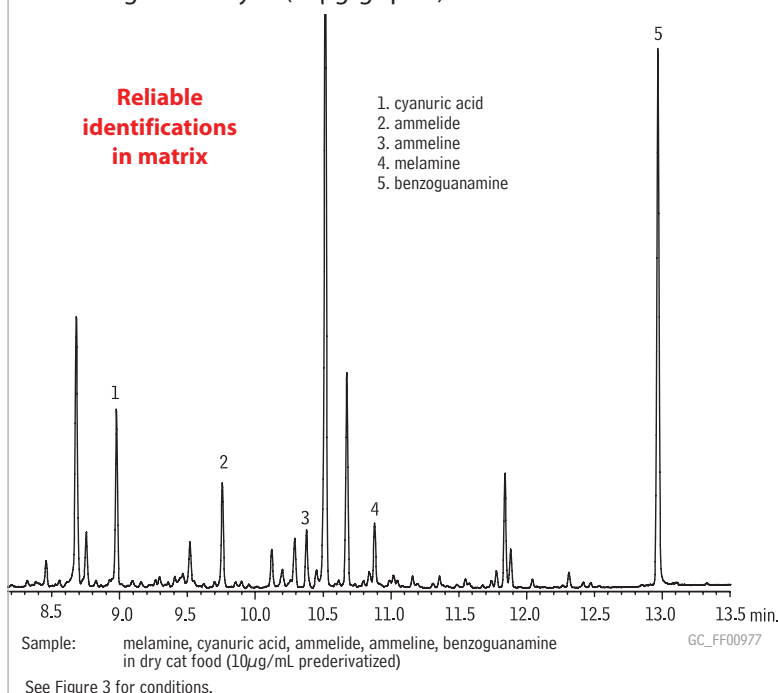


Figure 4 Melamine production analytes are easily identified in cat food using SIM analysis (50µg/g spike).



the scan method was modified to begin scanning at m/z 85. The solvent delay was also increased to approximately 8 min. due to the high background levels. This extra time helps increase the filament lifetime and ensures all the analytes will be detected.

This method provides excellent separation of melamine and cyanuric acid, the suspected toxic compounds, as well as ammelide and ammeline (Figure 3). Reproducible and reliable retention times were obtained for matrix spikes; this, along with SIM mass spectrometric detection, allows easy identification of analytes at both the high and low spike levels (Figure 4).

Conclusions

This work demonstrates that the FDA method is a valuable guideline for analysts screening melamine and related analogs. Using an Rtx®-5MS column and modifying the original method provides additional benefits: 1) decreasing the derivitization reagent volume results in longer column lifetime and less inlet maintenance, and 2) increasing the solvent delay decreases pyridine ion background, resulting in higher sensitivity, approximately 5 times higher, for the analytes of interest.

References

GC-MS Method for Screening and Confirmation of Melamine and Related Analogs, Version 2, May 7, 2007, U.S Food and Drug Administration, <http://www.fda.gov/cvm/GCMSscreen.htm>.

Rtx®-5MS—Low-bleed GC/MS Columns (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	12623

Splitless Liners for Shimadzu 17A, 2010, and 2014 GCs

ID* x OD & Length (mm)	qty.	cat.#
3.5mm Splitless		
3.5 ID x 5.0 OD x 95	ea.	22286
3.5 ID x 5.0 OD x 95	5-pk.	22287

*Nominal ID at syringe needle expulsion point.

Silylation Derivatization Reagents

Compound	CAS#	cat.#
BSTFA w/1% TMCS (N,O-bis[trimethylsilyl]trifluoroacetamide) w/1% trimethylchlorosilane)		
10-pk. (10x1g)	25561-30-2	35606
25g Flex Tube	25561-30-2	35607



Fast, Simple Sample Cleanup

Using QuEChERS SPE Tubes

By Julie Kowalski, Innovations Chemist, Lydia Nolan, Innovations Chemist,
Jack Cochran, Director of New Business and Technology, and Irene DeGraff, Product Marketing Manager



- Achieve a four-fold increase in sample throughput.
- Significantly reduce material costs.
- Convenient, ready to use centrifuge tubes with ultra pure, pre-weighed adsorbent mixtures.

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method for extracting pesticides from food is based on research by the US Department of Agriculture.¹ In addition to using less solvent and materials versus conventional SPE methods, QuEChERS employs a novel and much quicker dispersive solid phase extraction cleanup (dSPE). QuEChERS methods, including an AOAC Official Method² and modifications to the methods, have been posted on the Internet.³ These methods have several basic steps in common:

Step 1: Sample preparation and extraction—Commodities are uniformly comminuted. Acetonitrile solvent is added for a shake extraction. Salts, acids and buffers may be added to enhance extraction efficiency and protect sensitive analytes. Surrogate standards can be added to monitor extraction efficiencies.

Step 2: Extract cleanup – A subsample of solvent extract is cleaned up using dSPE, a key improvement incorporated in the QuEChERS technique. Small polypropylene centrifuge tubes are prefilled with precise weights of MgSO₄ and SPE adsorbents to remove excess water and unwanted contaminants from the extracted samples. After agitation and centrifugation, the cleaned extracts are ready for analysis.

Step 3: Sample analysis – Samples may be pH adjusted to protect sensitive pesticides and/or solvent-exchanged to improve analysis by either GC/MS or LC/MS. Internal standards can be added.

QuEChERS methods are convenient, rugged methods that simplify extract cleanup, reduce material costs, and improve sample throughput. Here we demonstrate the effectiveness of QuEChERS sample cleanup using a multiresidue analysis of pesticides on strawberries.

Experimental

Strawberry extracts were prepared, spiked, and dSPE treated according to Table I. Analytical conditions are presented in Table II.

One microliter splitless injections of the extracts were performed by a Shimadzu AOC-20i autosampler using “mid” injection speed into a Shimadzu QP-2010 Plus GC-MS system operated under the conditions in Table II.

Table I Modified mini-multiresidue QuEChERS for pesticides from strawberries.

Sample preparation and extraction

Sample:	10g of strawberries were homogenized and placed in a 50mL PTFE centrifuge tube
Solvent:	10mL of acetonitrile were added to homogenate Shake for 1 minute, until uniform
Salts:	4.0g MgSO ₄ (powder or granular) 1.1.0g NaCl 1.0g trisodium citrate dihydrate 0.5g disodium hydrogen citrate sesquihydrate Salts were added and vigorously shaken for 1 minute. Sample was centrifuged and the supernatant removed for cleanup. Pesticides standards (200ng/mL) were spiked in at this point.

Sample extract cleanup

QuEChERS tubes:	1mL of supernatant from the previous step was placed into several 2mL polypropylene centrifuge tubes, each containing one of the following adsorbent mixes: A. 50mg PSA + 150mg MgSO ₄ (cat.# 26124) B. 50mg PSA + 150mg MgSO ₄ + 50mg C18 (cat.# 26125) C. 50mg PSA + 150mg MgSO ₄ + 50mg GCB (cat.# 26123)
Cleanup:	Samples were shaken with the adsorbents for 30 seconds (carbon for 2 minutes), then centrifuged to produce a clear supernatant for GC/MS analysis.

Internal standard: Pentachloronitrobenzene in a formic acid solution, pH 5.

PSA—primary and secondary amine exchange material.

GCB—graphitized carbon black

Table II Instrument conditions.

Column:	Rtx®-CLPesticides2 20m, 0.18mm ID, 0.14µm (cat.# 42302)
Sample:	custom pesticide mix 200µg/mL each pesticide, internal standards: 8140-8141 ISTD, 1000µg/mL (cat.# 32279), 508.1 ISTD 100µg/mL (cat.# 32091), triphenylphosphate 1000µg/mL (cat.# 32281)
Inj.:	1.0µL splitless (hold 1 min.)
Inj. temp.:	250°C
Carrier gas:	helium
Flow rate:	constant linear velocity @ 40cm/sec
Oven temp.:	40°C (hold 1 min.) to 320°C @ 12°C/min.
Det:	Shimadzu GCMS-QP2010 Plus
Transfer line temp.:	300°C
Ionization:	Electron ionization
Mode:	Selected ion monitoring



Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.14	-60 to 310/330°C	20-Meter	42302

Results and Discussion

Primary and secondary amine exchange material (PSA) is the base sorbent used for dSPE cleanup of QuEChERS fruit and vegetable extracts because it removes many organic acids and sugars that might act as instrumental interferences.

A pesticide-spiked strawberry extract (200ng/mL) subjected to dSPE with PSA was used to generate one-point calibration curves. Spiked strawberry extracts subjected to additional dSPE sorbents were analyzed and the results versus PSA dSPE are shown as percent recoveries in Table III. C18 is suggested for use when samples might contain fats; not an issue for a strawberry extract, but it was important to verify that gross losses of more hydrophobic pesticides (e.g. Endrin and DDT) would not occur. GCB is used to remove pigments, and when treated, the pink/red strawberry extract became clear. However, GCB can also have a negative effect on certain pesticides, especially those that can assume a planar shape like chlorothalonil and thiabendazole.

Restek dSPE products in a variety of standard sizes and formats make QuEChERS even simpler. The centrifuge tube format, available in 2mL and 15mL sizes, contains magnesium sulfate (to partition water from organic solvent) and a choice of SPE sorbents, including PSA (to remove sugars and fatty acids), C18 (to remove nonpolar interferences such as fats), and GCB (to remove pigments and sterols). Custom products also are available by request. If you are frustrated by the time and cost involved with your current approach to pesticide sample cleanup, we suggest you try this simple and economical new method.

References

1. Michelangelo Anastassiades, Steven J. Lehotay, Darinka Štajnbaher, Frank J. Schenck. "Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and Dispersive Solid-Phase Extraction for the Determination of Pesticide Residues in Produce." *J. AOAC International*, 2003, vol. 86(22), pp.412-431.
2. AOAC Official Method 2007.01, "Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate."
3. <http://www.quechers.com/>

References not available from Restek

Table III Pesticide percent recoveries in strawberry extracts treated with C18 or GCB dSPE, relative to PSA only.

Rt (min.)	pesticide	CAS Number	action/Use	classification	C18*	GCB**
9.50	Dichlorvos	62-73-7	Insecticide	Organophosphorus	111	116
9.67	Methamidophos	10265-92-6	Insecticide	Organophosphorus	105	107
11.75	Mevinphos	7786-34-7	Insecticide	Organophosphorus	112	130
12.02	o-Phenylphenol	90-43-7	Fungicide	Unclassified	106	97
12.14	Acephate	30560-19-1	Insecticide	Organophosphorus	128	147
13.89	Omethoate	1113-02-6	Insecticide	Organophosphorus	120	119
14.74	Diazinon	333-41-5	Insecticide	Organophosphorus	108	127
14.98	Dimethoate	60-51-5	Insecticide	Organophosphorus	124	151
15.69	Chlorothalonil	1897-45-6	Fungicide	Organochlorine	125	13
15.86	Vinclozolin	50471-44-8	Fungicide	Organochlorine	102	98
16.21	Metalaxyl	57837-19-1	Fungicide	Organonitrogen	105	117
16.28	Carbaryl	63-25-2	Insecticide	Carbamate	114	111
16.60	Malathion	121-75-5	Insecticide	Organophosphorus	124	160
16.67	Dichlofluanid	1085-98-9	Fungicide	Organohalogen	122	103
17.51	Thiabendazole	148-79-8	Fungicide	Organonitrogen	88	14
17.70	Captan	133-06-2	Fungicide	Organochlorine	88	91
17.76	Folpet	133-07-3	Fungicide	Organochlorine	108	63
18.23	Imazalil	35554-44-0	Fungicide	Organonitrogen	115	95
18.39	Endrin	72-20-8	Insecticide	Organochlorine	104	101
18.62	Myclobutanil	88671-89-0	Fungicide	Organonitrogen	119	114
19.07	4,4-DDT	50-29-3	Insecticide	Organochlorine	102	95
19.22	Fenhexamid	126833-17-8	Fungicide	Organochlorine	118	77
19.40	Propargite 1	2312-35-8	Acaricide	Organosulfur	110	95
19.43	Propargite 2	2312-35-8	Acaricide	Organosulfur	121	114
19.75	Bifenthrin	82657-04-3	Insecticide	Pyrethroid	106	81
20.04	Dicofol	115-32-2	Acaricide	Organochlorine	98	54
20.05	Iprodione	36734-19-7	Fungicide	Organonitrogen	118	90
20.21	Fenpropathrin	39515-41-8	Insecticide	Pyrethroid	113	96
21.32	cis-Permethrin	52645-53-1	Insecticide	Pyrethroid	106	65
21.47	trans-Permethrin	51877-74-8	Insecticide	Pyrethroid	109	71
23.74	Deltamethrin	52918-63-5	Insecticide	Pyrethroid	97	52

*50mg PSA, 50mg C18, **50mg PSA, 50mg GCB

$$\% \text{ recovery} = \frac{\text{RRF C18 or GCB}}{\text{RRF PSA}} \times 100$$

QuEChERS SPE Tubes

AOAC Method 2007.1	Benefits/Uses	qty.	cat#
2mL QuEChERS SPE Micro-Centrifuge Tube Contains 150mg Magnesium Sulfate and 50mg PSA	Cleanup of agricultural produce extracts, 1mL sample volume.	100-pk.	26124
2mL QuEChERS SPE Micro-Centrifuge Tube Contains 150mg Magnesium Sulfate, 50mg PSA, and 50mg Graphitized Carbon	Cleanup of 1mL sample extract with residual pigments and sterols.	100-pk.	26123
2mL QuEChERS SPE Micro-Centrifuge Tube Contains 150mg Magnesium Sulfate, 50mg PSA, and 50mg C18	Cleanup of 1mL sample extract with residual fat.	100-pk.	26125
15mL QuEChERS SPE Centrifuge Tube Contains 900mg Magnesium Sulfate, 300mg PSA, and 150mg Graphitized Carbon	Cleanup of 6mL sample extract with residual pigments and sterols.	50-pk.	26126

PSA—primary and secondary amine exchange material.



FREE Sample Packs Available!

To receive your free sample pack, add -248 to the item number. (One sample per customer)

Multi-task with an Ultra IBD Column

A Versatile Column with Many Applications

By Rick Lake, Pharmaceutical Innovations Chemist

- Effective in normal or reversed mode; compatible with 100% aqueous mobile phases.
- Excellent base deactivation—superior peak shape for basic compounds.
- Enhanced retention of hydrophilic compounds.

Reversed phase HPLC analyses are predominantly performed on C18 columns, which, in many cases, are suitable. There are, however, situations in which a conventional C18 column produces less than optimal chromatography. For example, C18 columns have little retention for hydrophilic compounds, basic compounds often exhibit peak tailing, and highly aqueous conditions can cause inconsistent retention or even phase collapse.

One way in which column manufacturers attempt to address these issues, and yet maintain the favorable hydrophobic interaction of a C18 column, is to impart polar functionality into an alkyl phase. The Ultra IBD column is an example of such a polar embedded column. Compared to a C18 column, this column offers enhanced retention and selectivity towards a wider range of compounds, orthogonal separations, improved base-deactivation, and compatibility with entirely aqueous mobile phases.

Degree of Polarity

The Ultra IBD column exhibits a high degree of polarity relative to conventional and aqueous C18 phases. Because the Ultra IBD column possesses both nonpolar and highly polar characteristics, it can be used in both normal phase mode and reversed phase mode. The bonding chemistry used in the Ultra IBD column makes it a very adaptable column capable of unique separations.

Base-Deactivation

The Ultra IBD column bonding chemistry alleviates one of the common problems associated with alkyl phases—peak tailing of basic analytes. Comparing the analysis of amitriptyline on a conventional C18 column and an Ultra IBD column demonstrates the effectiveness of this bonding chemistry. Amitriptyline is a highly basic, tricyclic antidepressant that commonly tails on silica-based alkyl phases. Even at a neutral pH and, importantly, with no modifiers, the Ultra IBD column exhibits excellent peak shape for amitriptyline (Figure 1). This is advantageous because it provides needed flexibility for method development, especially for analytes that are labile under acidic conditions. In applications where Gaussian peak shape is needed for accurate integrations, such as potency assays, or when tighter system suitability criteria are required, an intrinsically base-deactivated stationary phase offers a benefit that a conventional C18 column cannot—exceptional peak shape with a simplified mobile phase.

Retention and Selectivity

In contrast to conventional C18 columns, the Ultra IBD has a polar functional group embedded within the alkyl chain. Retention, therefore, is attributed not only to hydrophobic interactions (the major retention mechanism of an alkyl (or C18) phase, but also to polar attraction between the analyte and stationary phase. This mixed-mode mechanism results in high retention for hydrophilic compounds or compounds with polar moieties, such as purines (Figure 2).

Orthogonal separations also can be achieved through the Ultra IBD phase chemistry. For example, a small group of hydroxybenzoic acids was also assayed on a C18 and IBD column under identical conditions. The elution order of the analytes differed and dihydroxybenzoic acid was more retained on the Ultra IBD column (Figure 3). Additionally, the unique phase chemistry of the Ultra IBD column makes it suitable for a simultaneous analysis of a wide range of compounds—acidic through basic, as well as zwitterions (Figure 4).

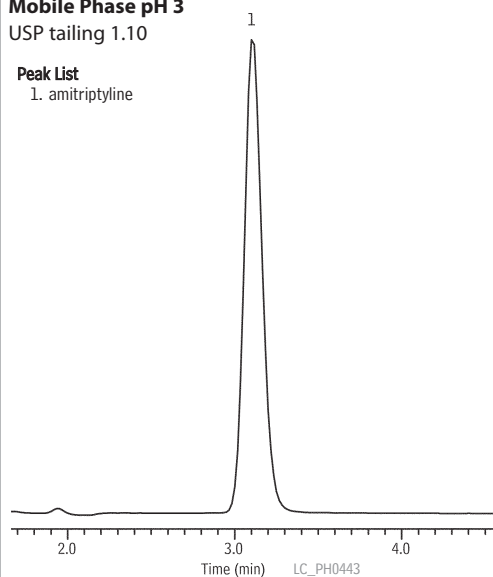
Figure 1 Ultra IBD offers more flexibility in method development giving excellent peak shape for highly basic compounds—even without mobile phase modifiers—over a wide mobile phase pH range.

Mobile Phase pH 3

USP tailing 1.10

Peak List

1. amitriptyline



Mobile Phase pH 7

USP tailing 1.13

Sample:

Inj.: 10 µL
Conc.: ~100 µg/mL
Sample diluent: mobile phase

Column:

Ultra IBD
Cat.#: 9175565
Dimensions: 150mm x 4.6mm
Particle size: 5 µm
Pore size: 100 Å

Conditions:

Mobile phase: 10:90 20mM
potassium phosphate
(pH 3):methanol
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

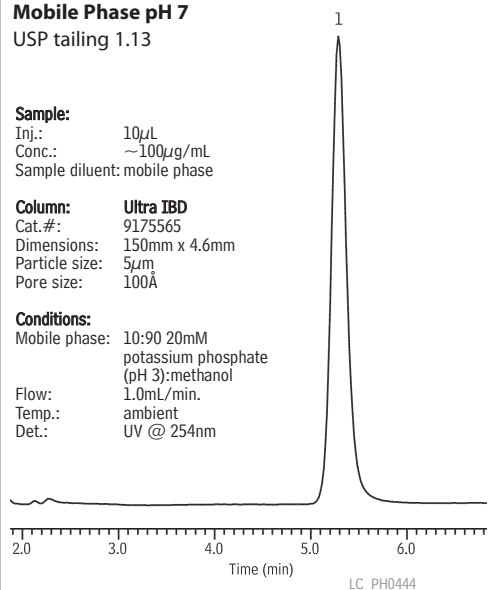


Figure 2 The Ultra IBD column exhibits high retention for hydrophilic compounds or compounds with polar moieties, and is compatible with up to 100% aqueous mobile phases ruggedness in aqueous mobile phases.

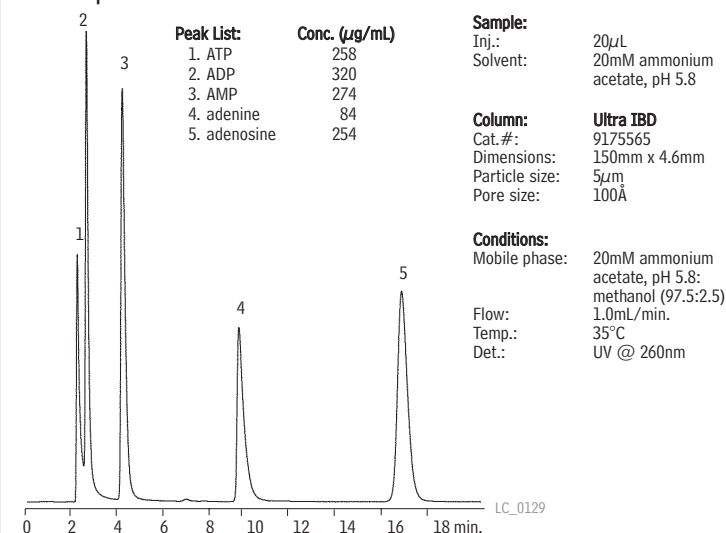


Figure 3 The Ultra IBD column gives needed flexibility for polar compounds; it increases retention, enhances resolution, and creates alternate selectivity.

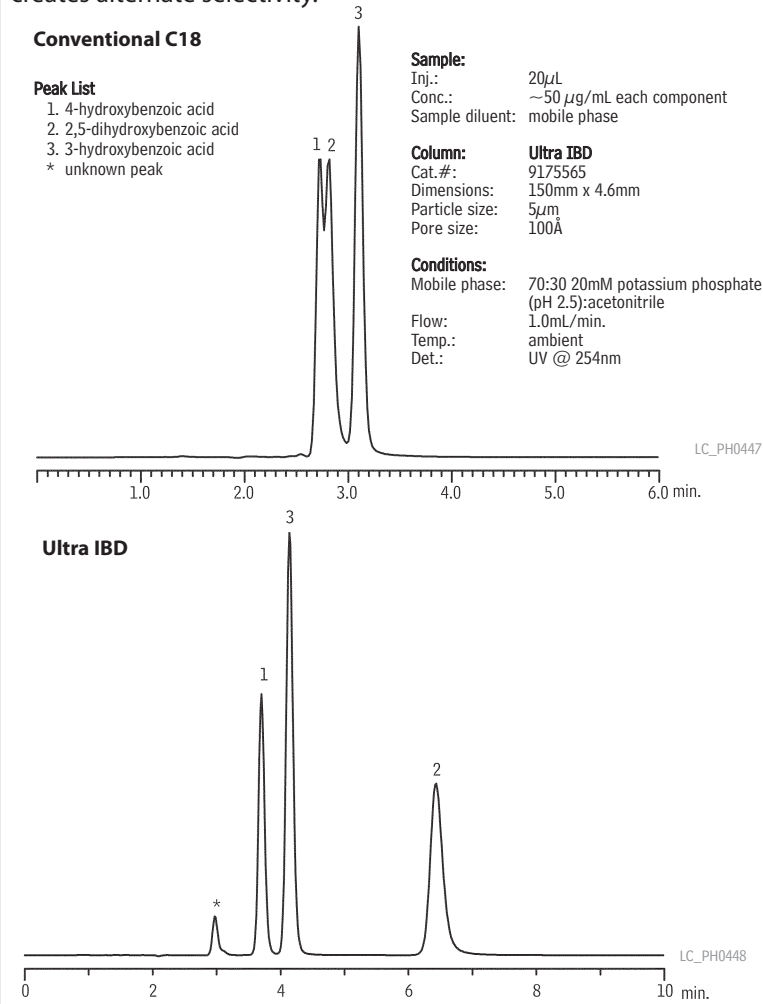
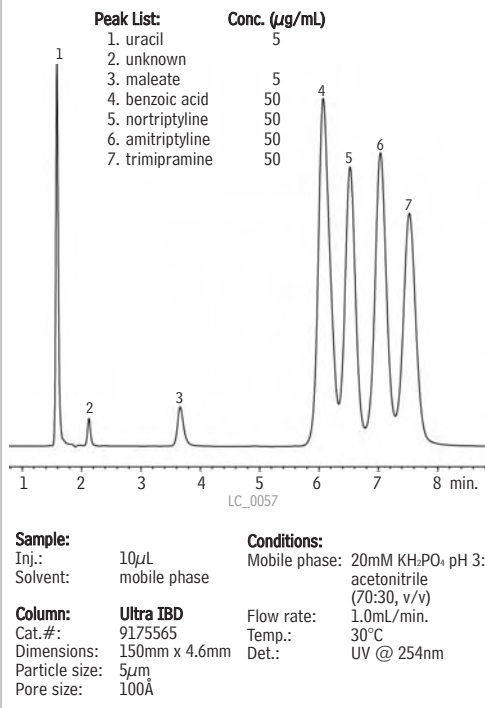


Figure 4 The versatility of the Ultra IBD makes it well-suited for analyzing a wide range of compounds.



Conclusion

The Ultra IBD, through unique bonding chemistry, is an extremely versatile HPLC column. It offers alternate selectivity, and a high degree of both polar and nonpolar retention, making it a powerful tool for analyzing a wide range of compounds. The Ultra IBD also addresses the inherent problems attributed to linear alkyl phases, providing excellent peak shape for basic compounds and heightened retention of hydrophilic compounds. The versatility of the Ultra IBD makes it an excellent tool for the practicing method developer.

Ultra IBD Columns Specialized Columns for Mixed Polar and Nonpolar Compounds

Physical Characteristics:

particle size: 3µm or 5µm, spherical	endcap: no
pore size: 100Å	pH range: 2.5 to 7.5
carbon load: 12%	temperature limit: 80°C

Ultra IBD, 5µm Columns

5µm Column, 4.6mm	cat. #
150mm	9175565
150mm (with Trident Inlet Fitting)	9175565-700
Ultra IBD Guard Cartridges	qty. cat. #
10 x 2.1mm	3-pk. 917550212
10 x 4.0mm	3-pk. 917550210
20 x 2.1mm	2-pk. 917550222
20 x 4.0mm	2-pk. 917550220

Fast, Sensitive Analysis of Benzodiazepines by LC/MS/MS

Quantify an Order of Magnitude below Typical Methods

By Kristi Sellers, Clinical/Forensic Innovations Chemist

- Achieve full chromatographic separation of compounds with shared precursor ions
- Quantify compounds at 10ng/mL or less in urine.
- Increase accuracy with improved desolution efficiency from highly organic mobile phase.

Benzodiazepines are widely prescribed drugs used for treating anxiety and sleep disorders. Since addiction and abuse can occur, efficient screening methods are critical to clinical, forensic, and toxicology laboratories. The liquid chromatography tandem mass spectrometry (LC/MS/MS) method presented here offers several advantages over other techniques: minimal sample preparation, fast analysis times, multiple reaction monitoring transitions for quantification and confirmation, and sensitivity down to 0.10-10ng/mL. This method uses the Allure® PFP Propyl stationary phase, which retains compounds long enough to minimize matrix interferences and chromatographically separate compounds that share the same precursor ion.

Procedure

Samples were prepared by adding 100µL of internal standard solution (1µg/mL D5-Diazepam and D3-Dioxepine) to 100µL urine, diluting with 800µL LC grade water, and centrifuging. The samples were then analyzed by LC/MS/MS. Compound separation was achieved using an Allure® PFP Propyl column and a mobile phase gradient program.

A 3200 QTrap® LC/MS/MS system equipped with a Turbo V™ source with electrospray ionization was used to develop and detect the two MRM transitions (Table 1). For each compound, MRM 1 was used to quantify, and the ratio to MRM 2 was used to confirm.

Cliquid™ Drug Screen & Quant Software was used to process data and generate automatic reporting relevant to forensic guidelines. Limits of quantification were determined and the automated reporting allowed for positive confirmation based on the detected MRM ratios.

Results

By diluting the urine samples ten-fold, matrix effects are reduced (reducing ion suppression) and LOQs between 0.10ng/mL and 10ng/mL can be achieved (Table 1). Ion suppression is further reduced by using a retentive column which 1) elutes matrix interferences before the compounds of interest, and 2) allows for better desolvation efficiency due to the ability to use 90% organic in the mobile phase composition. The Allure® PFP Propyl is such a column; it has high retention and selectivity for basic drug compounds, such as ben-

Figure 1 MRM transitions of 27 benzodiazepines, 3 nonbenzodiazepine hypnotics, and two internal standards on the Allure® PFP Propyl column.

Sample: benzodiazepines		
Inj.:	20µL	
Conc.:	NA	
Solvent:	NA	
Column: Allure® PFP Propyl		
Cat.#:	9169552	
Dimensions:	50mm x 2.1mm	
Particle size:	5µm	
Pore size:	60Å	
Conditions:		
Mobile phase: A: 0.1% formic acid and 1mM ammonium formate in water		
B: 0.1% formic acid and 1mM ammonium formate in acetonitrile		
Time (min.)	Flow (µL/min.)	%B
0.0	500	10
10.00	1000	90
15.00	1000	90
15.50	500	10
17.50	500	10
Flow: see gradient table		
Temp.: 40°C		
Det.: Applied Biosystems/MDS Sciex		
API 3200™ MS/MS system		
Ion Source: Electrospray, positive		
IonSpray Voltage: NA		
Gas 1: NA		
Gas 2: NA		
Source Temperature: 500°C		

Data courtesy of: Applied Biosystems MDS Sciex

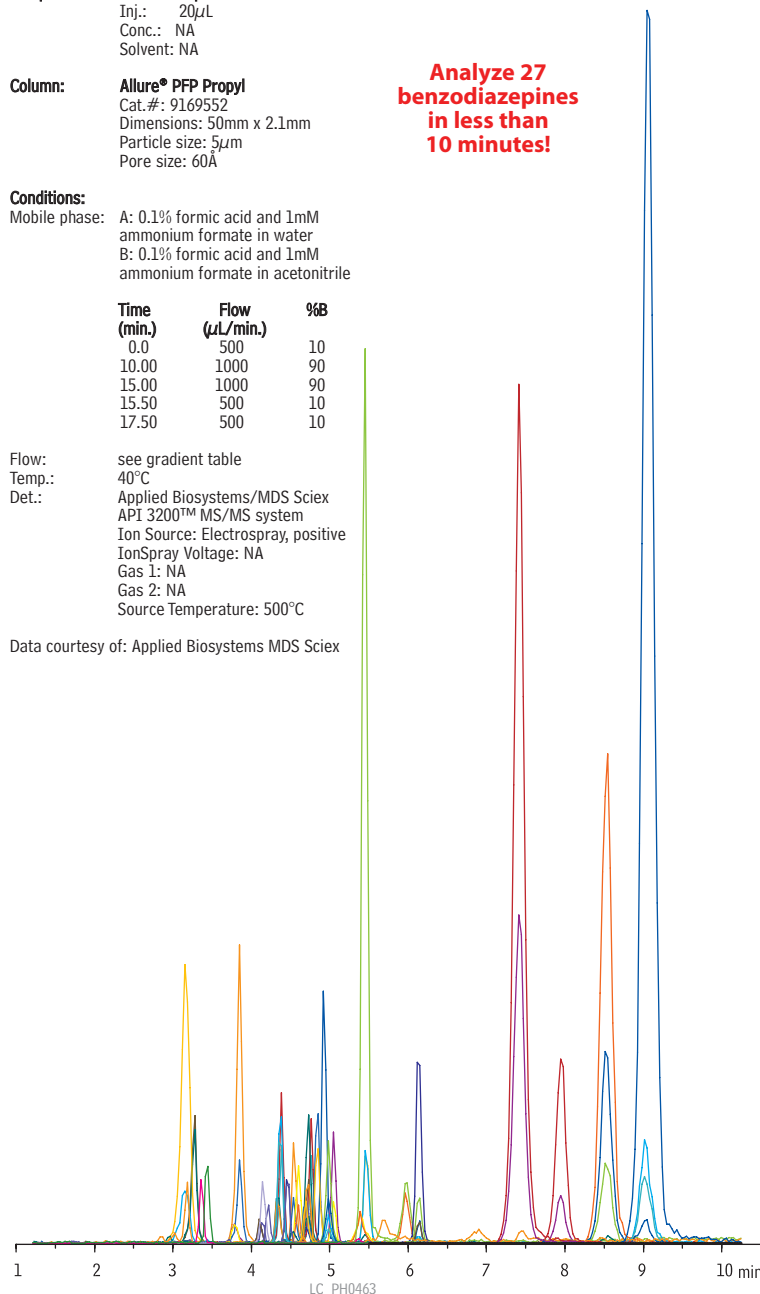


Table I MRM Transitions, retention times, and LOQ values.

Compound Name	Retention Time (min.)	Precursor Ion (amu)	MRM 1 (amu)	MRM 2 (amu)	DP	CE (MRM 1)	CE (MRM 2)	LOQ (ng/mL)
7-aminonitrazepam	3.2	252.1	121.1	94.0	51	35	53	1.0
7-aminoclonazepam	3.3	286.1	121.0	222.2	46	41	35	0.5
7-aminoflunitrazepam	3.8	284.1	135.1	226.0	51	39	49	0.5
Bromazepam	3.8	316.0/318.0	182.1	182.1	51	45	45	5.0
α -hydroxyalprazolam	4.1	325.1	297.2	204.9	51	31	59	2.0
α -hydroxytriazolam	4.1	359.0	239.2	176.0	61	63	37	5.0
Oxazepam	4.2	287.0	241.1	268.9	41	27	19	10.0
Lorazepam	4.3	321.0/323.1	275.0	277.0	41	31	27	5.0
Estazolam	4.4	295.0	205.0	267.1	51	53	31	2.0
Zaleplon	4.4	306.2	236.3	264.2	56	35	27	0.5
2-hydroxyethylflurazepam	4.5	333.1	211.2	109.0	56	51	41	1.0
Desmethyflunitrazepam	4.5	300.1	254.2	198.2	56	35	51	2.0
Nitrazepam	4.6	282.0	236.1	180.2	71	35	51	2.0
Clonazepam	4.7	316.0	270.2	214.0	56	41	51	2.0
Desalkylflurazepam	4.7	289.1	140.1	226.1	71	41	39	2.0
Temazepam	4.7	301.1/303.1	255.1	257.2	35	30	30	5.0
Triazolam	4.7	343.0	238.9	314.9	61	53	37	1.0
Alprazolam	4.8	309.1	205.1	281.1	56	53	35	1.0
Lormetazepam	4.8	335.0/337.1	289.0	291.1	41	29	29	2.0
Clobazam	4.9	301.1	259.1	224.3	46	29	47	1.0
Flunitrazepam	5.0	314.0	268.1	239.1	56	35	49	1.0
Nordiazepam	5.0	271.1	140.2	164.9	46	37	35	2.0
Zolpiclone	5.4	389.1	244.8	217.0	16	25	41	1.0
D5-Diazepam	5.4	290.1	198.2	-	55	41	-	-
Diazepam	5.5	285.0	193.2	154.1	55	41	37	1.0
Chlordiazepoxide	6.0	300.1	227.1	283.2	36	31	21	5.0
Prazepam	6.1	325.1	271.1	140.0	81	31	53	2.0
Zolpidem	7.4	308.1	235.1	236.1	56	39	35	0.2
Midazolam	7.9	326.1	291.3	222.0	56	33	63	0.5
Flurazepam	8.5	388.2	315.1	317.1	36	27	27	0.1
Medazepam	9.0	271.0	91.1	207.3	46	41	39	2.0
D3-Doxepine	9.1	283.0	107.1	-	41	35	-	-

Bar color indicates shared precursor ions. Note compounds with shared precursor ions are baseline resolved on the Allure® PFP Propyl column, as shown by retention time comparison. Data courtesy of Applied Biosystems MDS Sciex.

The Allure® PFP Propyl stationary phase provides baseline resolution for compounds sharing the same precursor ion, such as nordiazepam and medazepam. The ability to chromatographically separate compounds with similar spectra allows this method to be adapted for single stage MS, however, the LOQ values would be affected. Tandem MS is advantageous since two MRM transitions are collected, allowing quantification and confirmation to be accomplished in a single run, without loss of sensitivity.

Conclusion

The method presented here provides significant advantages over other techniques for benzodiazepine analysis: simple sample preparation, fast analysis time (less than 10 minutes), LOQs of 0.10-10ng/mL in matrix, and quantification and confirmation in a single run. Further, using the retentive Allure® PFP Propyl column eliminates coelution of matrix peaks with target compounds and assures full chromatographic resolution of analytes with shared precursor ions.

Acknowledgement

We sincerely thank Andre Schreiber of Applied Biosystems and Houssain El Aribi and John Gibbons of MDS Sciex for supplying the method and data.

References

Schreiber, Andre PhD, El Arbi, Houssain and Gibbons, John. 2007. A Fast and Sensitive LC/MS/MS Method for the Quantitation and Confirmation of 30 Benzodiazepines and Nonbenzodiazepine Hypnotics in Forensic Urine Samples. Applied Biosystems MDS Sciex.

Trident Direct Guard Cartridge System

Description	qty.	cat.#
High-pressure filter	ea.	25082
10mm guard cartridge holder without filter	ea.	25083
10mm guard cartridge holder with filter	ea.	25084
20mm guard cartridge holder without filter	ea.	25085
20mm guard cartridge holder with filter	ea.	25086

*The standard PEEK™ tip in Trident Direct systems is compatible with Parker®, Upchurch Scientific®, Valco™, and other CPI-style fittings. To use Trident Direct systems with Waters-style end fittings, replace the tip with cat.# 25088.

Allure® PFP Propyl Columns (USP L43) Excellent Columns for LC/MS and ELSD

Physical Characteristics:

particle size: 3µm or 5µm, spherical	endcap: fully endcapped
pore size: 60Å	pH range: 2.5 to 7.5
carbon load: 17%	temperature limit: 80°C

5µm Column, 2.1mm	cat. #
50mm	9169552
50mm (with Trident Inlet Fitting)	9169552-700

Allure® PFP Propyl Guard Cartridges	qty.	cat. #
10 x 2.1mm	3-pk.	916950212
10 x 4.0mm	3-pk.	916950210
20 x 2.1mm	2-pk.	916950222
20 x 4.0mm	2-pk.	916950220

Exempted Drug of Abuse Reference

Materials: Benzodiazepines

Concentration is µg/mL. Volume is 1mL/ampul.

Compound	CAS#	Solvent Code	Conc.	cat.#
alprazolam	28981-97-7	PTM	1,000	34042
bromazepam	1812-30-2	PTM	1,000	34043
chlordiazepoxide	438-41-5	PTM	1,000	34044
clobazam	22316-47-8	PTM	1,000	34045
clonazepam	1622-61-3	PTM	1,000	34046
diazepam	439-14-5	PTM	1,000	34047
flunitrazepam	1622-62-4	PTM	1,000	34049
flurazepam	1172-18-5	PTM	1,000	34050
lorazepam	846-49-1	PTM	1,000	34051
nitrazepam	146-22-5	PTM	1,000	34053
oxazepam	604-75-1	PTM	1,000	34054
prazepam	2955-38-6	PTM	1,000	34055
temazepam	896-50-4	PTM	1,000	34056
triazolam	28911-01-5	PTM	1,000	34057

PTM=purge & trap grade methanol

Accurate, Reproducible Amphetamines Analysis

Clean Up Procedure Improves Chromatography and Reduces Maintenance

By Kristi Sellers, Clinical/Forensic Innovations Chemist, and Amanda Rigdon, Innovations Chemist

- Derivatization improves peak symmetry, for more accurate results.
- Clean up procedure reduces system contamination, and extends column lifetime.
- Rtx®-5MS column produces a stable baseline for derivatized compounds, ideal for GC/MS analysis.

Introduction

Analyzing amphetamines by GC/MS is challenging whether the compounds are derivatized or underivatized. Underivatized amphetamines appear as irregular and asymmetric peaks, which are difficult to integrate, and may lead to irreproducible results. Derivatized amphetamines result in symmetric peaks, but derivatizing reagents can contaminate the inlet/column. This contamination can shorten column lifetime and cause noisy, elevated baselines that interfere with the analysis of target compounds.

In this study, we evaluated the effects of several sample pretreatment methods. These methods included: 1) no pretreatment, 2) converting the salt forms into free bases, 3) derivatizing the free bases with heptafluorobutyric acid anhydride (HFAA), and 4) derivatizing the free bases with HFAA followed by a clean up. Our objectives were to obtain symmetric shapes, reduce baseline noise, and maintain low column bleed from injection to injection for GC/MS analysis.

Procedure

The first method had no pretreatment. The untreated standard was prepared in methanol and diluted to a final concentration of 100µg/mL. It was then injected without any further preparation. The second pretreatment involved converting the drug standard to the free base form. The free base forms were prepared by mixing the standard (100µg/mL) with water, then adding saturated sodium borate water, and extracting the amphetamines with butylchloride. The resulting sample was then analyzed by GC.

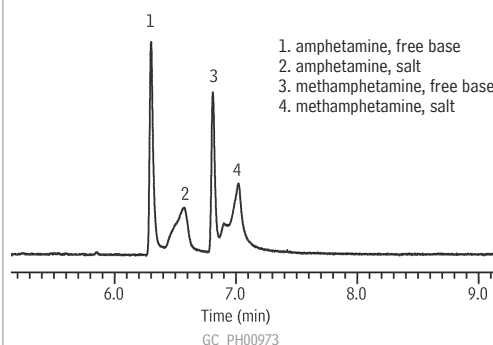
The third pretreatment procedure included both conversion and derivatization. The HFAA derivatized amphetamines were prepared by converting the compounds to free bases (as described above), reacting with derivatizing reagent HFAA, and diluting the sample before injection. The fourth pretreatment procedure consisted of free base conversion, HFAA derivatization, and a clean up step to remove the acidic byproducts of derivatization. The clean up procedure included mixing the sample with a phosphate buffer (pH=7.0) before dilution, removing the butylchloride layer, and then diluting the sample just before injection. An Rtx®-5MS column (30m x 0.25mm ID x 0.25µm) was used for analysis; instrument conditions are presented in Figure 1. Repetitive GC/MS runs (over 190 injections) were evaluated to confirm symmetry, baseline, and bleed results.

Results

Analyzing untreated amphetamine and methamphetamine results in peak doublets caused by the presence of both the salt (hydrochloride) and free base forms (Figure 1). Peak doublets were eliminated by conversion to free base form, however, some tailing was still observed (Figure 2). This pretreatment improves reproducibility, but is still not optimal as tailing can cause irreproducible integration and significant variation in peak area counts.

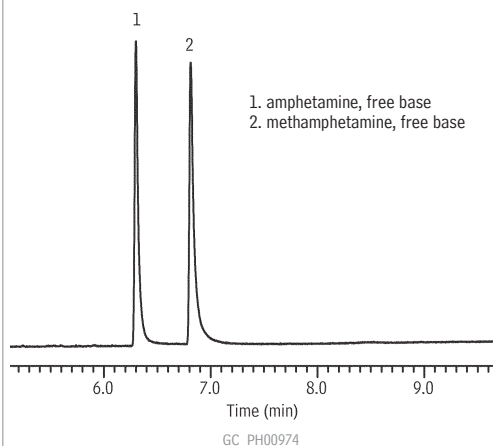
The most symmetric peak shapes were obtained by derivatizing the amphetamines with HFAA (Figure 3). Although peak shape was improved, the acidic derivatization byproducts generated a noisy baseline and shortened column life. This system contamination increases injector and column maintenance.

Figure 1 Untreated standard contains both salt and free base forms causing inaccurate, irreproducible results.



Column: Rtx®-5MS, 30m, 0.25mm ID, 0.25µm (cat.# 12623)
Sample: 100µg/mL amphetamine and methamphetamine in methanol
Inj.: 1µL, split (split ratio 10:1), 4mm single gooseneck w/ wool inlet liner (cat.# 20798-211.1)
Inj. temp.: 250°C
Carrier gas: hydrogen, constant flow
Flow rate: 1mL/min.
Oven temp.: 70°C to 250°C @ 15°C/min. (hold 5 min.)
Det: FID @ 300°C

Figure 2 Conversion to free base form improves chromatography, but produces tailing factors over 2.0.



See Figure 1 for conditions.

Figure 3 Derivatizing with HFAA yields symmetric peaks but results in system contamination and a noisy baseline.

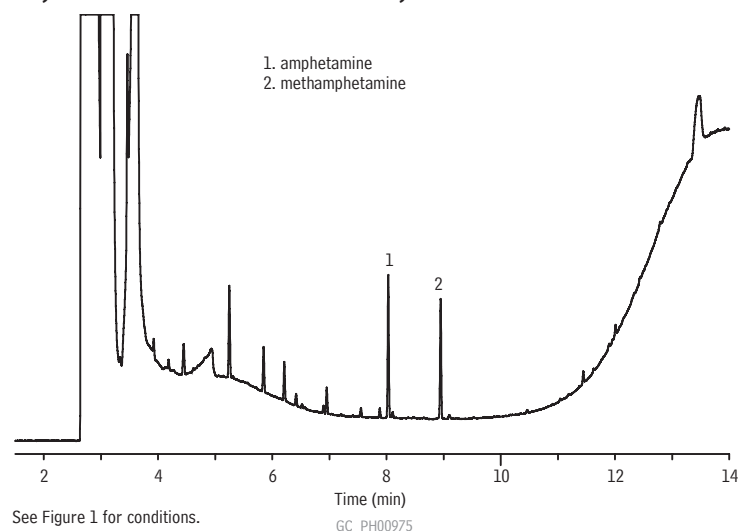


Figure 4 A post-derivatization clean up procedure results in symmetric peaks and a clean baseline.

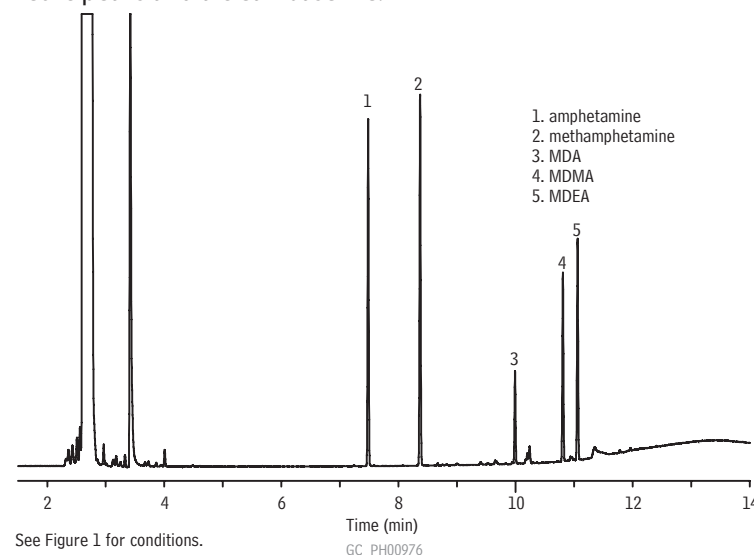


Table I Tailing factor comparison of pretreatments.

Pretreatment	TF Amp	TF Meth	TF MDA	TF MDMA	TF MDEA
Sodium Borate Wash (GC/FID)	2.115	2.837	NA	NA	NA
HFAA Only (GC/FID)	1.010	0.989	NA	NA	NA
HFAA w/Post clean Up (GC/FID)	0.981	0.996	1.007	0.997	0.992

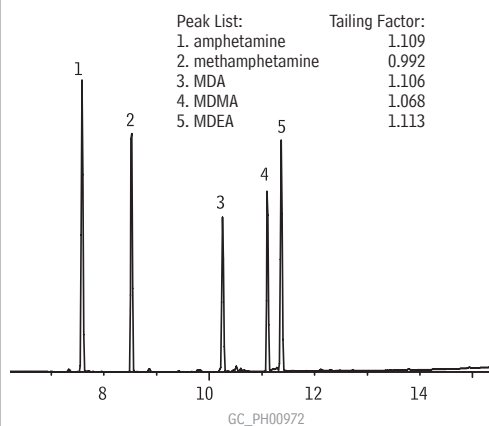
NOTE: A perfectly symmetric peak exhibits a tailing factor of 1.0. Tailing factors shown were generated using the USP tailing factor calculation.

Rtx®-5MS—Low-bleed GC/MS Column (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (μm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	12623

Figure 5 Post-derivatization clean up also produces symmetric peaks and a stable baseline when analyzed by GC/MS.



Column: Rtx®-5MS, 30m, 0.25mm ID, 0.25μm (cat.# 12623)
Sample: 100μg/mL each amphetamine, methamphetamine, MDA, MDMA, and MDEA extracted from methanol and HFAA derivatized
Inj.: 1μL, splitless (hold 0.5 min.), 3.5mm custom splitless inlet liner w/IP deactivated wool; Inj. temp.: 220°C; Carrier gas: helium, constant flow; Flow rate: 1.25mL/min.; Oven temp.: 70°C (hold 1 min.) to 290°C @ 15°C/min. (hold 4 min.); Det: MS; Transfer line temp.: 280°C; Scan range: 43-450amu; Ionization: EI; Mode: scan.

Incorporating a post conversion/derivatization clean-up procedure removed derivatization contaminants while maintaining chromatographic quality (Table I), thus reducing the need for frequent system maintenance and extending column lifetime. These benefits were also seen when samples were analyzed by GC/MS (Figure 5).

Conclusion

The conversion/derivatization/clean-up procedure presented here produces symmetric peaks while reducing the amount of contamination that can enter the GC system. This method ensures accurate area count reproducibility, a clean GC system, and a stable baseline, even for GC/MS work.

Acylation Derivatization Reagents

Compound	CAS#	cat.#
HFAA (heptafluorobutyric acid anhydride)		
10-pk. (10x1g)	336-59-4	35622
25g Flex Tube	336-59-4	35623

Exempted Drug of Abuse Reference

Materials: Amphetamines & Metabolites

Concentration is μg/mL. Volume is 1mL/ampul.

Compound	CAS#	Solvent	Code	Conc.	cat.#
d-amphetamine	51-63-8	PTM	1,000	34020	
3,4-MDA HCl	4764-17-4	M	1,000	34070	
3,4-MDEA HCl	82801-81-8	M	1,000	34072	
3,4-MDMA HCl	42542-10-9	M	1,000	34071	

M=methanol

PTM=purge & trap grade methanol

The Forgotten Septum

How to Correctly Diagnose the Source of Bleed Contamination

By Amanda Rigdon, Innovations Chemist



- Avoid lengthy inlet troubleshooting.
- Reduce interference with correct solvent-septum compatibility.

Septum bleed is not common, but when it occurs it is observed as sharp, repetitive peaks in high temperature portions of an analysis. Bleed peaks can come from either the injection port septum or the vial cap septum. Interfering peaks and inaccurate data can result, so it is important to correctly identify the source and understand how to eliminate or minimize the bleed.

Diagnose the Bleed Source

The bleed from either septum shows a similar pattern (Figure 1), but it is easy to determine the source with a simple test. Isolate the injection port by setting the instrument to perform a run without an injection. Perform an analysis; if the bleed disappears, then the vial cap septum was the source. Determining if the vial cap septum is the source of the bleed can save time by preventing unnecessary troubleshooting and maintenance of the injection port. If the vial cap septum is causing bleed, the problem can be eliminated or minimized with the following considerations.

Check Solvent-Septum Compatibility

Most of the time, septum bleed is negligible. However, when a solvent and vial cap septum are incompatible, extreme contamination can occur. Figure 2 compares the first and fifth injections from a vial containing a derivatized amphetamine sample. In this case, the septum bleed peaks are almost as large as the analyte peaks. This level of bleed can interfere with analyses, especially those geared for trace levels. Reduce the risk of septum bleed by using a compatibility chart, such as the one in the on-line version of this article (www.restek.com/general) to determine which septum material is compatible with the sample solvent used.

Use Lined Septa

Most vial cap septa are lined with a protective layer of polytetrafluoroethylene (PTFE) to prevent solvent attack. As shown in Figure 3, PTFE effectively prevents septum breakdown due to solvent exposure. In comparison, unlined septa exhibit bleed after just 24 hours at room temperature. Bleed levels for unlined septa varied by material, but even a low level of bleed can interfere with integration and is of particular concern for trace analyses (Figure 4).

Figure 1 Sharp, repetitive peaks are typical of septum bleed from the vial cap or injection port.

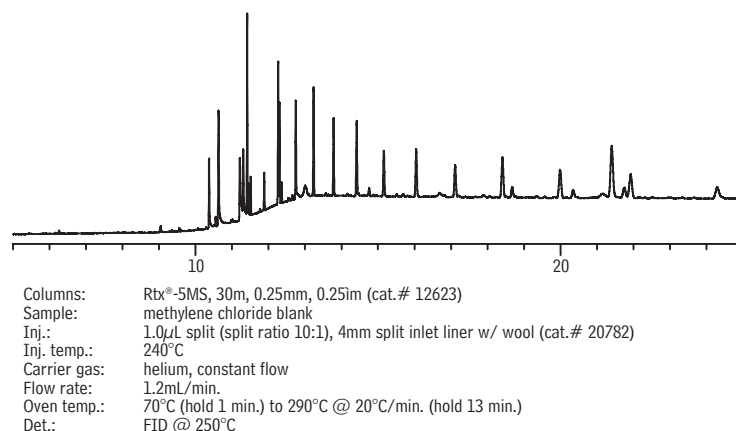


Figure 2 Contamination from septum bleed can cause significant interference with target analytes.

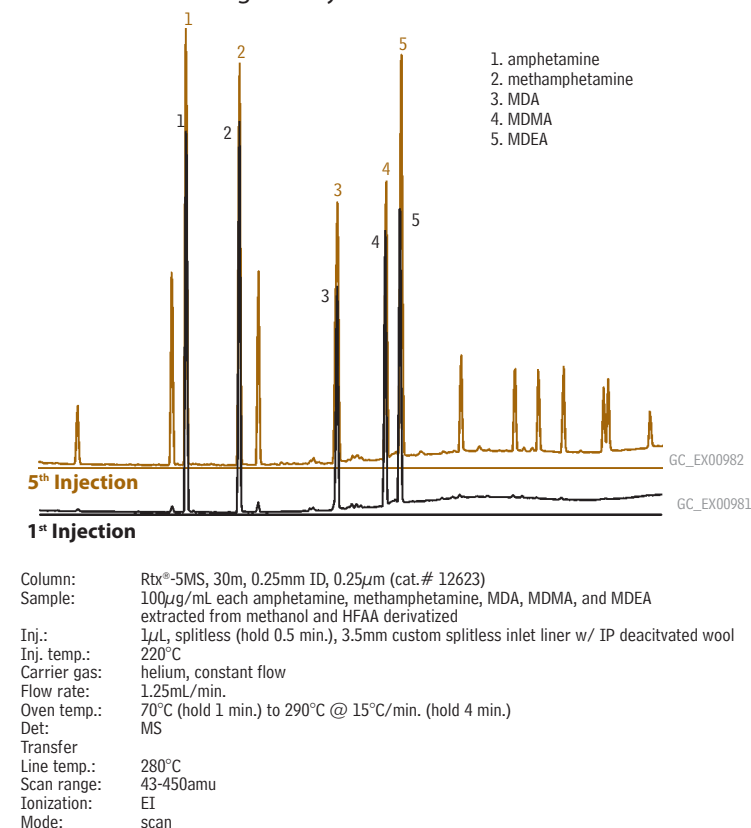


Figure 3 PTFE lining prevents bleed due to solvent/septum interaction.

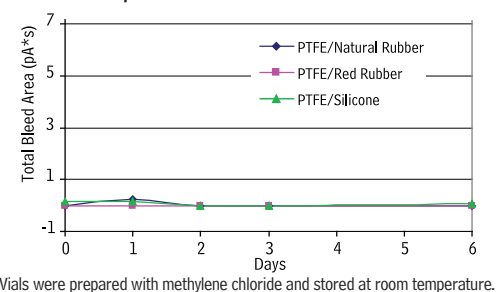


Figure 4 Unlined septa show bleed contamination within 24 hours.

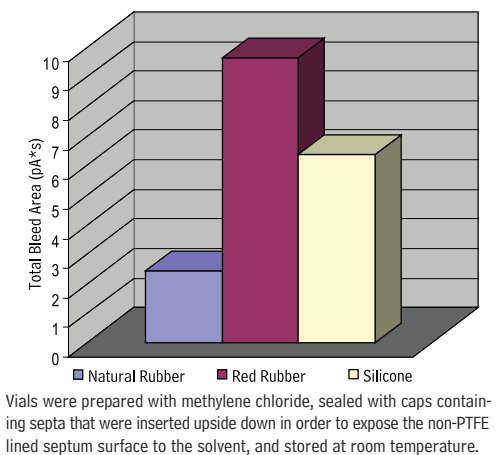


Figure 5 Bleed contamination increases over multiple injections.

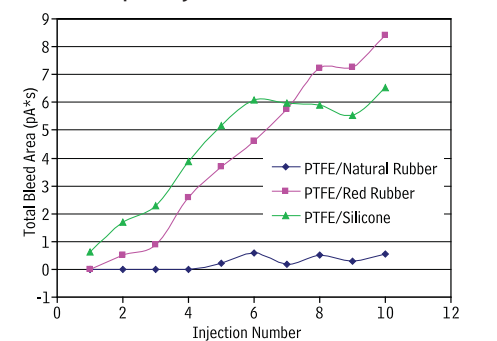
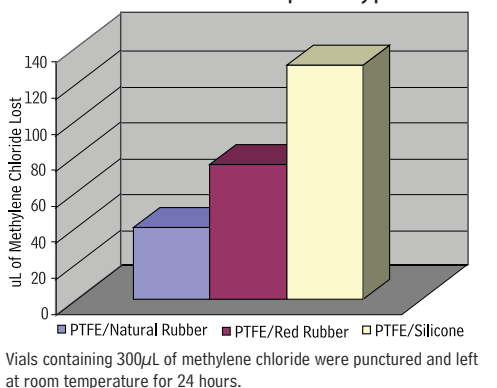


Figure 6 This bar graph shows the volume of solvent lost for three septum types.



Consider Resealability

Multiple injections can core the vial cap septum and lead to significant bleed. Resistance to coring varies by septum material (Figure 5). Coring can be minimized by preparing separate vials for replicate injections, when feasible, and by carefully considering the type of septum material when multiple injections are necessary. Septum resealability also affects evaporative loss, which can be a significant source of error for low volume samples. For example, a relatively nonvolatile analyte in a volatile solvent can concentrate significantly due to evaporative loss (Figure 6). Vials should be recapped when necessary for extended runs or long term storage.

Conclusion

Septum bleed is not a very common occurrence, but when bleed does occur, it is easy to assume the injection port septum is the source because the vial cap septum often is not considered. However, correctly diagnosing the source of bleed contamination can save time and effort by preventing unnecessary injection port maintenance. Effectively and efficiently reducing interfering peaks by controlling septum bleed can significantly improve analytical performance, particularly for trace analyses.

Crimp-Top Vials, Snap Seal™ Style—12 x 32mm, 11mm Crimp

Description	100-pk.	1000-pk.
2.0mL Clear Glass Vial w/White Graduated Marking Spot	24383	24384
2.0mL Amber Glass Vial w/White Graduated Marking Spot	24385	24386
2.0mL Clear Glass Vial without Graduated Marking Spot	21152	21153

2.0mL, 11 mm Aluminum Crimp Seals with Septa

Description	100-pk.	1000-pk.
Silver Seal, PTFE/Natural Rubber Septa	21174	21175
Red Seal, PTFE/Red Rubber Septa	24355	24356
Silver Seal, PTFE/Silicone Septa	24359	24360

Limited Volume Inserts for 2.0mL Crimp-Top & Short-Cap, Screw-Thread Vials

Description	100-pk.	1000-pk.
350μL Glass, Flat Bottom Insert w/ ID Ring	24692	24693

Rtx®-5MS—Low-bleed GC/MS Columns (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (μm)	temp. limits	length	cat. #
0.25mm	0.25	-60 to 330/350°C	30-Meter	12623

Split Liners for Agilent GCs

ID* x OD & Length (mm)	qty.	cat.#
4mm Split w/ Wool		
4.0 ID x 6.3 OD x 78.5	ea.	20781
4.0 ID x 6.3 OD x 78.5	5-pk.	20782
4.0 ID x 6.3 OD x 78.5	25-pk.	20783

*Nominal ID at syringe needle expulsion point.



Selecting the Right HPLC Guard Column

By Terry Reid, Technical Service

HPLC guard columns (cartridges) are installed in front of an analytical column in order to protect it from strongly retained impurities. Understanding the significant factors that affect guard column performance can help you protect your analytical column and save money by extending column lifetime.

Packing & Dimensions

It is best to use a guard column that contains the same packing material as the analytical column. In other words, the best guard column for a Pinnacle™ II C18 analytical column is a Pinnacle™ II C18 guard column. Trident guard cartridges come in two lengths, 10mm or 20mm. The 10mm length is adequate for most applications, but a 20mm guard should be considered for samples that contain a lot of impurities, such as crude extracts. Regarding internal diameter (ID), the general rule is that the guard column ID should be the same as, or one size smaller than, the ID of the analytical column to prevent a loss of efficiency.

Cartridge Holder Options

Trident guard cartridges can be used with three different styles of Trident guard holder: integral, in-line, and direct. Note that guard cartridges from one manufacturer should never be used in another manufacturer's holder.

The Trident Integral guard system is a cost-effective, low dead volume option that is recommended when purchasing a guard and analytical column at the same time. This system includes an analytical column, a guard cartridge, and extra frit, all of which are integrated into a single unit. The advantages of the Trident Integral Guard column system are that it has the lowest dead volume of any of the holders and is also the most cost effective option.

Restek also offers in-line and direct holders. Trident In-Line holders are traditional stand-alone style holders that require an additional piece of HPLC capillary tubing to connect the guard holder to the analytical column. The Trident Direct Holder differs in that it contains a threaded PEEK™ tip. The threaded tip allows the holder to screw directly into the analytical column's inlet end fitting, eliminating the need for any additional tubing. The Trident In-Line and Trident Direct holders both can be connected to any HPLC column, even those from other manufacturers. Both these holders are available in either a "with filter" or "without filter" version. The "with filter" versions have an XF (extra filter) fitting that contains a cap frit that can be changed independently of the guard cartridge.

Filters

The ID of the cap frit should match the ID of the guard cartridge; however, frit porosity is largely a matter of preference. The smaller porosity will provide the greatest protection against particles, but also may mean that the frit needs to be changed more frequently. Choosing a filter porosity that matches the porosity of the analytical column protects against particles lodging in the column inlet frit.

Restek offers a wide selection of HPLC guard options. An understanding of the significance of different guard column parameters, including dimensions, holder styles, and extra filters, can simplify the selection process. Choosing the proper guard column will maximize the lifetime of your analytical column by effectively protecting it from sample contaminants.

Trident In-Line 10mm guard cartridge holder with filter

Components



Assembled

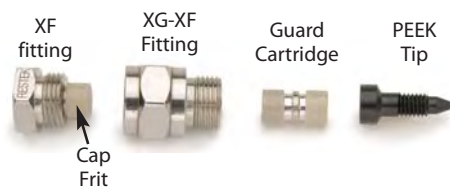


Installed onto column



Trident Direct 10mm guard cartridge holder with filter

Components



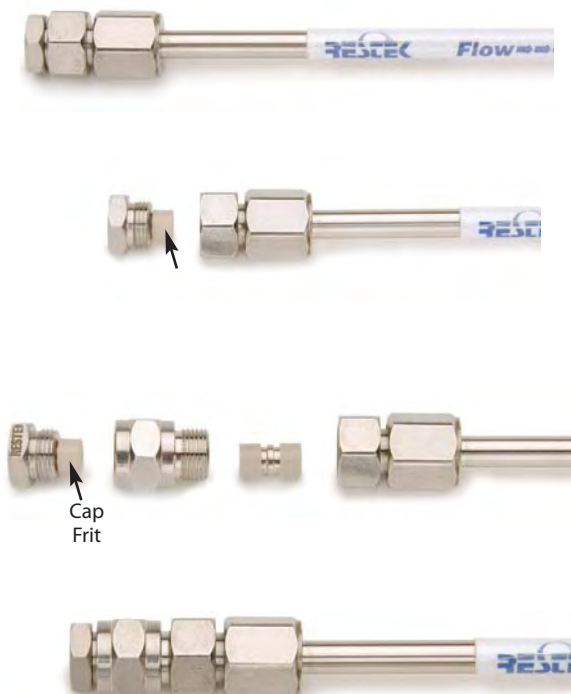
Assembled



Installed onto column



Trident Integral Guard System



Looking for HPLC guard column options?

Visit us at www.restek.com, or call Technical Support at 800-356-1688, to discuss your applications.

NEW! Waste Overflow Indicator for HPLC Systems

By Becky Wittrig, Ph.D, HPLC Product Marketing Manager

- Avoid messy pooling around mobile phase waste containers.
- Audible alarm instantly alerts user, preventing overflow.
- Compact, battery operated unit.

The new Restek Waste Overflow Indicator will help to keep your mobile phase waste where it belongs—in the waste container! Compact, battery operated unit accommodates two lines and fits securely on 4-liter solvent bottles. An audible alarm is given as the solvent waste container approaches capacity, giving you time to empty or change the container. Another innovative design from Restek!

Waste Overflow Indicator for HPLC Systems

Description	qty.	cat.#
Waste Overflow Indicator for HPLC Systems	kit	26543
Replacement AA Battery for the Waste Overflow Indicator	ea.	26544
Replacement AA Batteries for the Waste Overflow Indicator	3-pk.	26545



Peak Performers

Replacement Parts for Agilent FID Detectors

By Donna Lidgett, GC Accessories Product Marketing Manager and Sue Benes, GC Accessories Product Marketing Manager



FID Replacement Jets

Standard Version

- Engineered with a fluted tip to guide the capillary column into the jet.
- Threads specially coated for easy installation and removal.
- Special processing ensures the highest degree of cleanliness.

High-Performance Version

- Similar to the standard version, but Siltek® treated.
- Extremely inert, for use with active compounds.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs

	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
0.011-Inch ID Tip					
Standard, 0.011-Inch ID Tip	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek® Treated, 0.011-Inch ID Tip	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850/7890 GCs

	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
0.011-Inch ID Tip					
Standard, 0.011-Inch ID Tip	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek® Treated, 0.011-Inch ID Tip	G1531-80560	ea.	21620	3-pk.	21683

Packed Column FID Replacement Jets for Agilent 5890/6890/6850 GCs

	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
0.018-Inch ID Tip*					
Standard, 0.018-Inch ID Tip	18710-20119	ea.	21694	3-pk.	21695
High-Performance Siltek® Treated, 0.018-Inch ID Tip	18710-20119	ea.	21696	3-pk.	21697

	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
0.030-Inch ID Tip*					
Standard, 0.030-Inch ID Tip	18789-80070	ea.	21688	3-pk.	21689
High-Performance Siltek® Treated, 0.030-Inch ID Tip	18789-80070	ea.	21686	3-pk.	21687

* 0.018-inch ID jets: Used for most general-purpose packed column applications.

** 0.030-inch ID jets: For packings that exhibit high bleed and that frequently clog the tip of smaller 0.018-inch jets.

tech tip

Which FID Jet Should I Use?

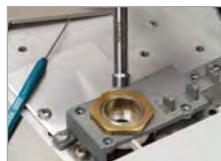
There are two FID jet configurations for Agilent GCs. The longer "adaptable" jet fits both 5890 and 6890 GCs, and can be used with capillary or packed columns. The shorter "dedicated" jet is for the FID in the 6890 GC that is designed only for use with capillary columns.

restek
innovation!



FID Jet Removal Tool for Agilent 5890/6890/6850/7890 FIDs

- Securely grips jet in socket for easy removal or installation.
- Unique, ergonomic handle—easy to hold.



Slip tool
over FID
jet...



...loosen
jet...



...and
remove.

Description	qty.	cat.#
FID Jet Removal Tool for Agilent 5890/6890/6850/7890 FIDs	ea.	22328



Rugged design!

FID Gauge Pack for Agilent 5890 GCs

Pressure regulators and gauges for air & hydrogen. The 1/8-inch bulkhead allows easy hookup to instrument. Rated for inlet pressures to 250psi (1724kPa), outlet pressures of 0 to 60psi (0-414kPa).

Description	qty.	cat.#
FID Gauge Pack for Agilent 5890 GCs	ea.	22071

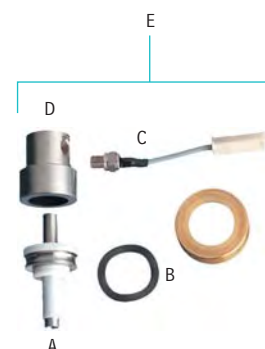
Direct Replacement FID Collector Assembly Kit for Agilent 5890 GCs

Description	Similar to Agilent part #	qty.	cat.#
E) FID Collector Assembly Kit (includes insulators)	19231-60690	kit	23010
E) FID Collector Assembly Kit w/Siltek® Ignitor Castle	—	kit	21131

Replacement FID Parts for Agilent 5890 GCs

Description	Similar to Agilent part #	qty.	cat.#
A) FID Collector (includes insulators)	19231-20970 19231-20960 19231-20950	ea.	21138
B) FID Collector Nut and Washer	19231-20940 5181-3311	set	21136
C) FID Ignitor*	19231-60680	ea.	21001
D) FID Ignitor Castle	19231-20910	ea.	21137
Siltek® FID Ignitor Castle	—	ea.	21135

*Also fits OI Analytical 4410 detector (similar to OI part # 191833).



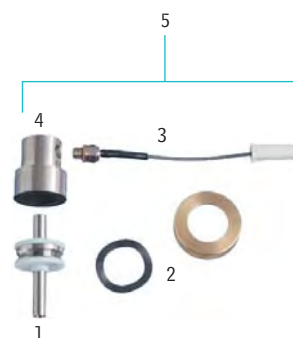
Direct Replacement FID Collector Assembly Kit for Agilent 6890/6850/7890 GCs

Description	Similar to Agilent part #	qty.	cat.#
5) FID Collector Assembly Kit (includes insulator)	G1531-60690	kit	21699
5) FID Collector Assembly Kit w/Siltek® Ignitor Castle	—	kit	21132

Replacement FID Parts for Agilent 6890/6850/7890 GCs

Description	Similar to Agilent part #	qty.	cat.#
1) FID Collector (includes insulators)	G1531-20690 G1531-20700	ea.	21139
2) FID Collector Nut and Washer	19231-20940 5181-3311	set	21136
3) FID Ignitor*	19231-60680	ea.	21001
4) FID Ignitor Castle	19231-20910	ea.	21137
Siltek® FID Ignitor Castle	—	ea.	21135

*Also fits OI Analytical 4410 detector (similar to OI part # 191833).



FID Base Weldment for Agilent GCs

- Meets or exceeds manufacturer's performance.
- Includes brass nut.

Description	Similar to Agilent part #	qty.	cat.#
A) FID Base Weldment for Agilent 5890 GCs	19231-80580	ea.	23041
B) FID Base Weldment, Pack Column FID, for Agilent 6850/6890 GCs	G1531-80580	ea.	23052
C) FID Base Weldment, Capillary Column FID, for Agilent 6850/6890 GCs	G1531-80630	ea.	23053

Note: 6890 GC connections to EPC modules are not compatible with the 7890 EPC modules.



Spanner Wrench for Agilent 5890/6890/6850/7890 FID Collector Assemblies

- Easily remove the nut from the FID collector without damaging the nut.
- Unique, ergonomic handle—easy to grip.



Remove FID ignitor castle.



Easily loosen the nut by aligning the two pins on the bottom of the wrench with the two open slots on the nut...



...then turn counter-clockwise...



...and remove.



Description	Similar to Agilent part #	qty.	cat.#
Spanner Wrench for Agilent 5890/6890/6850/7890 FID Collector Assembly	19231-00130	ea.	22329

NEW! Electron Multipliers for Mass Spectrometry

By Sue Benes, GC Accessories Product Marketing Manager

- The multi-dynode approach of all ETP electron multipliers results in longer lifetimes and better sensitivity compared with channel electron multipliers or continuous dynode multipliers.
- Optimized ion and electronic optics and unique dynode shapes for maximum performance.
- Increased surface area for enhanced sensitivity and extended operational life.



Cat# 23074

Features of ETP Electron Multipliers

- Proprietary specialized surface material resulting in very high secondary electron emission.
- Air stable.
- 2-year shelf life guarantee.
- Discrete dynode design results in extended operating life.

The electron multipliers manufactured by ETP use a proprietary dynode material. This material has a number of properties that make it very suitable for use in an electron multiplier. It has very high secondary electron emission, which allows exceptional gain to be achieved from each dynode. This material is also very stable in air. In fact, an ETP multiplier can be stored for years before being used. As a direct result of the high stability of the active materials used in ETP multipliers, they come with a 2-year shelf life warranty (store in original sealed package). Many testing laboratories take advantage of this long shelf life by keeping a replacement ETP multiplier on hand, ready for immediate installation. This keeps the instrument down time to a minimum.

For a typical ETP electron multiplier for GC/MS, the total active dynode surface area is ~1000mm². This can be compared to a standard continuous dynode multiplier that has a total channel surface area of only around 160mm² (for a channel with 1mm diameter and 50mm length). This increased surface area spreads out the work-load of the electron multiplication process over a larger area, effectively slowing the aging process and improving operating life and gain stability.

ETP Electron Multipliers for Mass Spectrometry

Description	qty.	cat.#
Electron Multipliers for Agilent GC-MS and LC-MS		
For Agilent 5970 GC-MS	ea.	23072
For Agilent 5971, 5972, GCD GC-MS	ea.	23073
For Agilent 5973 & 5975 GC-MS (includes mount for initial installation)*†	ea.	23074
For Agilent 5973 & 5975 GC-MS and LC-MSD (Replacement Multiplier)*†	ea.	23075
For Agilent LC-MSD (includes mount for initial installation)*†	ea.	23076
Electron Multipliers for Applied Biosystems (Sciex)		
For API 300, 3000 & 4000 Applied Biosystems	ea.	23077

*Note: The electron multipliers have been specifically developed to retrofit the original manufacturer's equipment. The detector incorporates a modular design to facilitate ease of replacement and additional innovations intended to enhance performance. First time installation requires a mount which includes the mechanical housing. After initial installation, only the replacement electron multiplier is required.

†This unit is designed for use in the 5975, 5973 GC and the LC/MSD.

for **more** info

For more information on ETP Electron Multipliers, request lit. cat.# GNFL1000.

please note

Other electron multipliers are available upon request. Call 800-356-1688 ext. 4, or contact your local Restek representative, for information on other models.

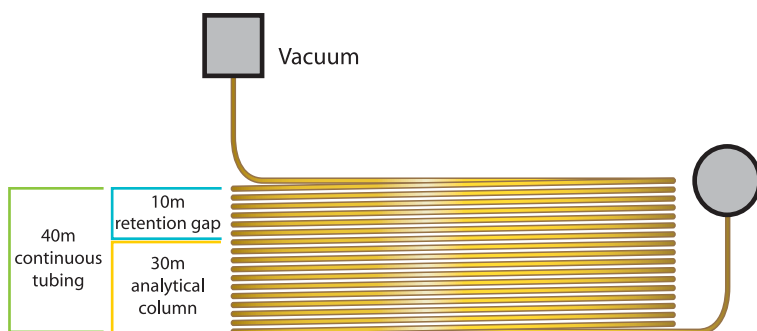
Using Guard Columns and Retention Gaps in GC (Part 2) *Continued from page 2.*

Segment coating technology eliminates problematic connections

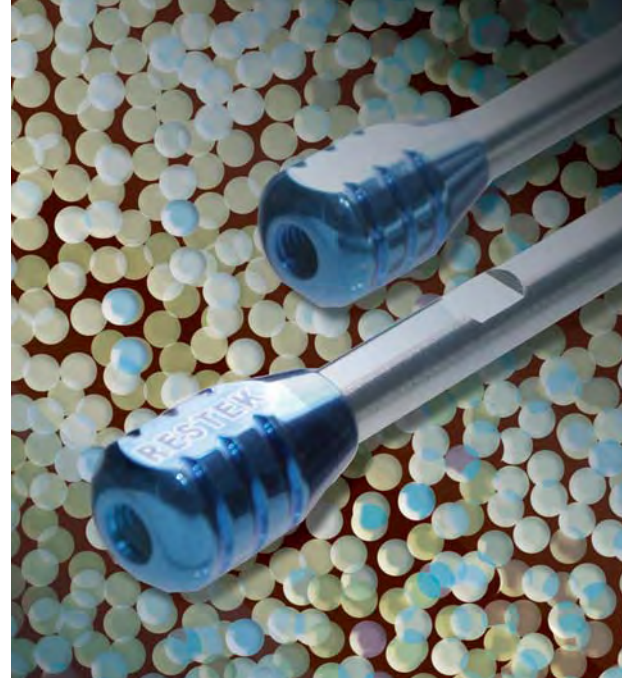
Both retention gaps and guard columns must be coupled to the analytical column. While there are several types of effective coupling devices, all can create dead volume and can be a potential source of leaks and reactivity. Segment coating technology allows the retention gap or guard column to be built directly in the same piece of tubing as the analytical column, eliminating the connector and associated risks. This technology, available from Restek, is termed Integra-Guard™ or Integra-Gap™ and is based on the static coating method. In this process the capillary column is filled with a coating solution of stationary phase in a volatile solvent. The column is sealed on one end and on the other side a vacuum is applied. The solvent is evaporated and the dissolved polymer is deposited on the inside deactivated wall of the fused silica column. The static coating method allows columns to be coated by segment. When filling, for example, a 40m capillary with the coating solution, only 30m are filled. The first 10m remain uncoated, having only the deactivation treatment (Figure 1). This method deposits the stationary phase only in a designated portion of the capillary, creating the Integra-Guard™ or the Integra-Gap™. The advantages of this technology are clear: eliminating the connector removes a potential source of leaks and reduces dead volume. Additionally, maintenance is faster and simpler since there is no manual connection to make.

Guard columns and retention gaps are useful tools to the practicing chemist, and it is important to understand the difference between them. While they help protect analytical columns and focus samples, respectively, they are also a source of potential problems, such as leaks. Segment coating technology offers a better solution—integrated columns containing both the guard or gap section and the analytical column together in a single piece of tubing. These Integra-Guard™ and Integra-Gap™ columns are a simple, effective solution; they eliminate the risks of a separate connection and provide stable, accurate data.

Figure 1 Static coating allows Integra-Gap™ integrated retention gaps to be built directly into the analytical column tubing.



For more information on Integra-Gap™ technology, see “Selecting a GC Column for Glycerin in Biodiesel” on page 10.



Interested in Learning More About UHPLC?

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Course Topics

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- The HPLC (and UHPLC) Column
- Developing a UHPLC Method
- Transferring Methods
- Tips and Techniques for UHPLC

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March 17	Columbia, MD	65765
March 18	Bridgewater, NJ	65766
March 20	Malvern, PA	65767
April 23	Atlanta, GA	65768
June 10	St. Louis, MO	65769
June 12	Cincinnati, OH	65770
June 13	Pittsburgh, PA	65771
<i>Canada</i>		
April 24	Montreal, PQ	65772
April 21	Toronto, ON	65773
April 22	Toronto, ON	65774
April 23	Burlington, ON	65775

Visit us at www.restek.com/uhplc for more information or to register.

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your friends from Restek.

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Marriot New Orleans Convention Center
located at 859 Convention Center Blvd.,
New Orleans, LA.

Monday & Tuesday

March 3-4 from 5-8pm

Register online at
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or give us a call at 800-356-1688

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" some" **promos / Products / Offers** in the **ADVNews**
have been since been progressively superceded
/ **UPDATED OR Since Discontinued**

CHECK THE latest Restek ADVantage Newsletter, Restek ESSENTIALS
... Or The Restek Catalog ... Or other Restek publications for updates
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Lit. Cat.# GNAD1017

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