the RESTEK Advantage

Innovators of High Resolution Chromatography Products

Low-Bleed, High-Temperature Column for ASTM Method D-6352

MXT®-1HT Sim Dist GC Column

by Neil Mosesman, GC Columns Product Marketing Manager, and Dinesh V. Patwardhan, Ph.D., Senior Research Chemist

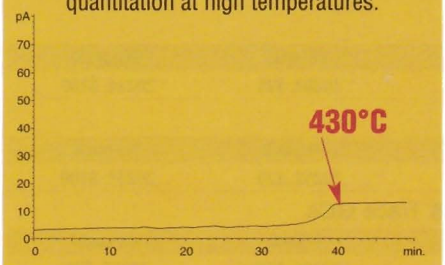
new

- ✔ Durable metal tubing and high-temperature polymer allows analysis of hydrocarbons up to C110.
- ✔ Low bleed at high temperatures help achieve accurate quantitation.
- ✔ Meets all criteria of ASTM Method D-6352.

The American Society for Testing and Materials (ASTM) is an organization that publishes consensus standards for materials, products, and services. Because ASTM methods are developed by consensus from the laboratories following the method, they are recognized worldwide. ASTM Method D-6352 is a gas chromatography (GC) method developed for the determination of petroleum distillates with a boiling point range of 174°C to 700°C. Often referred to as high-temperature simulated distillation or Sim Dist, this method requires a capillary column capable of withstanding high GC oven temperatures, up to 430°C. This presents many challenges for analysts because most capillary

Figure 1

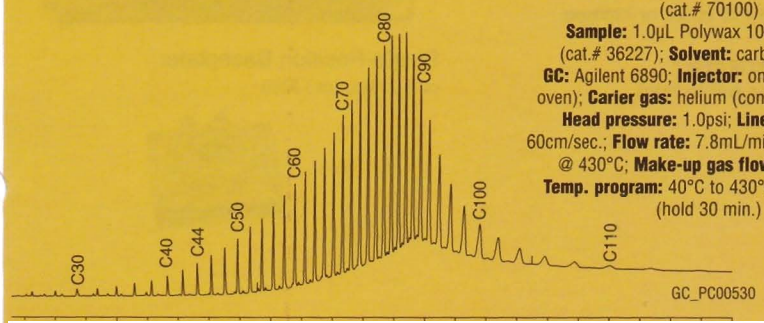
The MXT®-1HT Sim Dist column exhibits low bleed, which gives accurate quantitation at high temperatures.



5m, 0.53mm ID, 0.10µm MXT®-1HT (cat.# 70100) **GC:** Agilent 6890; **Injector:** on-column (track oven); **Carier gas:** helium (constant pressure); **Head pressure:** 1.0psi; **Linear velocity:** 60cm/sec.; **Flow rate:** 7.8mL/min.; **Detector:** FID @ 430°C; **Make-up gas flow:** 40cc/min.; **Temp. program:** 40°C to 430°C @ 10°C/min. (hold 30 min.)

Figure 2

The MXT®-1HT Sim Dist column allows analysis of hydrocarbons up to C110 in less than one hour.



5m, 0.53mm ID, 0.10µm MXT®-1HT Sim Dist (cat.# 70100) **Sample:** 1.0µL Polywax 1000 standard (cat.# 36227); **Solvent:** carbon disulfide; **GC:** Agilent 6890; **Injector:** on-column (track oven); **Carier gas:** helium (constant pressure); **Head pressure:** 1.0psi; **Linear velocity:** 60cm/sec.; **Flow rate:** 7.8mL/min.; **Detector:** FID @ 430°C; **Make-up gas flow:** 40cc/min.; **Temp. program:** 40°C to 430°C @ 10°C/min. (hold 30 min.)

in this issue

RESTEK
800-356-1688 ★ 814-353-1300
www.restekcorp.com

Fall 2001

columns are manufactured using polyimide-coated fused silica tubing. At temperatures above 380°C, even the best polyimide coating becomes brittle, which leads to very short column lifetimes. In addition, the methyl silicone stationary phase recommended in the method also must survive these high temperatures.

Extensive research by scientists at Restek has led to a major improvement in columns for high-temperature simulated distillation. By combining a new proprietary polymer synthesis technology, Siltek™ deactivation chemistry, and rugged Silcosteel® tubing, we developed a capillary column that meets all the criteria of ASTM Method D-6352. Because the MXT®-1HT Sim Dist column is coated with a 100% dimethyl polysiloxane polymer, it will give the correct retention time/boiling point curve. The MXT®-1HT Sim Dist column exhibits low bleed at 430°C and excellent peak shape due to the unique polymer synthesis and Siltek™ deactivation (Figure 1).

The rugged Silcosteel® tubing will hold up indefinitely to temperatures in excess of 430°C, so column lifetime is not limited by the tubing. To demonstrate the utility of this innovative product, we analyzed Polywax® 1000 using the MXT®-1HT Sim Dist column (Figure 2). Notice the excellent peak shape of hydrocarbons up to C110.

To maintain the low bleed and high performance of the MXT®-1HT Sim Dist column, it is critical to prevent oxygen from entering the column. This can be achieved by electronically leak checking your entire system. We also recommend the use of graphite ferrules; Vespel® or Vespel®/graphite ferrules are more likely to loosen over time.

The MXT®-1HT Sim Dist column is available in a 5m, 0.53mm ID, 0.1µm film to conform to the requirements of ASTM Method D-6352. It exceeds the criteria for resolution, peak shape, and bleed for hydrocarbons ranging up to C110.

Hot Tech Tip

Many GC problems can be avoided by electronic leak checking the system during the plumbing process. Thorough leak checking will prevent loss of GC gases, damage to capillary columns, and increased detector maintenance. Oxygen can move into the system via a leak due to the Venturi effect, and irreversible damage can occur if a column is exposed to oxygen at high temperatures, such as those needed for simulated distillation. Also, some detectors are very sensitive to oxygen. Leak checking the instrument before column installation and conditioning prevents column degradations indicated by high bleed and short lifetimes. Leak checking should be performed on the entire gas system and GC. Begin by checking all fittings inside the GC. Next check the external fittings along the carrier gas lines, all the way to the tanks. Never use liquid leak detectors that contain soap or surfactants because liquids can be drawn inside the fitting at the site of the leak and contaminate the system.

MXT®-1HT SimDist (metal column)

Length (m)	ID (mm)	df(µm)	Temp. Limits	cat.#
5	0.53	0.10	-60 to 430°C	70100, $300

***Please note:** For high-temperature analyses such as simulated distillation, Restek strongly recommends the use of the following accessories to ensure low bleed and maintain high performance.*

Capillary Graphite Ferrules

For 1/16" compression-type fittings

Ferrule ID (mm)	Fits Column ID (mm)	10-pack	50-pack
0.8	0.53	20202, $25	20224, $100

For Agilent GCs (compact ferrules)

Ferrule ID (mm)	Fits Column ID (mm)	10-pack	50-pack
0.8	0.53	20252, $25	20253, $100

For M4 fittings for QCQ ThermoQuest 8000 & Trace GCs

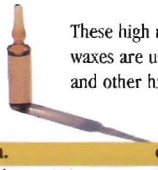
Ferrule ID (mm)	Fits Column ID (mm)	2-pack	10-pack
0.8	0.53	20284, $18	20285, $75

Standard Graphite Ferrules

For 1/4" fittings

Fitting Size (")	Ferrule ID (")	10-pack
1/4	1/4	20210, $25

D6352-98 Polywax® Standards



These high molecular weight hydrocarbon waxes are useful for simulated distillation and other high-temperature GC work.

Ea.	cat.#	qty.	price
Polywax 500	36224	1 gram	$10
Polywax 655	36225	1 gram	$10
Polywax 850	36226	1 gram	$10
Polywax 1000	36227	1 gram	$10

Super-Clean™ SGT Gas Filters

- High-purity output (99.9999% purity).
- Features a "quick connect" for fast and simple cartridge changes.
- Full glass/metal design with easy-to-read indicators.

Ultra-High Capacity Oxygen Filter:
cat.# 22029, (ea.) $115

Single-Position Baseplate:
cat.# 22025, (ea.) $190

Leak Detective™ Electronic Leak Detector

- Compact, lightweight, hand-held design.
- Lowest-cost thermal conductivity leak detector available.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at ≥3 x 10^{-6}cc/sec. or ≥200ppm.
- Audible alarm and LED readout.
- Responds in less than 2 seconds to trace leaks of gases.*
- Operates on two 9-volt batteries or AC adaptor, both included.

(110 VAC): cat.# 21607, (ea.) $595
(220 VAC): cat.# 21609, (ea.) $645
European 2-prong plug (220 VAC): cat.# 21382 (ea.) $645
*Not designed for use in explosive atmospheres.

Trace-Level Analysis of Explosives by HPLC

Pinnacle II™ C18 & Cyano Columns

by Greg France, HPLC Product Marketing Manager

- ✔ Strictly controlled silica manufacturing ensures reproducible chromatography.
- ✔ Sharp peak shape and excellent efficiency for explosives.
- ✔ Economically priced.

Pinnacle II™ high performance liquid chromatography (HPLC) stationary phases were designed to function well under the difficult matrices encountered in environmental samples. The original Pinnacle™ columns served as benchmarks for the selectivity and efficiency of these new Pinnacle II™ columns. While striving to create columns with characteristics similar to Pinnacle™ columns, Restek designed them using Restek silica. Now, we can go a step further in providing consistent quality and reproducibility by controlling the manufacturing process back to the raw material stage.

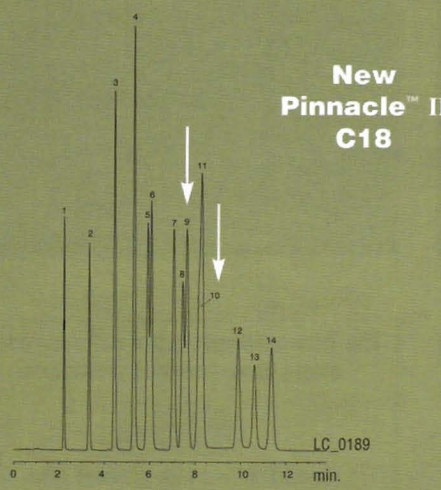
The new Pinnacle II™ C18 and Cyano columns function as primary and confirmation columns (respectively) to efficiently separate explosives according to US Environmental Protection Agency (EPA) Method 8330A. Environmental methods frequently employ a confirmation column for two reasons. First, many environmental methods require scanning for a large number of related compounds. Because of their similarities, analysts often will encounter coelutions when using a single type of stationary phase. Second, the matrices encountered in many environmental samples can contain components that may interfere or obscure the analytes of interest. By using two columns with different selectivities, analysts can more accurately identify the analytes of interest.

Selectivity for the 14 explosives of interest listed in Method 8330A are similar on the original Pinnacle™ and the new Pinnacle II™ C18 columns (Figure 1). On these C18 columns, there are closely eluting peaks or coelutions for the following compounds: tetryl/nitrobenzene; 2-amino-4,6-dinitrotoluene/4-amino-2,6-dinitrotoluene; and 2,6-dinitrotoluene/2,4-dinitrotoluene. Closer examination shows that the new Pinnacle II™ C18 column achieves better resolution for two of these pairs. This may be caused by the slightly higher surface area and carbon load, and the smaller pore size on the Pinnacle II™ column (110Å) as compared to the Pinnacle™ column (120Å). The higher carbon load of 13% for the Pinnacle II™ column, versus 11% for the Pinnacle™ column, translates into longer compound retention, and better resolution and column lifetime.

According to Method 8330A, these 14 compounds also need to be analyzed on a Cyano column for confirmation (Figure 2). Changing from a reversed phase C18 column to a normal phase Cyano column is fairly easy. The method recommends using the same mobile phase for both columns, which allows a quick changeover from the primary analysis to the confirmation analysis. Because the mobile phase is a simple mixture of water and methanol, the process of switching from the C18 to the Cyano column is only a matter of removing the primary column and installing the confirmation column on the same HPLC system There is relatively little downtime, only that required for the system to re-equilibrate.

Figure 1

The new Pinnacle II™ C18 column achieves better resolution for difficult-to-analyze pairs of explosive compounds.

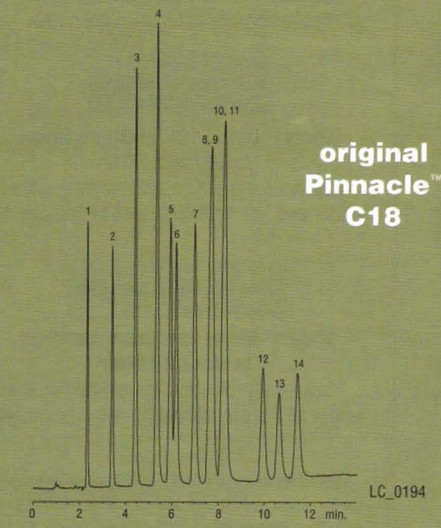


Column: Pinnacle II™ C18
Catalog #: 9214575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5 mL/min
Temp.: 27°C
Det.: UV @ 254nm

Sample:
Inj.: 3µL
Conc.: 500µg/mL each component
Solvent: acetonitrile

Restek 8330 Calibration mix #1 (cat. # 31450)
Restek 8330 Calibration Mix #2: (cat. # 31451)



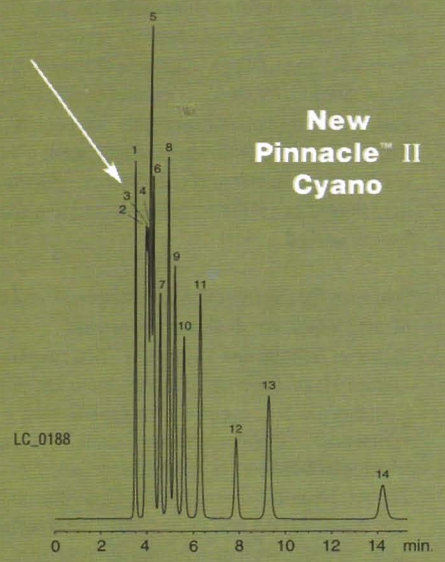
Column: Pinnacle™ C18
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Peak List for Figure 1

1. HMX
2. RDX
3. 1,3,5-trinitrobenzene
4. 1,3-dinitrobenzene
5. tetryl
6. nitrobenzene
7. 2,4,6-trinitrotoluene
8. 2-amino-4,6-dinitrotoluene
9. 4-amino-2,6-dinitrotoluene
10. 2,6-dinitrotoluene
11. 2,4-dinitrotoluene
12. 2-nitrotoluene
13. 4-nitrotoluene
14. 3-nitrotoluene

Figure 2

The Pinnacle II™ Cyano column shows better resolution than the original Pinnacle™ Cyano column for explosives compounds, and is an excellent confirmational column to the Pinnacle II™ C18 for this analysis.

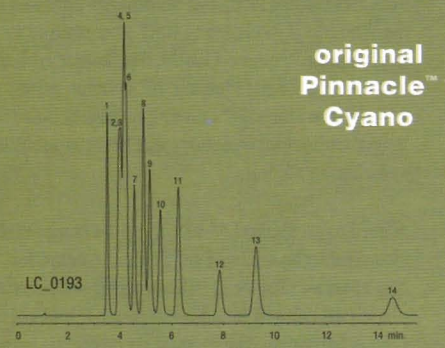


Column: Pinnacle II™ Cyano
Catalog #: 9216575
Dimensions: 250 x 4.6mm
Particle Size: 5µm
Pore Size: 110Å

Conditions:
Mobile phase: water:methanol (50:50, v/v)
Flow: 1.5mL/min
Temp.: 27°C
Det.: UV @ 254nm

Sample:
Inj.: 3µL
Conc.: 500 µg/mL each component
Solvent: acetonitrile

Restek 8330 Calibration mix #1 (cat.# 31450)
Restek 8330 Calibration Mix #2 (cat.# 31451)



Column: Pinnacle™ Cyano
Catalog #: 9116575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 120Å

Notice that all of the coeluting pairs from the C18 column are resolved from one another on the Cyano column. There is a cluster of compounds: 2-nitrotoluene, 3-nitrotoluene, 4-nitrotoluene, 1,3,5-trinitrotoluene, and 1,3 dinitrotoluene on the Cyano column, but these compounds are well resolved on the C18 column. Again, the selectivity between the original Pinnacle™ Cyano and the new Pinnacle II™ Cyano columns is similar, but the Pinnacle II™ column shows better resolution.

Restek controls the raw material quality from the very beginning of the silica manufacturing process. Add our phase bonding and column packing experience to this high level of quality control, and you benefit from even better column-to-column reproducibility. Because of this and their economical production, Pinnacle II™ HPLC columns provide a cost-effective analytical tool for many traditional methods used in the environmental industry.

Peak List for Figure 2

1. nitrobenzene
2. 2-nitrotoluene
3. 4-nitrotoluene
4. 3-nitrotoluene
5. 1,3-dinitrobenzene
6. 1,3,5-trinitrobenzene
7. 2,6 dinitrotoluene
8. 2,4-dinitrotoluene
9. 2,4,6-trinitrotoluene
10. 4-amino-2,6-dinitrotoluene
11. 2-amino-4,6-dinitrotoluene
12. RDX
13. tetryl
14. HMX

Pinnacle II™ C18 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID	
50mm	9214551	9214552	9214553	9214555	$235
100mm	9214511	9214512	9214513	9214515	$250
150mm	9214561	9214562	9214563	9214565	$265
250mm	9214571	9214572	9214573	9214575	$295

Pinnacle II™ Cyano 5µm Columns

Length	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID	
50mm	9216551	9216552	9216553	9216555	$235
100mm	9216511	9216512	9216513	9216515	$250
150mm	9216561	9216562	9216563	9216565	$265
250mm	9216571	9216572	9216573	9216575	$295

Trident™ HPLC Guard Column Cartridges

Guard Column Cartridge	10 x 2.1mm 3-pk.	10 x 4.0mm 3-pk.	20 x 4.0mm 2-pk.	
Pinnacle II™ C18	921450212	921450210	921450220	$125
Pinnacle II™ Cyano	921650212	921650210	921650220	$125

Trident™ Direct Guard Column System*

Description	qty.	cat.#	
High pressure filter	each	25082	$75
1cm guard cartridge holder with filter	each	25084	$95
2cm guard cartridge holder with filter	each	25086	$95
PEEK® connection tip for Waters®-style end fittings	each	25088	$10
Replacement cap frits: 4mm, 2.0µm	5-pack	25022	$25
Replacement cap frits: 4mm, 0.5µm	5-pack	25023	$30
Replacement cap frits: 2mm, 2.0µm	5-pack	25057	$30

The Trident™ Direct guard column system offers three levels of protection:

Trident™ Direct high-pressure filter
Protection against particulate matter

Trident™ Direct 1cm guard cartridge holder with filter
Moderate protection against particulate matter and irreversibly-adsorbed compounds

Trident™ Direct 2cm guard cartridge holder with filter
Maximum protection against particulate matter and irreversibly-adsorbed compounds

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

Fast Semivolatile Analysis by GC/MS Using Performance-Based Measurement Systems

Rtx®-5Sil MS Columns and Uniliner® Liners

by Gary Stidsen, Innovations Team Manager

- ✔ Decrease analysis time to 22 minutes for increased lab throughput.
- ✔ Resolve key analytes.
- ✔ Analytical conditions can be used for all MS detectors.

Restek has developed a fast GC/MS method for analyzing semivolatile compounds [e.g., US Environmental Protection Agency (EPA) Method 8270] that will help increase productivity in the lab. The changes include modification of the final extract volume, use of the DI Uniliner with a hole, shorter GC analysis time, and a modification of the calibration curve to offset the increased extract volume. Following is an explanation of each modification. For more detailed information, please request application note #59125.

1) Increase the extract volume

Increase the final extract volume from 1mL to 5mL. This will reduce preparation time and the amount of low-boiling compounds lost from evaporation. Also, one-fifth the amount of matrix imterferences will be injected into the GC. The reduction of matrix interferences will allow the instrument to stay calibrated for more sample windows.

2) Use a Uniliner® DI injection port liner with a hole

This unique inlet liner can be used for direct and splitless injection. The column is fixed via a press-tight connection at the bottom of the liner, eliminating any sample contact with metal parts below the liner. In order for the carrier gas to be routed through the split vent line, a hole has been drilled into the side of the liner. This hole allows the carrier gas to be vented through the split vent line during the split operation of the injection port. The Uniliner® liner with a hole provides a more inert sample pathway that minimizes injection port discrimination and active compound loss.

3) Use a thin-film Rtx®-5Sil MS column

Lower concentration standards allow the use of a thinner-film column, which can reduce analysis time. A thin-film Rtx®-5Sil MS column can achieve improved resolution of difficult-to-analyze compounds in less than 22 minutes. This column features a silarylene stationary phase and optimized dimensions that are ideal for the analysis of semivolatile compounds (Figure 1).

4) Oven temperature programming

Adjusting the oven temperature program of the GC will help optimize the separation of critical pairs. Use of an initial hold time resolves early-eluting compounds; then a fast ramp rate can be used through non-critical areas, and a lower ramp rate used to elute later compounds.

5) Calibration curve

We used 1/5 the recommended concentration level of Method 8270—1µL injection of 4, 10, 16, 24, and 32ppm standard. Notice how the 4ppm (4ng on-column) injection shows excellent signal-to-noise ratio, and low column bleed and injection port discrimination (Figure 1).

Conclusion

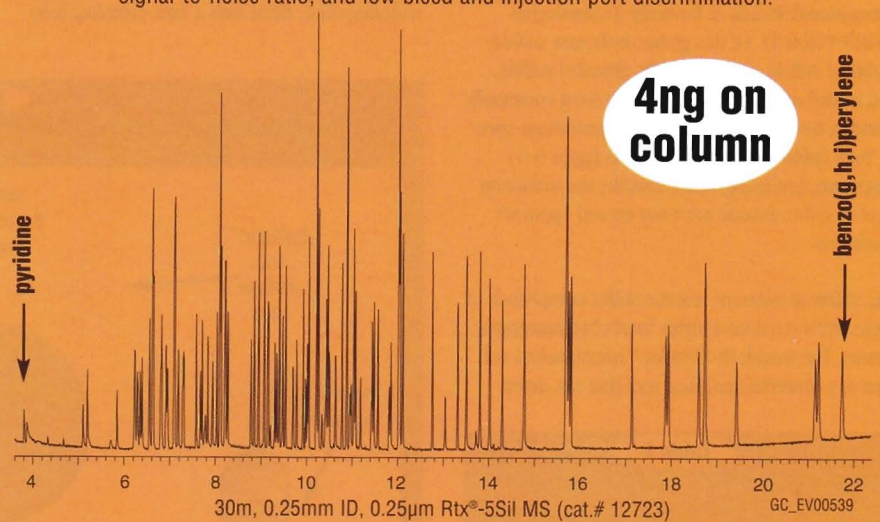
A number of techniques can be used to increase sample throughput for the analysis of semivolatile compounds according to US EPA Method 8270. Increasing extract volume will reduce preparation time and injection port contamination. Using a Uniliner® injection port liner with a hole results in a more inert sample pathway and eliminates injection port discrimination. In addition, the use of a thin-film column reduces analysis time.

for more info

on the analysis of semivolatile compounds, request lit. cat.# #59411, *A Guide to Preparing and Analyzing Semivolatile Organic Compounds.*

Figure 1

Fast analysis of Method 8270 semivolatile compounds with a Uniliner® liner achieves excellent signal-to-noise ratio, and low bleed and injection port discrimination.



30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)

Inj.: 1µL splitless injection of 8270 Calibration Mix #1 (cat.# 31618), 8270 Calibration Mix #2 (cat.# 31619), 8270 Calibration Mix #3 (cat.# 31620), 8270 Calibration Mix #4 (cat.# 31621), 8270 Calibration Mix #5 (cat.# 31622), BN Surrogate (cat.# 31024), Acid Surrogate (cat.# 31025), SU Internal Standard (cat.# 31006); **Flow rate:** helium, constant flow @ 1mL/min.; **Oven temp.:** 35°C (hold 2 min.) to 260°C @ 20°C/min. to 330°C @ 6°C/min. (hold 1 min.); **Inj. temp.:** 300°C; **Detector:** Agilent 6890 w/ 5973 MS; **MS transfer temp.:** 280°C; **Inlet liner:** 4.0mm ID Uniliner® w/hole (cat.# 21054)

Rtx®-5Sil MS (fused silica)

Length (m)	ID (mm)	df(µm)	Temp. Limits	cat.#
30	0.25	0.25	-60 to 330/350°C	12723, $435
30	0.25	0.50	-60 to 330/350°C	12738, $435

Uniliner® Liner with Hole

4.0mm ID, 6.3mm OD, 78.5mm length

each	5-pk.
21054, $60	21055, $240

Analyzing Trace Sulfur Compounds in CO_2

Rt-XLSulfur™ Packed Column and Sulfinert™ System

by Neil Mosesman, GC Columns Product Marketing Manager, and Barry Burger, Innovations Chemist

✔ Detects sulfur compounds at low ppbv levels.
✔ Thermal stability to 300°C for longer column lifetime.

The taste and aroma of a carbonated beverage can be affected by trace impurities from the carbonation process. Therefore, gas producers go to great lengths to purify carbon dioxide (CO_2). Carbon dioxide, a by-product of oil refining, fermentation, and power generating facilities, must be extremely pure to be suitable for a beverage additive. The beverage industry has spent much research time and money monitoring the impurities in CO_2.

The most common impurities in CO_2 are hydrocarbons, alcohols, permanent gases, and sulfur compounds. Sulfur impurities are the predominant problem, adding unwanted taste and odor to beverages. The most common volatile sulfur compounds (VSC) impurities are targeted for monitoring by the International Society of Beverage Technologists (ISBT) (Table I). Of this group, hydrogen sulfide, carbonyl sulfide, sulfur dioxide, dimethyl sulfide, and methyl mercaptan are the ones most commonly found in beverage-grade CO_2. ISBT guidelines specify Total Sulfur Content* (TSC) as 0.1ppm (v/v) maximum, excluding sulfur dioxide; the maximum level of sulfur dioxide must not exceed 1ppm v/v maximum.

The ability to measure reactive sulfur compounds at these levels requires a highly inert chromatography system. The Restek Rt-XLSulfur™ micropacked column is a powerful analytical tool that can detect sulfurs in CO_2 at levels of 20ppbv, far below the ISBT guideline for total sulfur content. This column also achieves the critical separation of hydrogen sulfide, carbonyl sulfide, and sulfur dioxide as defined in ISBT Procedure 14.0. The Rt-XLSulfur™ micropacked column contains a modified divinyl benzene polymer packed into Sulfinert™ tubing, which is a metal tubing specially deactivated for monitoring ppbv levels of active sulfur compounds. Other features of the Rt-XLSulfur™ column include low bleed and thermal stability up to 300°C.

Sample introduction into the column is another critical aspect of obtaining accurate analytical results for sulfur compounds. The sample is introduced onto the column using a Valco® six-port sampling valve, fitted with a 1mL sampling loop (cat. #22845). When the valve, sample loop, and all other surfaces in the sample pathway are deactivated using the Sulfinert™ process, the analyst will see improved response compared to systems using conventional deactivations. We suggest connecting your bulk CO_2 via this system (Figure 1). The specialized inertness of the Sulfinert™ process is critical for the system to achieve detection limits of 50ppbv for sulfur dioxide and the other target sulfur impurities.

We evaluated the effectiveness of the RT-XLSulfur™ column and Sulfinert™ sampling system by analyzing bulk CO_2 and CO_2 spiked with a sulfur standard (Figure 2). Notice how even low ppbv of sulfurs can be detected. We also sampled and measured the TSC* of two top brands of cola and a domestic beer (Figures 3 and 4). The colas show no sulfur content, verifying that the CO_2 used for carbonation was clean. The beer sample shows sulfur compounds that naturally occur during the fermentation process.

This system is sensitive enough to monitor the levels of sulfur in CO_2 during the carbonation process, or in the headspace of the beverage after carbonation. The TSC* generated from headspace sampling of these products demonstrates the ability of the RT-XLSulfur™ column and the Sulfinert™-deactivated GC system to easily detect sulfur compounds at the 20ppbv level. The combination of the Rt-XLSulfur™ micropacked column and a Sulfinert™-deactivated sample introduction system provide a state-of-the-art, robust sampling and analysis technique for ppb levels of VSCs in beverage-grade CO_2.

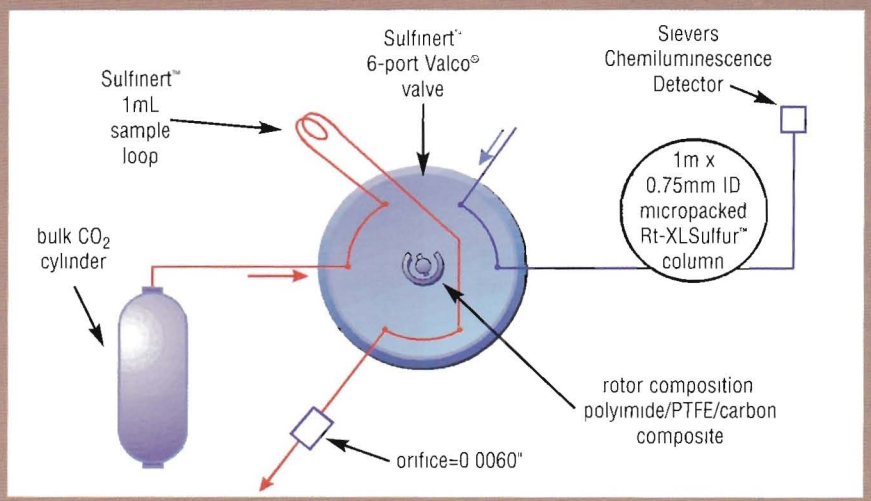
Table I

Sulfur compounds can affect the taste and aroma of beer.

hydrogen sulfide	isopropyl mercaptan
carbonyl sulfide	methyl ethyl sulfide
methyl mercaptan	*n*-propyl mercaptan
ethyl mercaptan	*tert*-butyl mercaptan
sulfur dioxide	*sec*-butyl mercaptan
dimethyl sulfide	diethyl sulfide
dimethy disulfide	isobutyl mercaptan
carbon disulfide	*n*-butyl mercaptan
tert-amyl mercaptan	

Figure 1

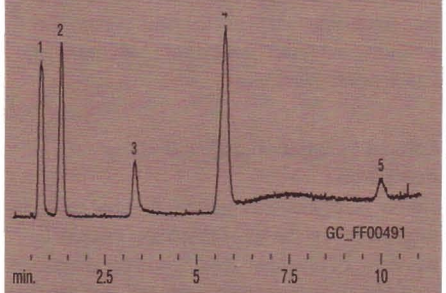
A sampling system was designed to optimize trace-level sulfur analysis without adsorption.



*Total Sulfur Content seen with an asterisk indicates it is without SO_2.

Figure 2

Easily achieve 20ppbv detection limits of reactive sulfur compounds using the Rt-XLSulfur™ packed column and Sulfinert™-treated sample pathways.

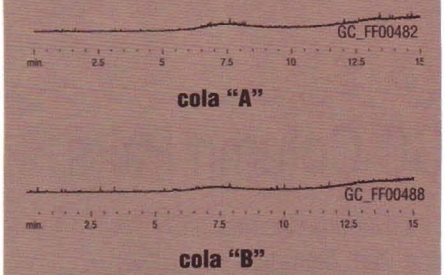


1. hydrogen sulfide
2. carbonyl sulfide
3. methyl mercaptan
4. ethyl mercaptan and/or dimethyl sulfide
5. dimethyl disulfide

1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.); **Det. temp.:** 800°C; **Carrier gas:** He; **Flow rate:** 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector) Attn. x 1; **Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** sulfur standard @ 20ppb each in CO_2

Figure 3

Two samples of popular colas show no sulfur compounds.



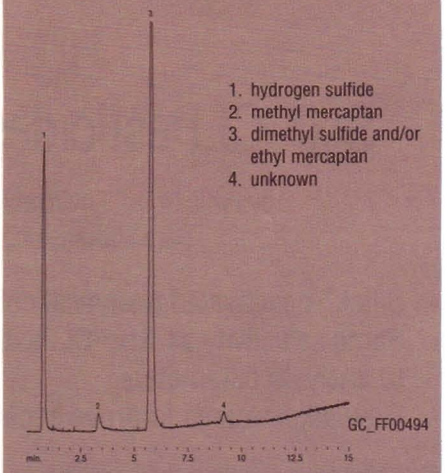
1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.); **Det. temp.:** 800°C; **Carrier gas:** He; **Flow rate:** 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector) Attn. x 1; **Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** head space of colas

for more info

on Restek's complete line of packed and micropacked columns, and bulk packing materials, request the *Packed Column Catalog* (lit. cat.# 59986A).

Figure 4

A sample of domestic (US) beer contains ppbv levels of hydrogen sulfide, dimethyl sulfide and/or ethyl mercaptan, and methyl mercaptan.



1. hydrogen sulfide
2. methyl mercaptan
3. dimethyl sulfide and/or ethyl mercaptan
4. unknown

1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.); **Det. temp.:** 800°C; **Carrier gas:** He; **Flow rate:** 10mL/ min. @ ambient temp.; **Detector sensitivity:** SCD (Sievers Chemiluminescence Detector) Attn. x 1; **Inj.:** 1cc sample loop; **Inj. method:** 6-port Valco® valve; **Std. concentration:** head space of a domestic (US) beer sample

Sulfinert™ Sample Loops

size	cat.#	
5µL	22840	$115
10µL	22841	$115
20µL	22842	$115
25µL	22843	$115
50µL	22844	$115
100µL	22845	$115
250µL	22846	$115
500µL	22847	$115
1cc	22848	$115
2cc	22849	$115
5cc	22850	$115

6-Port Valco® Valve

The 6-port Valco® valve was coated with Sulfinert™ treatment on a custom basis. For custom Sulfinert™ quotes, call customer service at ext. 3, or contact your local Restek representative.

* *Please include configuration suffix number when ordering.*

***Installation kit must be purchased with column when using for valve applications.*

for more info

on Sulfinert™ coating's features and benefits, and a detailed product listing, *Sulfinert™ Flyer* (lit. cat.# 59203).

Rt-XLSulfur™ Packed and Micropacked Columns

OD (in.)	ID (mm)	1-Meter	2-Meter
1/16**	1.0	19804, $125	19805, $150
0.95mm**	0.75	19806, $125	19807, $150
1/8**	2.0	80484*, $125	80485*, $210
3/16	3.2	80482*, $205	80483*, $380

Installation Kits

	for 0.75mm ID col.	for 1mm ID col.	for 2mm ID col.
For valve applications	21062, $105	21065, $30	21067, $25
For split applications	21063, $15	—	—
For all Agilent GCs	21064, $15	—	—
For direct injections	—	21066, $45	—

Packed Column Configurations

Custom configurations are available. Please contact Customer Service (ext. 3) or your local Restek representative.

General Configuration: **Suffix -800**

Agilent 5880, 5890, 5987: **Suffix -810**

Varian 3700, Vista Series, FID: **Suffix -820**

PE 900-3920, Sigma 1,2,3: **Suffix -830**

PE Auto System 8300, 8400, 8700 (Not On-Column): **Suffix -840**

Sulfinert™ Tubing (Price-per-foot by length)

ID, OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
316 Seamless Stainless Steel Tubing (0.035" wall thickness)					
0.011" (0.28mm), .022" (0.56mm),	22500	$10	$6.25	$4.50	$3.75
0.021" (0.53mm), .029" (0.74mm)	22501	$10	$6.25	$4.50	$3.75
0.010" (0.25mm), 1/16" (1.59mm)	22502	$10	$6.25	$4.50	$3.75
0.020" (0.51mm), 1/16" (1.59mm)	22503	$10	$6.25	$4.50	$3.75
0.030" (0.76mm), 1/16" (1.59mm)	22504	$10	$6.25	$4.50	$3.75
0.040" (1.02mm), 1/16" (1.59mm)	22505	$10	$6.25	$4.50	$3.75
0.085" (2.16mm), 1/8" (3.18mm)*	22506	$10	$6.25	$4.50	$3.75
0.210" (5.33mm), 1/4" (6.35mm)*	22507	$15	$8.75	$6.25	$5
316 Seamless Stainless Steel Tubing (0.035" wall thickness)					
0.055" (1.40mm), 1/8" (3.18mm)	22508	$18.75	$15	$12.50	$10
0.180" (4.57mm), 1/4" (6.35mm)	22509	$18.75	$15	$12.50	$10

Reduced Endrin Breakdown for Chlorinated Pesticides Analysis

Stx™-CLPesticides GC Column Pairs

by Lydia Nolan, Environmental Innovations Chemist, and
Gary Stidsen, Innovations Team Manager

- ✔ Siltek™-deactivated analytical columns decrease endrin breakdown.
- ✔ Same selectivity as Rtx®-CLPesticides column pairs—no modification to analysis parameters.
- ✔ Flow adjustments improve separation.

Many laboratories performing gas chromatography (GC) analysis of chlorinated pesticides struggle with endrin breakdown caused by the compound adsorbing to active sites throughout the analytical system, especially in the injection port and the analytical column. Restek Siltek™ technology—used successfully to passivate injection port liners and guard columns—is now available in Stx™-CLPesticides and Stx™-CLPesticides2 capillary columns. The combination of a properly deactivated injection system and inert analytical columns provides the lowest possible level of endrin breakdown.

Method Requirements

Chlorinated pesticide analyses following US Environmental Protection Agency (EPA) Methods 8081, 608, 505 and 508 recommend dual-column confirmation using electron capture detection (ECD). The most common analytes of interest are analyzed in Figure 1. As in all analytical methods, the instrument used for quantitative analysis must be calibrated to ensure accurate results are reported. For chlorinated pesticides this usually entails a calibration curve of three to five points and check standards injected at specified time intervals during sample analysis. In addition, performance standards containing endrin are analyzed periodically to ensure system inertness. Typically, endrin breaks down to endrin aldehyde and endrin ketone when there are active sites in the sample pathway, and their presence must also be verified.

Endrin Breakdown

Maintaining a low breakdown level for endrin is necessary for laboratories analyzing chlorinated pesticides. Reduction of endrin breakdown generally focuses on improving the inertness of the injection port (see sidebar on page 9). Recently, liners treated with Siltek™ deactivation were designed to prevent endrin breakdown in the injection port. This innovative deactivation technology also was incorporated into capillary guard tubing so that the entire sample introduction pathway is as inert as possible.

Endrin Response

In addition to breakdown, endrin response can be reduced by irreversable adsorption onto active sites in the sample pathway. To minimize on-column adsorption of endrin, we incorporated Siltek™ deactivation technology into the analytical columns.

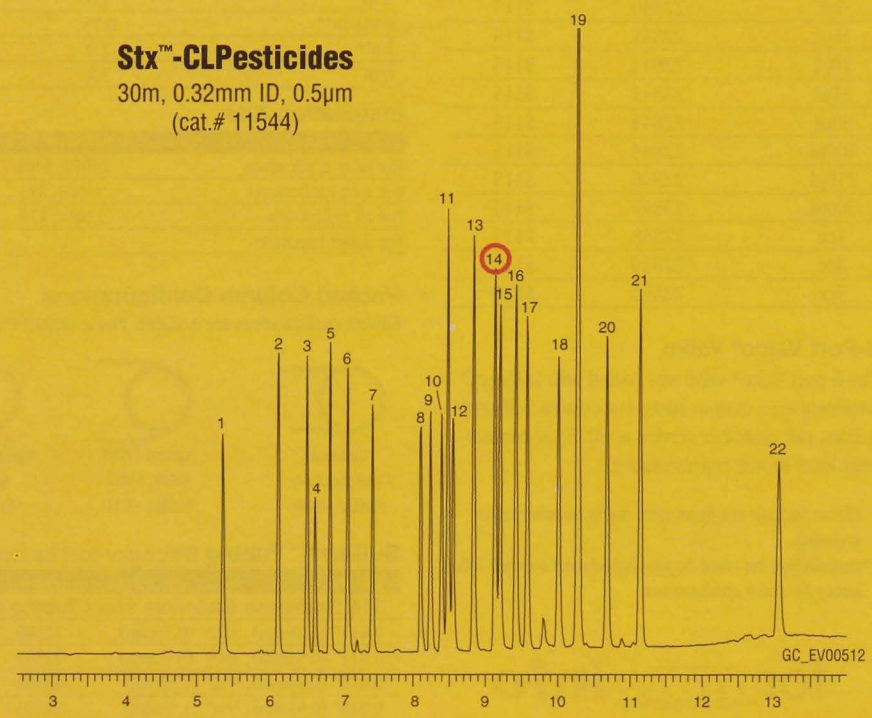
Combining Siltek™ deactivation with the unique selectivity of Restek CLPesticides phases results in the new Stx™-CLPesticides and Stx™-CLPesticides2 columns, and a significant improvement in endrin response compared to "Rtx" columns (Figure 1). Using the Stx™-CLPesticides and Stx™-CLPesticides2 columns, the endrin peak response is now higher than the analytes eluting in the same region—something not seen consistently in columns using traditional deactivations.

Column Installation and Optimizing Resolution

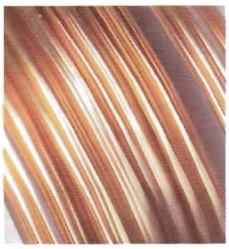
The Stx™-CLPesticides2 column is the ideal confirmational column to the Stx™-CLPesticides column. It was designed to achieve resolution of the chlorinated pesticides using the same flowrate and oven temperature program. The columns can be installed in parallel using a universal Siltek™ Press-Tight® "Y" connector or a metal MXT® "Y" connector. This parallel set-up reduces downtime caused by maintenance of multiple injection ports. Additionally, these columns can be installed in separate injection ports and mounted in the same GC oven.

Figure 1

Highly inert Stx™-CLPesticides and Stx™-CLPesticides2 columns provide excellent response for active pesticides such as endrin, DDT, and methoxychlor so you get accurate quantitation.



Oven temp.: 110°C (hold 1 min.) to 245°C @ 20°C/min. to 300°C @ 6°C/min.; **Inj. & det. temp.:** 210°C / 310°C; **Carrier gas:** helium; **Dead time:** 0.8min. @ 120°C; **Inlet liner:** Siltek™ Uniliner® w/hole (cat.# 21055-214.5); **Inj.:** 1µL direct injection of 20/40/200ng/mL std. concentration in hexane; **Make-up gas:** nitrogen



Stx™-CLPesticides Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter
0.25	0.25	-60 to 310/330°C	11540, $290	11543, $445
0.32	0.50	-60 to 310/330°C	11541, $310	11544, $475
0.53	0.50	-60 to 310/330°C	11542, $325	11545, $525

Stx™-CLPesticides2 Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter
0.25	0.20	-60 to 310/330°C	11440, $290	11443, $445
0.32	0.25	-60 to 310/330°C	11441, $310	11444, $475
0.53	0.42	-60 to 310/330°C	11442, $325	11445, $525

Siltek™ Inlet Liners

For Siltek™-deactivation, include the suffix number to the inlet liner catalog number.

Qty.	Siltek™	Siltek™ w/Siltek™ Wool	Siltek™ w/Carbofrit™
each	-214.1, $5 addl. cost	-213.1, $19 addl. cost	-216.1, $19 addl. cost
5-pk.	-214.5, $20 addl. cost	-213.5, $49 addl. cost	-216.5, $49 addl. cost
25-pk.	-214.25, $90 addl. cost	-213.25, $168 addl. cost	-216.25, $168 addl. cost

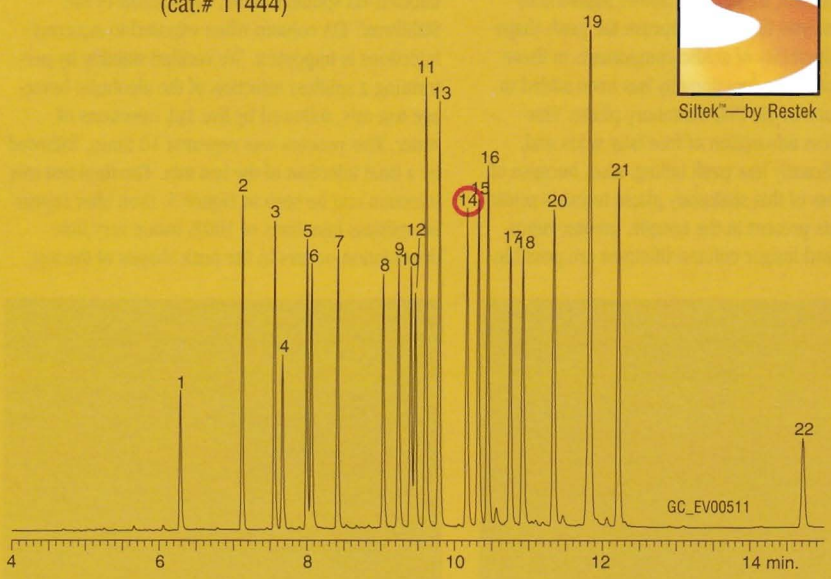
Siltek™ Guard Columns

ID	5-Meter (ea.)	10-Meter (ea.)
0.25mm	10026, $50	10036, $95
0.32mm	10027, $55	10037, $105
0.53mm	10028, $75	10038, $140

Stx™-CLPesticides2

30m, 0.32mm ID, 0.25µm
(cat.# 11444)

Siltek™—by Restek



1. 2,4,5,6 tetrachloro-*m*-xylene (IS)
2. α-BHC
3. γ-BHC
4. β-BHC
5. δ-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. 4,4' DDE
12. endosulfan I
13. dieldrin
14. **endrin**
15. 4,4' DDD
16. endosulfan II
17. 4,4' DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl (IS)

Injection Port Maintenance

Using Siltek™-treated Stx™-CLPesticides columns will improve your chlorinated pesticide analyses, but routine instrument maintenance will also help. The injection port is where a majority of analytical problems occur in the analysis of pesticides. The main problem is the cleanliness and inertness of the injection port with which the sample comes in contact. Endrin breakdown is usually indicative of a chemical reaction taking place in the injection port. The breakdown could be caused by impurities in the carrier gas, active metal surfaces, a non-deactivated inlet liner, or septa particles.

The carrier gas is usually the last troubleshooting area investigated and the hardest to eliminate. Endrin may react with a contaminant being carried into the injection port by the carrier gas. Having gas scrubbers in-line for the carrier gas will help keep this problem from occurring.

The metal surfaces of the injection port must be kept clean, including the inlet carrier gas line. Periodic rinsing of the carrier gas lines and cleaning the inside of the injection port may be necessary if endrin or 4,4'-DDT breakdown increases over short periods of time or when only analyzing standards. Rinsing of metal surfaces using solvents (e.g., methylene chloride, hexane or acetone), or in some cases silanizing the injection port, has helped. Also, Restek can coat your injection port with Sulfinert™ treatment for better inertness.

Improperly deactivated injection port liners are the primary cause of endrin breakdown. The best way to avoid this problem is to replace the liner with a Siltek™-deactivated liner when performing routine maintenance. Also, there is a standard procedure for deactivating liners that includes a process of cleaning the liners in acid and deactivating with dichlorodimethylsilane.

Septa particles are another cause of endrin breakdown. The septa particles will sit on top of a glass wool plug or at the bottom of the liner. To help eliminate septum coring, make sure your syringe needle does not have burrs. Another approach is to try different septa that features reduced coring, such as InfraRed™ septa (see pg. 14), and to change septa more often.

For more detailed information on chlorinated pesticide analysis, please request *A Guide to Preparing and Analyzing Chlorinated Pesticides* (lit. cat.# 59892).

GC Analysis of Acids, Esters, and Other Flavor Components in Distilled Liquor Products

Stabilwax®-DA GC Column—Part I

by Rebecca E. Wittrig, Ph.D., Food, Flavor, and Fragrance Innovation Team Leader; and Kevin MacNamara, Ph.D., Irish Distillers Ltd.*

- ✔ High thermal stability (250°C) and solvent rinsibility result in long column lifetime.
- ✔ Optimized configuration reduces analysis time by 4-fold.
- ✔ Low bleed for accurate quantitation.

Part I: The Separation

Distilled liquor products contain a wide range of volatile and non-volatile compounds in an ethanol/water matrix. The most abundant fusel alcohols and esters can be determined by gas chromatography (GC) with a simple split injection, which also minimizes the amount of matrix ethanol and water transferred to the column. However, many additional trace fatty acids and their esters—often used to indicate quality of alcoholic beverages such as whiskey and rum—cannot be determined by this approach. Because the concentrations can vary widely, splitless injection techniques with some type of preconcentration step usually are necessary to analyze fatty acids, esters, and other flavor compounds.

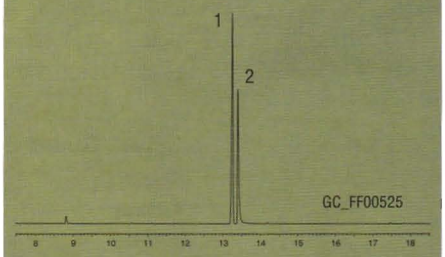
Large ranges of volatility and acidity for the component list make quantitating them in a single separation difficult to accomplish. The Stabilwax®-DA column, a bonded polyethylene glycol (PEG) phase, is excellent for analyzing alcoholic beverage samples (Figure 1). Using this column, flavor compounds in distilled liquor products can be quantitated in a single splitless injection. An optimized configuration of 30m, 0.18mm ID, 0.18µm allows significantly reduced analysis times. To improve the peak shape and reproducibility of acidic components in these samples, an acidic functionality has been added to the backbone of the PEG stationary phase. This results in less adsorption of free fatty acids and, thus, significantly less peak tailing. Also, because of the inertness of this stationary phase towards acidic components present in the sample, greater reproducibility and longer column lifetimes are possible.

Part II of this article will appear in the Winter '01/'02 Advantage. We will look at the Stabilwax®-DA column for alcoholic beverage analysis. Trace-level components, such as the ones displayed in Figure 1, often can be used to "fingerprint" a particular type or brand of distilled liquor. The application of this methodology to whiskey products will be discussed in Part II.

One of the critical pairs in the GC analysis of esters and acids in distilled liquor products is caproic acid and ethyl laurate. These components can be difficult to resolve on standard Carbowax®-type columns. This is especially true if peak tailing or broadening occurs, or if one component is present at a significantly higher concentration. The Stabilwax®-DA column achieves baseline resolution of these two compounds within a reasonable analysis time of 30 minutes (Figure 2).

Figure 2

The Stabilwax®-DA column achieves complete resolution of caproic acid and ethyl laurate in 30 minutes.



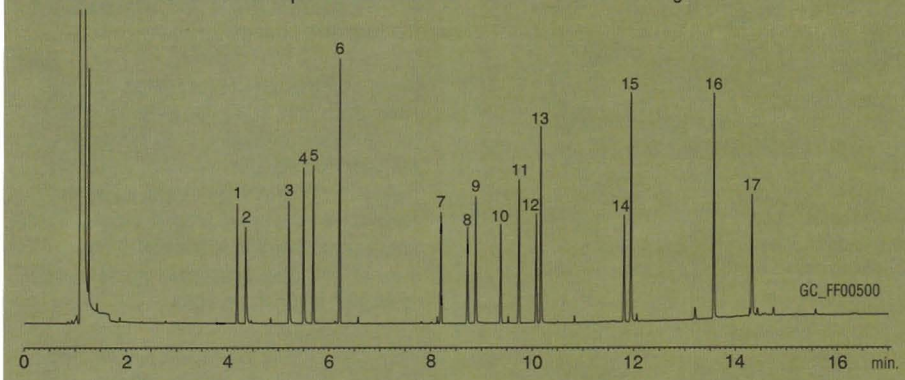
1. caproic acid 100 ppm
2. ethyl laurate 100 ppm

30m, 0.18mm ID, 0.18µm Stabilwax®-DA (cat.# 550752)
Oven temp.: 80 to 230°C at 5°C/min.;
See Figure 1 for complete conditions.

Because alcoholic beverage samples often are injected via splitless mode, the stability of the Stabilwax®-DA column when exposed to aqueous injections is important. We verified stability by performing a splitless injection of the alcoholic beverage test mix, followed by five 1µL injections of water. This process was repeated 10 times, followed by a final injection of the test mix. The final test mix injection can be seen in Figure 3. Even after repeated splitless injections of 100% water, very little degradation occurs in the peak shapes of the test

Figure 1

The Stabilwax®-DA column provides excellent peak shape of free fatty acids, esters, and other flavor compounds contained in an alcoholic beverage test mix.



30m, 0.18mm ID, 0.18µm Stabilwax®-DA (cat.# 550752)
Oven temp.: 70 to 240°C at 12°C/min. (hold 3 min.); **GC:** ThermoQuest Trace 2000; **Inj.:** 1µL splitless at conc. shown in peak list, in ethyl acetate; **Detector:** FID; **Inj. & det. temp.:** 240°C; **Liner:** 4mm ID splitless liner w/wool (cat.# 20814-202.1); **Hold time:** 0.5 min.; **Carrier gas:** hydrogen; **Make-up gas:** nitrogen; **Linear velocity:** 28psi @ 240°C

Peak List for Figures 1 & 3

	compound	conc. (ppm)
1.	ethyl octanoate	100
2.	acetic acid	100
3.	propionic acid	100
4.	isobutyric acid	100
5.	decanol 3	50
6.	ethyl decanoate	50
7.	ethyl laurate	50
8.	*cis*-lactone	100
9.	2-phenylethanol	50
10.	*trans*-lactone	100
11.	methyl myristate	50
12.	ethyl myristate	50
13.	octanoic acid	100
14.	ethyl palmitate	50
15.	decanoic acid	100
16.	dodecanoic acid	100
17.	vanillin	100

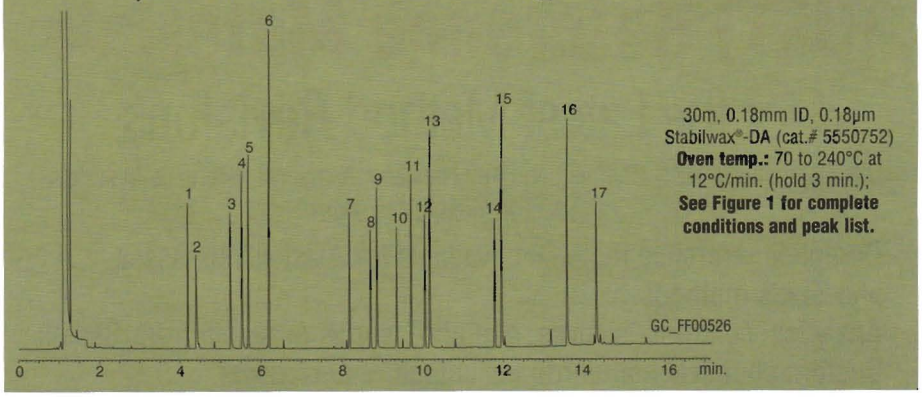
Irish Distillers Ltd., Bow Street Distillery, Smithfield, Dublin 7, Ireland

mix components. Over the course of the study, the variation in the peak retention times was 0.08-0.22% RSD. This includes the polar free fatty acids, which can be difficult to analyze under ideal conditions. The excellent stability of this stationary phase is proven by the reproducibility of the retention times over the course of the water stability study.

The Stabilwax®-DA column is an excellent choice for the analysis of acids, esters, and other flavor components in alcoholic beverage products. This highly stable column has been optimized for the analysis of acidic compounds, making it possible to analyze a wide range of compounds in a single injection. In addition, the column configuration shown in this article allows fast, efficient separation of the test compounds.

Figure 3

Excellent stability and peak shapes for free fatty acids after fifty 1µL splitless injections of water demonstrates column ruggedness and long lifetime.



Stabilwax®-DA Columns

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.18	0.18	40 to 250°C	—	550752, $610	—
0.25	0.10	40 to 250°C	11005, $250	11008, $390	11011, $690
	0.25	40 to 250°C	11020, $240	11023, $390	11026, $690
	0.50	40 to 250°C	11035, $240	11038, $390	11041, $690
0.32	0.10	40 to 250°C	11006, $260	11009, $425	11012, $750
	0.25	40 to 250°C	11021, $250	11024, $425	11027, $750
	0.50	40 to 250°C	11036, $250	11039, $425	11042, $750
	1.00	40 to 240/250°C	11051, $250	11054, $425	11057, $750
0.53	0.10	40 to 250°C	11007, $290	11010, $485	11013, $850
	0.25	40 to 250°C	11022, $280	11025, $485	11028, $825
	0.50	40 to 250°C	11037, $280	11040, $485	11043, $825
	1.00	40 to 240/250°C	11052, $280	11055, $485	11058, $825
	1.50	40 to 230/240°C	11062, $290	11065, $485	11068, $840

4mm Splitless Inlet Liner for ThermoQuest Trace GCs

4.0mm ID, 5.5mm OD, 79.5mm length
Add suffix -202.1 for wool packing.

each	5-pk.	25-pk.
20814, $20	20815, $80	20816, $360

for more info

request the Stabilwax®/MXT®-WAX Fast Facts flyer (lit. cat.# #59316).

Ethanol Analytical Reference Materials

For Blood Alcohol Testing

by Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Resolution control standard for calibration of whole analytical system.
- ✔ Custom mixes for 6-point calibration of your instrument.
- ✔ Confidence ensured with a Certificate of Analysis, raw materials test results, statistical QA results, analytical balance printout, and a lot sheet showing gravimetric weight of each analyte.

Ethanol Standards

All standards are available in a 5-ampul minimum purchase.

Expiration date for each solution is 36 months from the date of manufacture.

concentration	*cat.#*	*minimum purchase of 5 ampuls*
25mg/dL in water	550741	5 x 20mL ampuls, $45/ampul
40mg/dL in water	550742	5 x 20mL ampuls, $45/ampul
100mg/dL in water	550743	5 x 20mL ampuls, $45/ampul
150mg/dL in water	550744	5 x 20mL ampuls, $45/ampul
200mg/dL in water	550745	5 x 20mL ampuls, $45/ampul
300mg/dL in water	550746	5 x 20mL ampuls, $45/ampul

Resolution Control Standard

The ethanol concentration will be a certified value. The concentration of the other analytes will be reported as gravimetric concentration only.

Expiration date is 18 months from the date of manufacture.

Contains:

ethanol	methyl ethyl ketone
methanol	acetaldehyde
isopropanol	acetonitrile
acetone	ethyl acetate

concentration	*cat.#*
100mg/dL each compound in water	550778
minimum purchase of 10 ampuls	
10 x 1mL ampuls, $72/ampul	

Rtx®-BAC1 Columns

Temp. limits: -20 to 240/260°C

ID	df (µm)	30-Meter
0.32mm	1.80	18003, $462
0.53mm	3.00	18001, $490

Rtx®-BAC2 Columns

Temp. limits: -20 to 240/260°C

ID	df (µm)	30-Meter
0.32mm	1.20	18002, $462
0.53mm	2.00	18000, $490

for more info request the Application Note *Dual-Column Confirmational GC Analysis of Blood Alcohols Using the Rtx®-BAC1 and Rtx®-BAC2 Columns* (lit. cat.# #59598).

Underground Storage Tank (UST) Reference Materials

for the Latest Method Revisions

by Jingzhen Xu, R&D Chemist, and Ken Herwehe, Analytical Reference Materials Product Marketing Manager

- ✔ Complete reference materials available for Alaska, Tennessee, and Texas methods.*
- ✔ Analytical columns, sleeves, and accessories available from Restek.
- ✔ Custom chemical standards for unique requirements.
- ✔ Product listings for all states available soon.

*Products for 15 state methods listed in 2001 catalog. Call for information on other revisions and look for even more UST information in the next *Restek Advantage*.

State of Texas

Method and regulatory information is available from
Texas Natural Resources Conservation Commission
Petroleum Storage Tank Division
Mailing address:
MC: 133
P.O. Box 13087
Austin, TX 78711-3087
(*Street address:* 12100 Park 35 Circle, Austin, TX 78753)
Phone: (512) 239-2106
Fax: (512) 239-2177
UST: www.tnrcc.state.tx.us/permitting/r_e/pstta
LUST:
www.tnrcc.state.tx.us/permitting/remed/rpr/index.html

TNRCC 1005 for TPH (revision 3, 6-1-2001); Draft TNRCC 1006

Alternate Boiling Point/Carbon Number Distribution Marker Stock Standard

hexane (C6)
octane (C8)
decane (C10)
dodecane (C12)
hexadecane (C16)
heneicosane (C21)
octacosane (C28)
pentatriacontane (C35)
hexatriacontane (C36)
200µg/mL each in pentane
1mL per ampul

Ea.	5-pk.	10-pk.
31639 $28	31639-510 $126	—
	with data pack	
31639-500 $38	31639-520 $140	31739 $252

Need something special?

Let Restek's Analytical Reference Materials Team prepare a custom solution for you. We offer convenience, high quality, and quick turnaround without "custom" prices. Contact us at 800-356-1688, ext. 4, or contact your local Restek representative for more information.

State of Tennessee

Method and regulatory information is available from
Tennessee Dept. of Environment and Conservation
Division of Underground Storage Tanks
4th Floor, L & C Tower
401 Church Street
Nashville, TN 37243-1541
Phone: (615) 532-0945
Fax: (615) 532-0938
www.state.tn.us/environment/ust

TN GRO (v. 3-31-1999)

Gasoline Component Standard

Component	*Concentration, (ug/mL)*
2-methylpentane	1500
2,2,4-trimethylpentane	1500
heptane	500
benzene	500
toluene	1500
ethylbenzene	500
m-xylene	1000
p-xylene	1000
o-xylene	1000
1,2,4-trimethylbenzene	1000

10,000µg/mL total in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30486 $28	30486-510 $126	—
	with data pack	
30486-500 $38	30486-520 $140	30586 $252

New Stoddard Solvent Standards

Stoddard solvent is also known as Type I mineral spirits. Texsolve S®, or Varsol 1® mineral spirits. Restek now offers this type of mineral spirits for those who need to calibrate Stoddard solvent separately. This new standard is dissolved in methanol for analysis by either direct injection or purge and trap.
10,000µg/mL in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30487 $21	30487-510 $94.50	—
	with data pack	
30487-500 $31	30487-520 $105	30587 $189

State of Alaska

Method and regulatory information is available from
Alaska Department of Environmental Conservation
410 Willoughby Avenue
Juneau, AK 99801-1795
Phone: (907)465-5203
Fax: (907)465-5218
www.state.ak.us/dec/dspar/stp_home.htm

Alaska Method AK101 and AK101AA (v. 3-1-99)

Retention Time Marker

hexane decane dodecane
1,000µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30483 $25	30483-510 $112.50	—
	with data pack	
30483-500 $35	30483-520 $125	30583 $225

Surrogate Standard

1,4-bromofluorobenzene αααα-trifluorotoluene
2,500µg/mL each in P&T methanol
1mL per ampul

Ea.	5-pk.	10-pk.
30484 $25	30484-510 $112.50	—
	with data pack	
30484-500 $35	30484-520 $125	30584 $225

Certified Aromatics in Gasoline

An unleaded gasoline composite standard at 5,500µg/mL with certified concentration values for the following aromatics:

benzene
toluene
ethylbenzene
o-xylene
p-xylene
m-xylene
1,2,3-trimethylbenzene
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
isopropylbenzene
m-ethyltoluene
p-ethyltoluene
o-ethyltoluene
n-propylbenzene
methyl *tert*-butyl ether
naphthalene
1mL per ampul in P&T methanol

Ea.	5-pk.	10-pk.
30485 $60	30485-510 $270	—
	with data pack	
30485-500 $70	30485-520 $300	30585 $540

Alaska Methods AK 102, AK 103 (v. 3-1-99) and Methods AK102AA, AK 103AA (v. 6-30-98)

Retention Time Marker Standard

(Methods AK102/103/102AA/103AA)
decane C10 pentacosane C25
hexatriacontane C36
1,000µg/mL each in hexane
1mL per ampul

Ea.	5-pk.	10-pk.
31637 $25	31637-510 $112.50	—
	with data pack	
31637-500 $35	31637-520 $125	31737 $225

Surrogate Standard

squalane *o*-terphenyl
tetrahydronaphthol
1,000µg/mL each in methylene chloride
1mL per ampul

Ea.	5-pk.	10-pk.
31638 $25	31638-510 $112.50	—
	with data pack	
31638-500 $35	31638-520 $125	31738 $225

Natural and Refinery Gas Standards

by Gary Barone, GC Accessories, MPG, and Air Monitoring Products Marketing Manager

- ✔ Three different concentrations available to closely fit your method regulations.
- ✔ Mini-regulator designed specially for the standards.

Restek now offers standards for natural gas and refinery gas applications. Restek has developed many unique columns and sampling equipment for these industries and continues to expand product offerings for analysts working in these fields. These new standards are shipped in a DOT-4B-240ET cylinder that is 3" in diameter and 9 3/8" high. These cylinders use a CGA 170/110 connection. Restek also offers a mini-regulator specifically made for these standards.

Natural Gas	Natural Gas Standard #1* cat.# 34438, $295 ea. % of each compound	Natural Gas Standard #2* cat.# 34439, $295 ea. % of each compound	Natural Gas Standard #3* cat.# 34440, $295 ea. % of each compound
nitrogen	1.000	2.500	5.000
carbon dioxide	0.500	1.000	1.500
methane UHP	94.750	85.250	70.000
ethane UHP	2.000	5.000	9.000
propane	0.750	3.000	6.000
isobutene	0.300	1.000	3.000
n-butane	0.300	1.000	3.000
isopentane	0.150	0.500	1.000
n-pentane	0.150	0.500	1.000
hexanes plus EX2**	0.100	0.250	0.500
Concentration	mole	mole	mole
Volume	13.16L @ 200psig	13.16L @ 200psig	5.5L @ 75psig
Ideal Heating Value	1048 gross*	1142 gross*	1317 gross*

From rich to lean, each natural gas mix contains an extended list of C6+ compounds.

Refinery Gas	Refinery Gas Standard #1* cat.# 34441, $395 ea. % of each compound	Refinery Gas Standard #2* cat.# 34442, $395 ea. % of each compound	Refinery Gas Standard #3* cat.# 34443, $395 ea. % of each compound
hydrogen	40.650	12.500	12.500
argon	0.500	1.000	1.000
nitrogen	4.000	37.250	37.250
carbon monoxide	1.000	1.000	1.000
carbon dioxide	3.000	3.000	3.000
methane	8.500	5.000	5.000
ethane	6.000	4.000	4.000
ethylene	2.000	2.000	2.000
acetylene	-	1.000	1.000
propane	7.000	6.000	6.000
propylene	3.000	3.000	3.000
propadiene	0.850	1.000	1.000
isobutane	6.000	5.000	5.000
n-butane	4.000	4.000	4.000
isobutylene	2.000	1.000	1.000
1,3 butadiene	3.000	3.000	3.000
cis-2-butene	2.000	2.000	2.000
trans-2-butene	2.000	3.000	3.000
butene-1	2.000	2.000	2.000
2-methyl-2-butene	-	0.200	0.200
isopentane	1.000	1.000	1.000
n-pentane	1.000	1.000	1.000
cis-2-pentene	-	0.400	0.400
trans-2-pentene	-	0.150	0.150
pentene-1	-	0.400	0.400
n-hexane	0.500	0.100	0.100
Concentration	mole	mole	mole
Volume	5.2L @ 70psig	4.9L @ 60psig	4.6L @ 60psig

Each refinery gas mix contains varying degrees of C5 unsaturates or extended C6+ compounds.

**Dry BTU/SCF @ 14.696psia & 60°F.* ***Contact Restek to get a complete list of hexanes plus EX2.*

CGA 170 Mini-Regulator

- ✔ For natural gas and refinery gas standards.
- ✔ Inlet pressure range: 0-300psig; outlet pressure range: 0-15psig.
- ✔ Supplied with 0-15psig outlet pressure gauge, brass CGA 170 nut, and nipple.

cat.# 22032, (ea.) $125

Gas-Tight Syringes

- ✔ Teflon®-tipped plungers.
- ✔ Removable needles.
- ✔ Replaceable syringe barrels, plungers, and plunger tips.

Hamilton Syringes

Volume (µL)	Hamilton Model	Hamilton cat.#	Restek cat.#
10	1701	80065	21260, $36
25	1702	80265	21261, $42
50	1705	80965	21262, $44
100	1710	81065	21263, $46
250	1725	81165	21264, $50

SGE Syringes

Volume (µL)	SGE Model	SGE cat.#	Restek cat.#
10	10R-GT-LC	002313	24866, $36
25	25R-GT-LC	003312	24867, $36
50	50R-GT-LC	004312	24868, $37
100	100R-GT-LC	005312	24869, $42
250	250R-GT-LC	006312	24870, $42
500	500R-GT-LC	007312	24871, $42

InfraRed™ Septa

New! High-Performance, Low Bleed

by Gary Barone, GC Accessories Product Marketing Manager

- ✔ Incredible high-quality.
- ✔ Stable to 325°C.
- ✔ Low bleed.
- ✔ Excellent puncturability.
- ✔ Do not adhere to injectors.

Restek introduces the new InfraRed™ septa. These septa are formulated with silicone and filler that maximize thermal stability to 325°C, and provide smooth puncturability and very low bleed. Experience low bleed and long lifetimes using these new InfraRed™ septa.

handy Septum Size Chart

Instrument	Septum Size
Agilent (HP)	
5880A, 5890, 6890,6850	11mm
5700, 5880	9.5/10mm
On-Column Injection	5mm
CE Instruments (TMQ)	
TRACE GC	17mm
Finnigan (TMQ)	
GC 9001	9.5mm
GCQ	9.5mm
GCQ w/TRACE	17mm
QCQ™	9.5mm
TRACE 2000	9.5mm
Fisons/Carlo Erba (TMQ)	
8000 series	17mm
Gow-Mac	
6890 series	11mm
All other models	9.5mm
Perkin-Elmer	
Sigma series	11mm
900,990	11mm
8000 series	11mm
Auto SYS	11mm
Pye/Unicam	
All models	7mm
Shimadzu	
All models	Plug
SRI	
All models	Plug
Tracor	
540	11.5mm
550.560	9 5mm
220,222	12.5mm
Varian	
Injector type.	
Packed column	9.5/10mm
Split/splitless 1078/1079	10/11mm
1177	9mm

Measure your old septum here (size in mm)

5 · 7 · 9 · 9.5 · 10 · 11 · 11.5 · 12.5 · 17

InfraRed Septa

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417 $40	21418 $60	21419 $100
9.5mm (3/8")	21421 $40	21422 $60	21423 $100
10mm	21424 $40	21425 $60	21426 $100
11mm (7/16")	21427 $40	21428 $60	21429 $100
11.5mm	21430 $40	21431 $60	21432 $100
12.5mm (1/2")	21433 $40	21434 $60	21435 $100
17mm	21436 $43	21437 $80	21438 $155
Shimadzu Plug	21439 $40	21440 $60	21441 $100

IceBlue™ Septa

- ✔ General purpose septa.
- ✔ Excellent puncturability.
- ✔ Preconditioned and ready to use.
- ✔ Do not adhere to hot metal surfaces.
- ✔ Usable to 250°C inlet temperatures.
- ✔ Ideal for SPME.

Septum Diameter	50-pk.	100-pk.
9mm	22381 $35	22382 $65
9.5mm (3/8")	22388 $35	22389 $65
10mm	22390 $35	22391 $65
11mm (7/16")	22392 $35	22393 $65
11.5mm	22383 $35	22384 $65
12.5mm (1/2")	22394 $35	22395 $65
17mm	22396 $35	22397 $65
Shimadzu plug	22398 $35	22399 $65

Thermolite® Septa

- ✔ Usable to 340°C inlet temperatures.
- ✔ Each batch tested on FIDs, ECDs, & MSDs to ensure low bleed.
- ✔ Excellent puncturability.
- ✔ Preconditioned and ready to use.
- ✔ Packaged in non-contaminating glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351 $40	20352 $60	20353 $100
6mm (1/4")	20355 $40	20356 $60	20357 $100
7mm	20381 $40	20382 $60	20383 $100
8mm	20370 $40	20371 $60	— —
9mm	20354 $40	20358 $60	20362 $100
9.5mm (3/8")	20359 $40	20360 $60	20361 $100
10mm	20378 $40	20379 $60	20380 $100
11mm (7/16")	20363 $40	20364 $60	20365 $100
11.5mm	22385 $40	22386 $60	22387 $100
12.5mm (1/2")	20367 $40	20368 $60	20369 $100
17mm	20384 $43	20385 $80	20386 $155
Shimadzu Plug	20372 $40	20373 $60	20374 $100

Hot Tech Tip

What is Septum Bleed?

Septum bleed occurs when volatile compounds are off-gassed from the septum. During a temperature-programmed analysis, you can see septum bleed in baseline rise and/or extraneous peaks not associated with the sample or the column. Septum bleed is most noticeable during temperature-programmed analyses because the volatiles off-gassed from the septum collect on the head of the analytical column during the cool-down period and initial hold time. Then, these volatiles elute during subsequent runs. Under isothermal conditions, septum bleed is a continuous, steady interference that appears as part of the normal background noise. Either way, septum bleed interferes with quantitation and analysis accuracy. Septum bleed can be affected by inlet temperature, initial hold time, compression, injection mode, etc., but, the single most important variable is the septum quality. To maintain analytical integrity, use high-quality septa for senstitive or high-temperature analyses.

cool tools

Tired of burned fingers, leaking injection ports, and lost scoring wafers? Try these new tools from Restek.

by Brad Rightnour and Michael Goss, Instrument Innovations Team

special OFFER!

Buy a 25-pk. of inlet liners and receive a *free inlet liner removal tool!*
Offer good through 1/31/02.

GC Inlet Liner Removal Tool

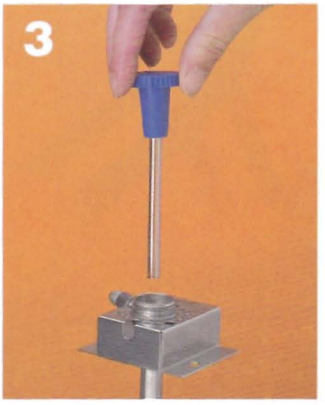
No more burnt fingers!

- ✔ Easily removes liners from injectors.
- ✔ Made from high-temperature silicone.
- ✔ Won't crack or chip the liner

cat.# 20181, (3-pk.) $25

Gently push the liner removal tool onto the liner in the injection port with a slight circular motion. This will ensure that the removal tool has a firm grip on the liner.

Slowly pull the liner out of the injection port in a straight vertical motion.



Use the liner removal tool to place a new liner into the injection port, carefully avoiding hot metal surfaces.

Injection Port Repair Tool

Remove contaminants, achieve a better seal!

- ✔ For Agilent split/splitless injection ports.
- ✔ Resurfaces critical inlet seal areas.
- ✔ Removes contaminants.

cat.# 21393, (ea.) $84

The inlet seal at the base of a split/splitless injector allows a seal to form between the injection port and the inlet liner. This inlet seal wears over time and may become scratched or pitted, which compromises the sealing ability of the injector. The new Restek injection port repair tool allows the user to easily resurface the inlet seal and remove contaminants; and it saves time and money by preventing unwanted leaks.

Try Restek's SILCOSTEEL Injection Ports

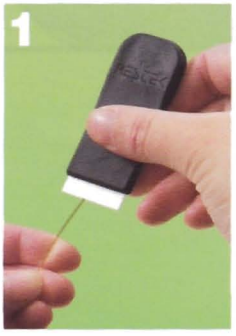
For more information, request the catalog *Genuine Restek Replacement Parts for Agilent GCs* (lit. cat.# 59627B).

Scoring Wafer with Handle

Same great scoring, better comfort!

- ✔ Unique, ergonomic handle is made of soft, comfortable plastic.
- ✔ Ceramic wafer is serrated on one side and straight-edged on the other.
- ✔ Cuts both fused silica and metal tubing cleanly.

cat.# 23015, (2-pk.) $27



Hold tubing firmly in one hand, allowing about two inches to extend freely for safe cutting. Hold the scoring wafer at a 45° angle to the tubing Exert slight pressure—just enough to put a slight arc in the tubing. Pull perpendicularly across the tubing. Move your whole arm, not just your hand. This will help ensure a square, consistent score.

If the tubing doesn't fall off on its own, it should easily break at the score with a slight tap of the wafer. If it doesn't, score again. Check the cut against the white of the scoring wafer for a clean, square cut.

RESTEK Behind the Scenes

Use your Wizard Dollars to help!

We all want to do what we can to help with the expenses incurred from the September 11th terror attack. From now until December 31st, 2001, we will donate one US$ for each Wizard$ to the fund of your choice.

If you choose to do so, you can donate the Wizard Dollars from your current order simply by specifying which organization should receive them. Also, you can donate accumulated Wizard Dollars by mailing them to Restek with a Wizard Dollar order form noting one of the listed charities.

Restek Corporation
Attn: Customer Service Wizard $
110 Benner Circle
Bellefonte, PA, 16823

One way we can help is by donating Restek Wizard Dollars to these charities:

- ★ United Way
- ★ Red Cross
- ★ Salvation Army

New Online Tool for GC Optimization

Our Restek webmaster has designed an interactive, multi-purpose tool that gives you the optimization data specific to your column configuration, carrier gas, and detector. Handy when installing or troubleshooting, and indispensable for obtaining the best analytical performance; with reference data, calculators, and procedural information. Find it on our homepage:

www.restekcorp.com

Restek Salutes Those in the Military

We salute all individuals involved in military and rescue efforts both at home and abroad. We especially thank the following members of the Restek family:

Roger Greene, Airforce 913th Security Force Squadron (Restek Personal Trainer)
Ken Herwehe, Army Reserves (Restek Analytical Reference Materials Product Marketing Manager)
Alvira Peak, Civilian Air Corps (Restek Environmental Health and Safety Coordinator)
Matt Reilly, Army National Guard (Restek Applied Technology Manufacturing Technician)
Pete Zucco, Naval Reserves (Restek Maintenance Technician)

New Restek Wizards

Craig Hall, Customer Service Representative
Debra Copenhaver, Customer Service Representative
Diane Thompson, Analytical Reference Materials Shipping Technician
James Weber, Maintenance Assistant
Courtney Johnson, Personal Trainer

ISO 9001

Lit. Cat.# 59426

Please direct your comments on this publication to Kristin Dick, Editor, at kristind@restekcorp.com or call Restek, ext. 2313.

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