Our expertise, experience, and enthusiasm is **your** Advantage.

RESTEK ADVANTAGE

Introducing…

Rxi®-17Sil MS Columns

Offering Unique Selectivity for PAHs

- **Separate PAHs** that cannot be distinguished by mass spectrometry.
- **Increase accuracy for PAHs** of regulatory and health concern.
- **Rxi® technology** assures reliable trace level results.

For more information on PAH analyses using **Rxi-17Sil MS** columns, see the **Food Safety** feature articles on pages 2 & 3.

Also in this issue

www.restek.com

New! Rxi®-17Sil MS Column

Separate PAHs that Cannot be Distinguished by Mass Spectrometry

- Unique phase chemistry provides better resolution than other "17" type columns.
- Optimized selectivity separates a wide range of key PAHs.
- Rxi® technology assures accurate, reliable trace level analyses.

Rxi® GCColumns

Polycyclic aromatic hydrocarbon (PAH) analysis is a growing area of environmental and food safety testing, due to the ubiquitous presence and reported genotoxicity and carcinogenicity of some compounds in this class. As target lists expand and health concerns drive detection levels lower, reporting requirements are more difficult to meet and column selectivity becomes an important factor in achieving accurate results.

New Rxi®-17Sil MS columns are optimized for PAHs and are the best choice for accurate, trace level detection. Rxi®-17Sil MS columns differ in phase chemistry from conventional 17 type (50% diphenyl) columns, and the resulting selectivity provides better resolution of critical PAHs (Figure 1). Not all 50% phenyl columns are equivalent—Rxi®-17Sil MS columns let you quantify isobaric PAHs that cannot be determined by mass spectrometry.

Unique Selectivity Means More Accurate PAH Data

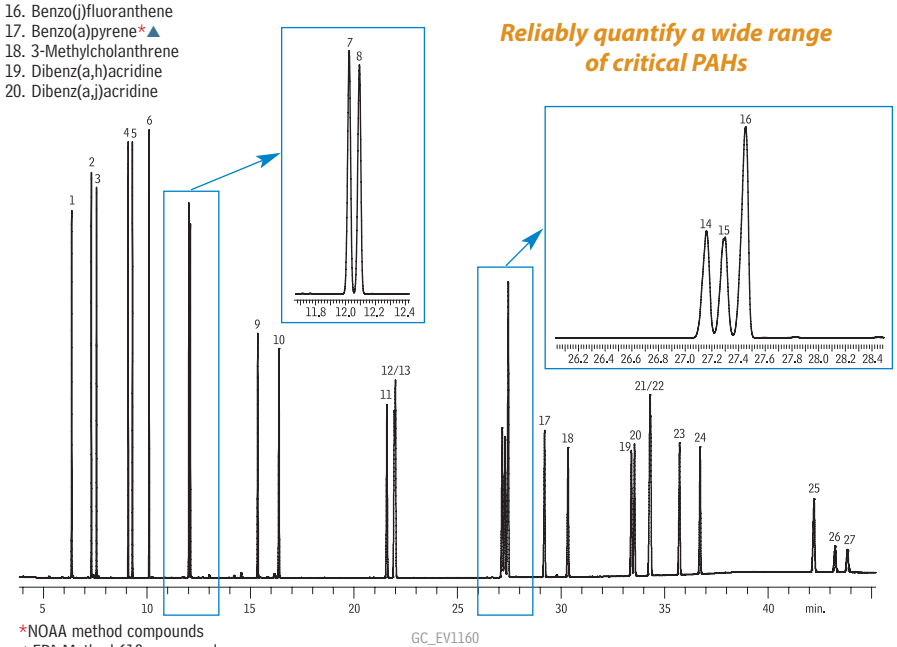
Little differences mean a lot. At first glance, PAH separations on the new Rxi®-17Sil MS and typical "17" type columns appear to be similar, but the difference in selectivity becomes clear when looking at critical separations (Figure 2). Isobaric compounds phenanthrene and anthracene have essentially indistinguishable mass spectra and must be chromatographically resolved. The Rxi®-17Sil MS column provides baseline resolution of these critical com-

Figure 1 Rxi®-17Sil MS columns ensure excellent resolution of PAHs of regulatory or health concern.

1. Naphthalene*▲
2. 2-Methylnaphthalene
3. 1-Methylnaphthalene
4. Acenaphthylene▲
5. Acenaphthene▲
6. Fluorene*▲
7. Phenanthrene*▲
8. Anthracene*▲
9. Fluoranthene*▲
10. Pyrene*▲
11. Benz(a)anthracene*▲
12. Chrysene*▲
13. Triphenylene
14. Benzo(b)fluoranthene*▲
15. Benzo(k)fluoranthene*▲
16. Benzo(j)fluoranthene
17. Benzo(a)pyrene*▲
18. 3-Methylcholanthrene
19. Dibenz(a,h)acridine
20. Dibenz(a,j)acridine
21. Indeno(1,2,3-cd)pyrene*▲
22. Dibenz(a,h)anthracene*▲
23. Benzo(ghi)perylene▲
24. 7H-Dibenzo(c,g)carbazole
25. Dibenzo(a,e)pyrene
26. Dibenzo(a,i)pyrene
27. Dibenzo(a,h)pyrene

Column: Rxi®-17Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 14123)
Sample: SV Calibration Mix #5 / 610 PAH Mix (cat.# 31011); EPA Method 8310 PAH Mixture (cat.# 31841);
Diluent: dichloromethane; Conc.: 10 ppm
Injection: Inj. Vol.: 0.5 µL splitless (hold 1.75 min.)
Liner: Auto SYS XL PSS Split/Splitless w/Wool (cat.# 21718)
Inj. Temp.: 320 °C; Purge Flow: 75 mL/min.
Oven: Oven Temp: 65 °C (hold 0.5 min.) to 220 °C at 15 °C/min. to 330 °C at 4 °C/min. (hold 15 min.)
Carrier Gas: He, constant flow; Flow Rate: 2.0 mL/min.
Detector:FID @ 320 °C
Instrument:PE Clarus 600 GC
Acknowledgement: Instrument provided by PerkinElmer

Reliably quantify a wide range of critical PAHs



*NOAA method compounds
▲EPA Method 610 compounds

pounds, which are only partially separated with a typical 17 type column. Similarly, benzofluoranthenes b, k, and j are isobaric compounds that must be reported separately, and the Rxi®-17Sil MS column reliably resolves all 3 isomers, even in a 15 m length. The unique selectivity of the Rxi®-17Sil MS column gives you more resolving power and better accuracy for challenging PAHs.

For more information on new Rxi®-17Sil MS columns, visit **www.restek.com/adv010**

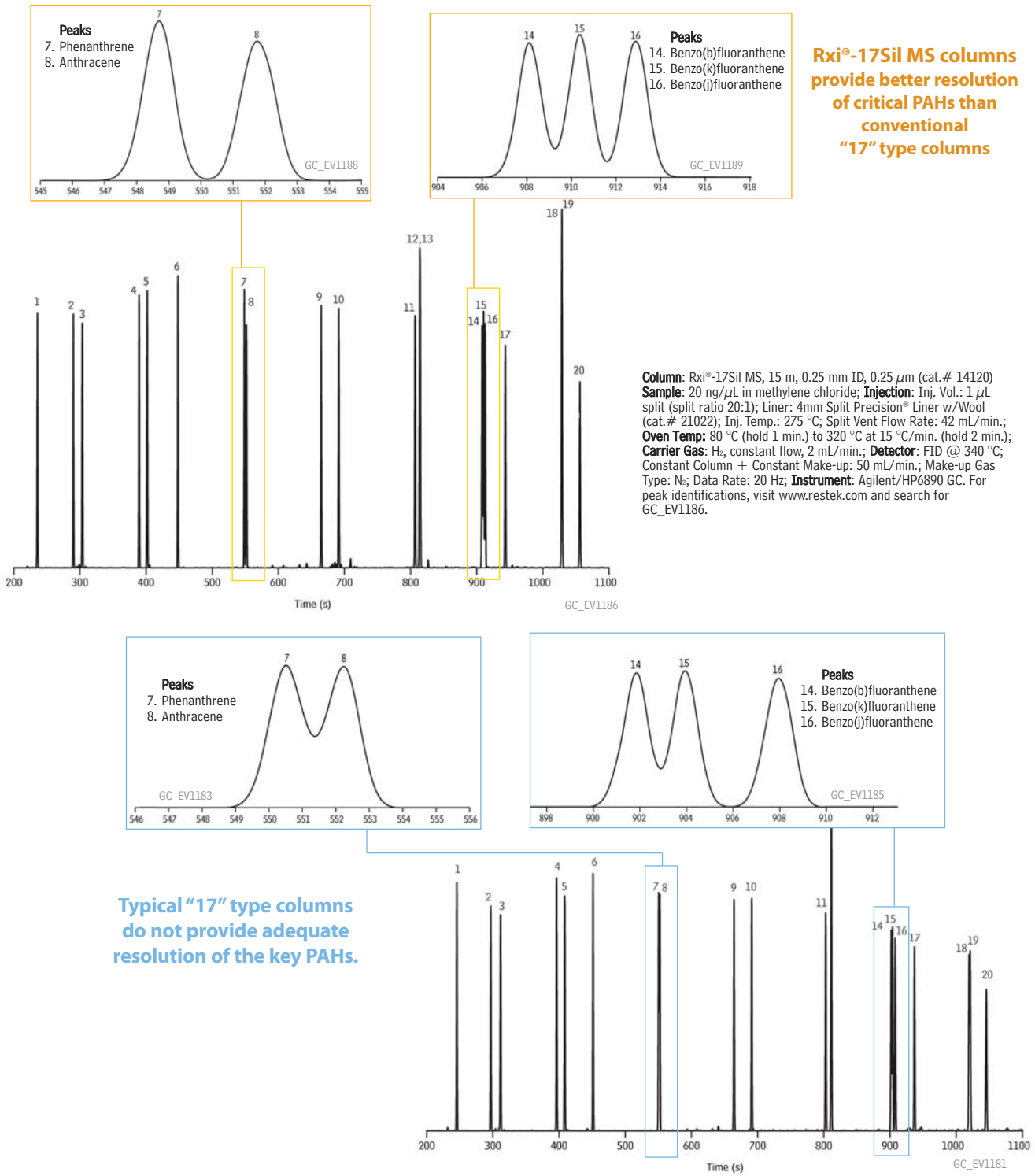
Rxi®-17Sil MS Columns (fused silica)

(mid polarity Crossbond® silarylene phase; equivalent to 50% phenyl methyl polysiloxane)

ID	df (µm)	temp. limits	length	qty.	cat. #
0.18mm	0.18µm	40 to 340/360°C	20m	ea.	14102
0.18mm	0.36µm	40 to 340/360°C	20m	ea.	14111
0.25mm	0.25µm	40 to 340/360°C	15m	ea.	14120
0.25mm	0.25µm	40 to 340/360°C	30m	ea.	14123
0.25mm	0.25µm	40 to 340/360°C	60m	ea.	14126
0.32mm	0.25µm	40 to 340/360°C	15m	ea.	14121
0.32mm	0.25µm	40 to 340/360°C	30m	ea.	14124

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Figure 2 Rxi®-17Sil MS columns let you accurately report PAHs that cannot be distinguished by MS.



Novel Approach for PAHs in Seafood:
Reduce Sample Prep from Days to Hours Using QuEChERS and GCxGC

By Jack Cochran, Director of New Business and Technology

- Prepare samples in hours vs. days, using QuEChERS instead of the NOAA method.
- GCxGC analysis minimizes matrix interference, for accurate trace-level results.
- Selectivity of Rxi®-17Sil MS column assures separation of benzofluoranthenes.

Consumer safety concern in the wake of the Deepwater Horizon oil spill has increased demand for rapid, accurate test methods for polycyclic aromatic hydrocarbons (PAHs) in seafood. The FDA has issued a protocol to reopen closed fishing waters that includes chemical testing of seafood for PAHs, but the NOAA sample preparation method that was proposed is extremely tedious and time-consuming, requires expensive pressurized fluid extraction and gel permeation chromatography equipment, and uses large volumes of environmentally-unfriendly methylene chloride. Alternative methods are being explored, and initial results for a novel approach that combines a rapid QuEChERS extraction with the accuracy of GCxGC-TOFMS are presented here.

QuEChERS Saves Time and Money

While QuEChERS was originally developed to simplify extraction and cleanup of pesticide residues in fruits and vegetables, it is rapidly expanding to other applications due to its speed, simplicity, and cost-effectiveness, so it was natural to consider it as a replacement for the NOAA method. For this work, samples of freeze-dried mussel tissue containing NIST certified levels of PAHs were prepared in less than 2 hours using a simple procedure that was quicker, easier, and more cost-effective than the NOAA method (Figure 1).

GCxGC with Rxi®-17Sil MS and Rxi®-1ms Columns Ensures Unbiased Separation of Key PAHs

Mussel samples were too complex for traditional GC/MS analysis, so GCxGC was employed. The key to maximizing separations between peaks with this technique is to choose columns that differ significantly in phase chemistry. An Rxi®-17Sil MS column was chosen for the first separation, as it is optimized for PAH separations (see article on p. 2), and a standard Rxi®-1ms column was used for the second dimension to separate interfering fatty acids and sterols from the PAHs of interest.

Figure 1 QuEChERS extraction and cleanup procedure.

Note: Dried sample was used here; for fresh samples, start at Step 3.

1. Weigh 1.0 g of NIST SRM 2974a tissue into a 50 mL FEP centrifuge tube and add 10 mL organic-free water. Shake 1 min. to wet sample.
2. Aggressively vortex the sample for 15 min., then allow sample to settle for 30 min.
3. Add 10 mL acetonitrile and 20 µL of 25 ng/µL Semivolatiles Internal Standard Mix (diluted from cat.# 31206).
4. Shake sample by hand for 1 min., then vortex for 15 min.
5. Add 1 packet of Q110 EN 15662 QuEChERS extraction salts (cat.# 26236) and shake hard.
6. Aggressively vortex the sample for 15 min., then centrifuge at 3,000 g for 5 min.
7. Transfer 1mL supernatant extract to a 10 mL FEP tube with 150 mg $MgSO_4$ and 50 mg PSA, and 50 mg C18 and shake 1 min. to remove some fatty acids and lipids.
8. Centrifuge at 3,000 g for 5 min.
9. Withdraw extract for GCxGC-TOFMS analysis of PAHs.

Example time savings:
Estimated processing time for 30 fresh samples

QuEChERS: 10 hours
NOAA: 3-5 days

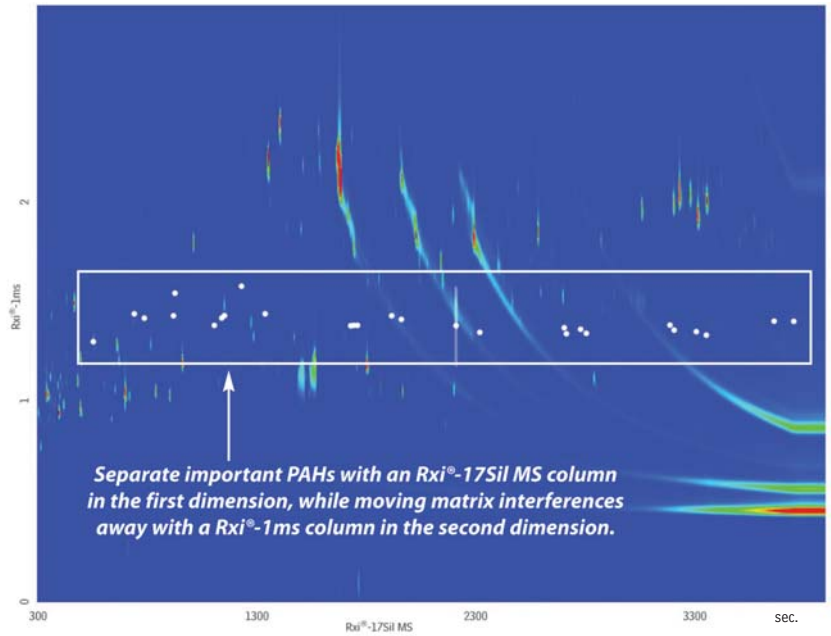
Results in Table I demonstrate that good recoveries were obtained under these preliminary conditions, especially considering that hydrophobic compounds were being extracted in hydrophilic QuEChERS solvent. High recoveries were noted for some compounds (e.g. fluoranthene), but were not due to isobaric interference as evidenced by high efficiency separation of PAHs from matrix (Figure 2) and by good agreement between sample and reference spectra (Figure 3).

Conclusion

Combining QuEChERS extraction with GCxGC-TOFMS, using Rxi®-17Sil MS and Rxi®-1ms columns shows great promise for analyzing PAHs in seafood. Labs interested in alternatives to the NOAA method should consider procedures based on this approach.

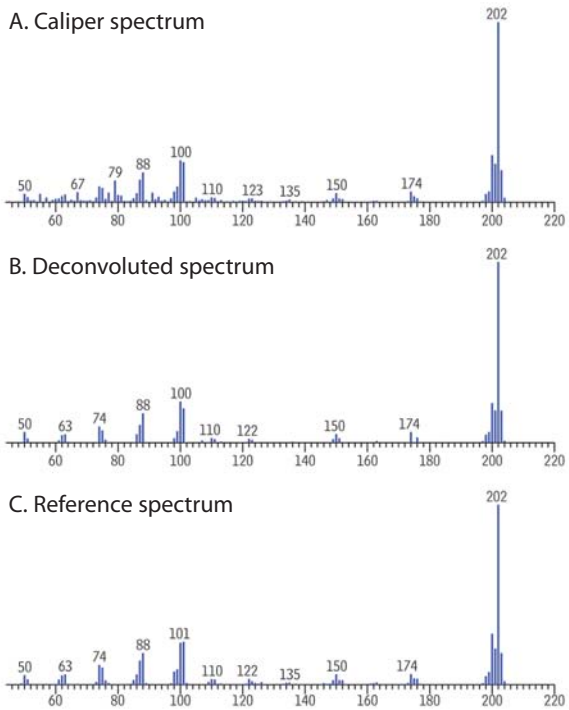
Visit our technical blog at **www.restek.com/adv011** for more details.

Figure 2 GCxGC-TOFMS contour plot of PAHs in mussels (QuEChERS extraction).



Column: Rxi®-17Sil MS 30 m, 0.25 mm ID, 0.25 µm (cat.# 14123); Rxi®-1ms 1.2 m, 0.15 mm ID, 0.15 µm (cat.# custom);
Sample: NIST SRM 2974a freeze-dried; mussel tissue with incurred residues; Diluent: Acetonitrile.
For complete conditions visit www.restek.com and search for GC_FF1197.

Figure 3 Good agreement between sample and reference spectra show target PAHs were separated from isobaric interferences.



Column:
Rxi®-17Sil MS 30 m, 0.25 mm ID, 0.25 µm (cat.# 14123);
Rxi®-1ms 1.2 m, 0.15 mm ID, 0.15 µm (cat.# custom);
Sample: NIST SRM 2974a freeze-dried mussel tissue with incurred residues;
Diluent: Acetonitrile.

For complete conditions visit www.restek.com and search for GC_FF1198.

Table I Preliminary conditions gave good recoveries for most PAHs (n = 3).

PAH	Q Mass	NIST µg/kg	Average µg/kg by GCxGC	RSD%
Naphthalene-D8	136	ISTD	ISTD	ISTD
Naphthalene	128	9.68	63	5
2-Methylnaphthalene	142	8.1	8.6	15
1-Methylnaphthalene	142	5.8	5.4	8
Biphenyl	154	NA	4.2	1
2,6-Dimethylnaphthalene	156	NA	9.1	3
Acenaphthylene	152	NA	1.7	18
Acenaphthene-D10	162	ISTD	ISTD	ISTD
Acenaphthene	154	NA	3.3	22
2,3,5-Trimethylnaphthalene	170	NA	4.0	13
Fluorene	166	NA	8.8	8
Phenanthrene-D10	188	ISTD	ISTD	ISTD
Phenanthrene	178	74.4	113	5
Anthracene	178	2.46	8.1	8
1-Methylphenanthrene	192	17.6	29	12
Fluoranthene	202	287	376	5
Pyrene	202	186	229	4
Benzo(a)anthracene	228	31.1	39	9
Chrysene-D12	240	ISTD	ISTD	ISTD
Chrysene	228	123.6	199	5
Benzo(b)fluoranthene	252	41.5	53	0
Benzo(k)fluoranthene	252	18.95	22	12
Benzo(j)fluoranthene	252	21.4	18	3
Benzo(a)pyrene	252	9.73	12	5
Perylene-D12	264	ISTD	ISTD	ISTD
Perylene	252	6.80	5.0	3
Indeno(1,2,3-cd)pyrene	276	14.9	13	1
Benzo(ghi)perylene	276	23.7	20	10

ISTD = internal standard
NA = not analyzed by NIST

Restek Innovation!

Cutting-Edge Products for Food Safety Applications

26235

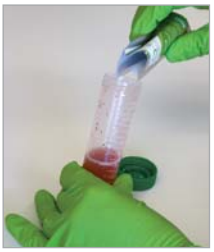
26239

26236

23997

Q-sep™ QuEChERS Extraction Salts

- Salt packets eliminate the need for a second empty tube to transfer salts.
- Go green by using packets with reusable tubes.
- Convenient and easy to use.



Description	Material	Methods	qty.	cat.#
Q110 Kit	4g $MgSO_4$, 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	European EN 15662	50 packets & 50 tubes	26235
Q110 Packets	4g $MgSO_4$, 1g NaCl, 1g TSCD, 0.5g DHS	European EN 15662	50 packets	26236
Q150 Kit	6g $MgSO_4$, 1.5g NaOAc with 50mL Centrifuge Tube	AOAC 2007.01	50 packets & 50 tubes	26237
Q150 Packets	6g $MgSO_4$, 1.5g NaOAc	AOAC 2007.01	50 packets	26238
Empty 50mL Centrifuge Tube, Polypropylene			50-pk.	26239
Empty 50mL Centrifuge Tube, Teflon FEP			2-pk.	23997

TSCD—trisodium citrate dihydrate
DHS—disodium hydrogen citrate sesquihydrate
NaOAc—sodium acetate

NEW! Q-sep™

Dimensions: 9"h x 14.5"w x 17"d
(22.9 cm x 36.8 cm x 43.2 cm)

26249

Q-sep™ 3000 Centrifuge

for QuEChERS

- Meets requirements of AOAC and European QuEChERS methodology.
- Supports 50 mL, 15 mL, and 2 mL centrifuge tubes.
- Small footprint requires less bench space.
- Safe and reliable—UL, CSA, and CE approved, 1-year warranty.

Priced to fit your laboratory's budget, the Q-sep™ 3000 Centrifuge is the first centrifuge specifically designed for QuEChERS methodology. This compact, quiet, yet powerful, unit spins at the 3,000 g force required by the European method.

Centrifuge includes 50 mL tube carriers (6), 50 mL conical tube inserts (6), 4-place 15 mL tube carriers (6), and 2 mL tube adaptors (24).

26232

26233

26234

Description	qty.	cat.#
Q-sep 3000 Centrifuge, 110V	ea.	26230
Q-sep 3000 Centrifuge, 220V	ea.	26231
Replacement Accessories		
50mL Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26232
50mL Conical Tube Insert for Q-sep 3000 Centrifuge	6-pk.	26249
4-Place Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26233
2mL Tube Adaptors for Q-sep 3000 Centrifuge	4-pk.	26234

Flip Seal Dual Vespel® Ring Inlet Seals

A **reversible** Dual Vespel® Ring Inlet Seal that lasts twice as long, for the same great price!

Our new Flip Seal greatly improves injection port performance while saving you time and money. This reversible inlet seal allows twice as many uses as other inlet seals, at the same cost. By using our patented Dual Vespel® Ring technology, the Flip Seal features two soft Vespel® rings, one on the top and one on the bottom, which eliminate the need for a washer. Our new reversible design allows you to flip the inlet seal and use it twice as many times.

1/16" Adaptor Fitting

Feature	Benefit
Reversible design.	Allows twice as many uses as other seals, at the same price.
Vespel® ring embedded in bottom surface.	Eliminates need for a washer.
Vespel® ring embedded in top surface.	Very little torque required to make seal—reduces operator variability.
Lower leak rate compared to OEM metal inlet seals.	Less detector noise.
Prevents oxygen from permeating the carrier gas.	Increases column lifetime.
Gold or Siltek Treated seals.	Reduces breakdown and adsorption of compounds, maximizing component transfer to GC column.
Kit includes 1/16-inch split/splitless adaptor fitting.	Works with standard OEM capillary ferrules.

0.8mm ID Flip Seal Dual Vespel Ring Inlet Seal	2-pk.	10-pk.
Gold-Plated	23407	23409
Siltek Treated	23408	23410
1.2mm ID Flip Seal Dual Vespel Ring Inlet Seal	**2-pk.**	**10-pk.**
Gold-Plated	23411	23413
Siltek Treated	23412	23414

Flip Seal Dual Vespel Ring Inlet Seal Kit	qty.	cat.#
Includes: gold-plated 0.8mm ID inlet seal, reducing nut adaptor, 1/16" SS nut	kit	23406

Fully Resolve Critical PAHs with an Optimized HPLC Column

Although most HPLC methods recommend a C18 column for the analysis of polycyclic aromatic hydrocarbons, resolution of isobaric compounds, such as the benzofluoranthenes, can be quite poor. Restek offers 2 HPLC columns that have been optimized specifically for PAHs and offer greater selectivity for these key compounds. Critical PAHs that cannot be distinguished by mass spectrometry can be reliably separated using either Pinnacle® II PAH or Pinnacle® DB PAH columns. Pinnacle® II PAH columns are available in standard formats, while the Pinnacle® DB PAH columns are offered on 1.9µm silica. Labs analyzing PAHs by either HPLC or UHPLC will benefit from the reliable separations obtained using Restek PAH columns.

Pinnacle® DB PAH UHPLC Columns

Physical Characteristics:

particle size: 1.9µm
pore size: 140Å
endcap: yes
pH range: 2.5 to 8
temperature limit: 80°C

1.9µm Columns	cat. #
50mm, 2.1mm ID	9470252
100mm, 2.1mm ID	9470212

Pinnacle® II PAH Columns

Physical Characteristics:

particle size: 4µm, spherical
pore size: 110Å
endcap: fully endcapped
pH range: 2.5 to 8
temperature limit: 80°C

4µm Columns	cat. #
50mm, 2.1mm ID	9219452
150mm, 2.1mm ID	9219462
50mm, 3.2mm ID	9219453
150mm, 3.2mm ID	9219463
50mm, 4.6mm ID	9219455
150mm, 4.6mm ID	9219465

ordering note

For guard cartridges for these columns, visit our website at **www.restek.com**.

Visit **www.restek.com/chromatograms** for PAHs and other HPLC applications.

Analyze 40% More Samples per Shift

Using Split Injection for Semivolatiles

By Michelle Misselwitz, Innovations Chemist, and Jack Cochran, Director of New Business and Technology

- Faster oven cycle increases sample throughput.
- Better precision at trace levels, compared to splitless injection.
- Reliably meet or exceed method requirements for sensitivity and linearity.

Semivolatiles are typically analyzed using splitless injection, but this approach results in slow analysis times and injection-to-injection variability. Combined, these factors reduce the number of samples that can be analyzed before quality control criteria are no longer met. This article demonstrates the advantages of split injection in terms of sample throughput, sensitivity, and linearity for EPA Method 8270D.

Increase Sample Throughput with Faster Oven Cycles

Split injection produces narrower injection bands and uses higher initial oven temperatures than splitless injection. Two oven programs starting at 80 °C were compared to a typical splitless program, and the faster oven cycle times used with split injection allowed up to 10 more samples to be analyzed per shift (Table I). The fastest program resulted in reduced separation of dibenz(a,h)anthracene and indeno(1,2,3-cd)pyrene (Figure 1), but these compounds were fully resolved using the alternate split conditions. The 80 °C oven start temperature could not be used with splitless injection, as it resulted in extremely broad peaks that could not be integrated.

Split Injection Results in More Reliable Sensitivity and Excellent Linearity

In addition to increasing sample throughput, split injection provided good sensitivity and better injection-to-injection repeatability at 0.5 ng on-column than splitless injection. Minimum response factor criteria were easily met and lower relative standard deviations (% RSD) for base/neutral and acid extractable compounds were achieved at the lowest calibration level (Table II). Calibration curves (5-160 ng/µL) were also assessed and, even with the 10:1 split, response factors met the method criterion of <20% RSD, except for 2,4-dinitrophenol (Table III). In this case, calibration was established based on the correlation (r = 0.9997). Better repeatability at low levels makes it easier to meet method criteria and allows more injections to be made before maintenance is required.

Table I Split injection significantly increases sample throughput compared to splitless injection.

	Split (Fast Cycle)	Split (Faster Cycle)	Splitless
Total run time (min.)	21	18.5	25.5
Sample analysis (min.)	18	15	20
Oven cooling (min.)	3	3.5	5.5
Sample throughput* (Samples/shift)	30	34	24
% Increase in sample throughput (vs. splitless)	25%	42%	--

* 12-hr. shift = 10.5 hr. sample analysis period + 1.5 hr. quality control/method performance analysis period. Sample throughput calculation based on number of samples that can be analyzed in 10.5 hours.

Table II Using split injection results in greater repeatability at 0.5 ng on-column, allowing more samples to be analyzed before maintenance is required.

		Split (10:1)		Splitless	
	8270D Min. RF	RF	%RSD	RF	%RSD
Pyridine	--	1.534	2	1.038	9
Phenol	0.800	1.861	0.7	1.857	5
1,4-Dichlorobenzene-d4	ISTD	ISTD	ISTD	ISTD	ISTD
N-Nitroso-di-*n*-propylamine	0.500	1.053	2	1.266	3
2,4-Dichlorophenol	0.200	0.317	2	0.325	3
Naphthalene-d8	ISTD	ISTD	ISTD	ISTD	ISTD
Naphthalene	0.700	1.249	0.5	1.238	2
Hexachlorocyclopentadiene	0.050	0.407	1	0.414	5
2-Nitroaniline	0.010	0.395	3	0.514	3
Acenaphthylene	0.900	2.188	0.9	2.139	1
Acenaphthene-d10	ISTD	ISTD	ISTD	ISTD	ISTD
2,4-Dinitrophenol	0.010	0.113	8	0.127	13
4-Nitrophenol	0.010	0.256	6	0.296	5
4,6-Dinitro-2-methylphenol	0.010	0.175	6	0.110	9
N-Nitrosodiphenylamine	0.010	0.712	1	0.694	1
Pentachlorophenol	0.050	0.115	3	0.098	5
Phenanthrene-d10	ISTD	ISTD	ISTD	ISTD	ISTD
Phenanthrene	0.700	1.252	0.7	1.259	2
Perylene-d12	ISTD	ISTD	ISTD	ISTD	ISTD
Benzo(ghi)perylene	0.500	0.940	4	0.252	26
		Avg. %RSD	3	Avg. %RSD	6

Comparison based on faster cycle split conditions shown in Figure 1; 0.5 ng on-column (n = 5).
ISTD = internal standard

Conclusion

Sample throughput for semivolatiles analysis can be significantly increased by employing split injection with a higher initial oven temperature and faster cycle time. Compared to splitless injection, analysis times are faster and repeatability is improved, allowing more samples to be run per shift.

For the complete technical details, visit **www.restek.com/adv012**

Table III Split injection provides excellent linearity across the typical calibration range of 5–160 ng/µL.

	Avg. RF	Avg. %RSD
Pyridine	1.533	0.9
Phenol	1.787	2
N-Nitroso-di-*n*-propylamine	0.991	2
2,4-Dichlorophenol	0.272	3
Naphthalene	0.998	5
Hexachlorocyclopentadiene	0.383	6
2-Nitroaniline	0.414	6
Acenaphthylene	1.824	3
2,4-Dinitrophenol	0.157	26
4-Nitrophenol	0.264	8
4,6-Dinitro-2-methylphenol	0.123	19
N-Nitrosodiphenylamine	0.608	3
Pentachlorophenol	0.127	16
Phenanthrene	1.082	5
Benzo(ghi)perylene	0.942	5

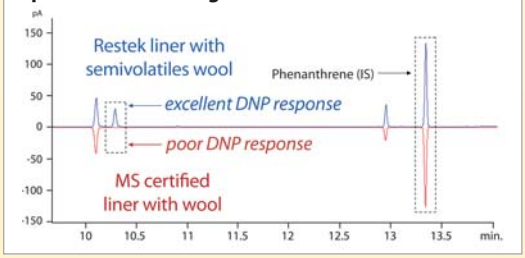
Data acquired using faster cycle split conditions shown in Figure 1; 6-point calibration curve (5, 20, 40, 80, 120, and 160 µg/mL), n = 3 at each level.

TECH TIP!

Increase Accuracy with an Inert Sample Path

Semivolatiles Wool from Restek improves precision and accuracy, while protecting your column from contamination. This new wool is more inert than a competitor's MS Certified Wool and gives you more reliable trace level results. For the complete comparison, visit **www.restek.com/adv017**

Response of 10 ng of 2,4-dinitrophenol relative to phenanthrene using a flame ionization detector.



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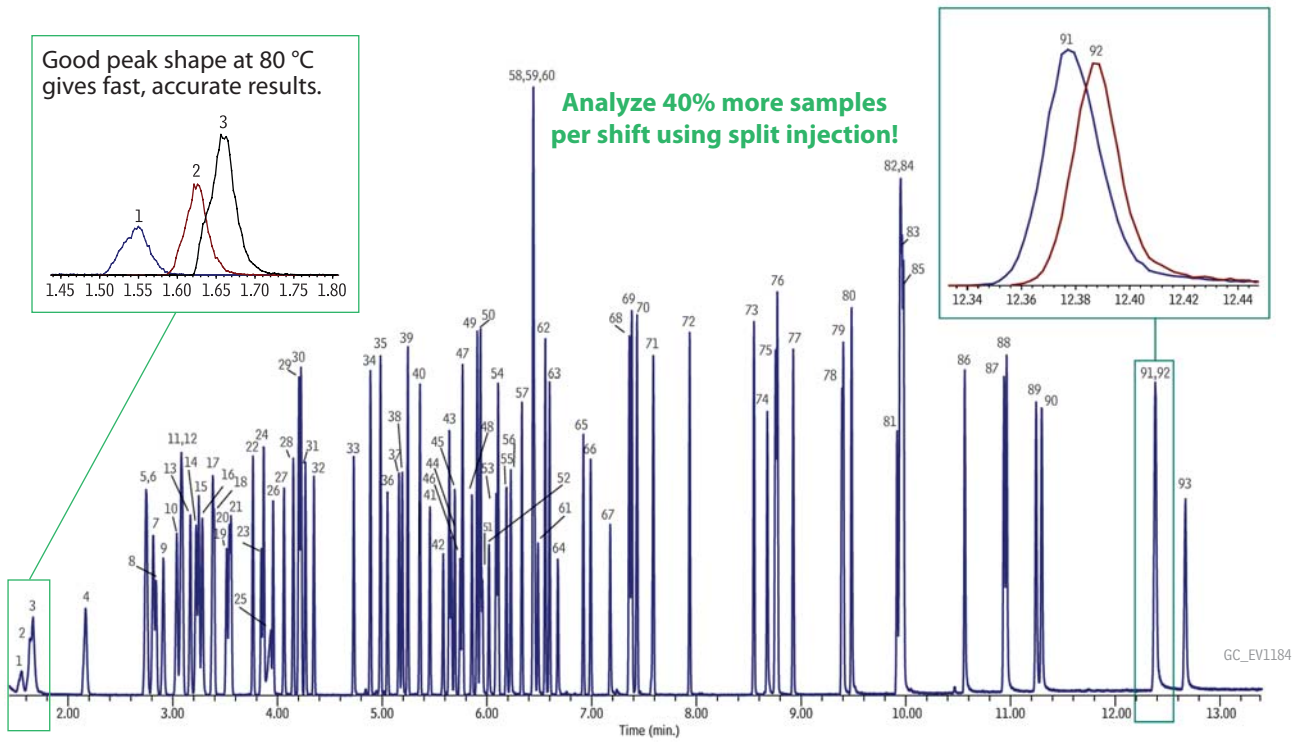
Visit **www.restek.com/liners** for a full product listing.

qty.	Deactivated Liner with Semivolatiles Wool
each	-231.1
5-pk.	-231.5
25-pk.	-231.25

For column information, see page 7 or visit **www.restek.com**

Figure 1 Analyzing semivolatiles in less than 13 min. is possible with split injection, due to faster temperature programming.



Column: Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623) **Sample:** 8270 MegaMix® (cat.# 31850) Benzoic acid (cat.# 31879); 8270 Benzidines Mix (cat.# 31852); Acid Surrogate Mix (4/89 SOW) (cat.# 31025); 1,4-dioxane (cat.# 31853); Revised B/N Surrogate Mix (cat.# 31887); SV Internal Standard Mix (cat.# 31206); Diluent: Methylene chloride; Conc.: 40 µg/mL (4 ng on-column); **Injection:** Inj. Vol.: 1.0 µL split (split ratio 10:1); Liner: 4mm Split Precision® Liner w/ Semivolatiles Wool (cat.# 21023-231.5); Inj. Temp.: 270 °C; Split Vent Flow Rate: 60 mL/min.; Oven Temp: 80 °C (hold 1 min.) to 320 °C at 25 °C/min. to 330 °C at 5 °C/min. (hold 2 min.); **Carrier Gas**: He, constant flow; Flow Rate: 1.2 mL/min.; **Detector**: MS; Mode: Scan; Transfer Line Temp.: 280 °C; Analyzer Type: Quadrupole; Source Temp.: 250 °C; Quad Temp.: 150 °C; Tune Type: DFTPP; Ionization Mode: EI; Scan Range: 35-400 amu; **Instrument**: Agilent 7890A GC & 5975C MSD. For peak list, enter chromatogram GC_EV1184 in search box on www.restek.com

Avoid Resampling Soil Vapors

Confirm Tracer Gas in the Field Using a Leak Detector

By Irene DeGraff, Air Monitoring Product Marketing Manager, Russell Pellegrino, Director of Technical Services*, and Kelli Steindl, GC Accessories Product Marketing Manager
* Centek Laboratories, LLC

- Confirm system integrity before sample collection.
- Minimize resampling by detecting leaks prior to sampling.
- Eliminate costly and time-consuming lab analysis of tracer gas.

Vapor intrusion occurs when pollutants from contaminated soil or ground water migrate into buildings and ambient air. Adverse health effects can result when vapors occur in high concentrations, or if toxic volatile organics are present. These compounds are monitored using a variety of sampling procedures, including soil vapor, sub-slab, indoor, and ambient air testing. Sample collection for volatile organic compounds (VOCs) typically is performed with an air canister and passive sampling kit according to EPA Method TO-15 or a similar method.

Costly Detection in Lab Doesn't Prevent Resampling

The primary challenge in vapor intrusion monitoring is distinguishing vapor intrusion from other sources of exposure. In order to establish that VOCs are from soil vapor, rather than from the surrounding environment, sampling systems (ports) must be tested with tracer compounds, such as helium, and shown to be properly sealed. Sample collection system integrity can be demonstrated by including the tracer gas in the list of target analytes reported by the laboratory; however, if high levels are found the sample is rejected and costly resampling may result.

Using a Leak Detector in the Field Saves Time and Money

Detection of tracer gas in the field is a cost-effective alternative to lab analysis that assures the integrity of the sampling system before sampling occurs. The Restek Leak Detector provides good screening of helium tracer gas at concentrations of 10%, the level at which sample port resealing is required. In addition, this unit is just a fraction of the cost of other field portable devices, such as photionization detectors, which may be too sensitive for screening purposes.

Real-time detection of helium tracer gas in the field using a Restek Leak Detector as shown in Figure 1 is a simple, inexpensive way to minimize resampling by establishing system integrity prior to sample collection. Centek Laboratories pioneered this technique and contributed to its inclusion in the New York State Department of Health method[1].

References
1. New York State Department of Health, October 2006, Guidance for Evaluating Soil Vapor Intrusion in the State of New York, http://www.nyhealth.gov/environmental/investigations/soil_gas/svi_guidance/docs/svi_main.pdf (accessed August 27, 2010).

Prevent costly resampling—use a Restek Electronic Leak Detector to ensure sample collection system integrity prior to sampling.

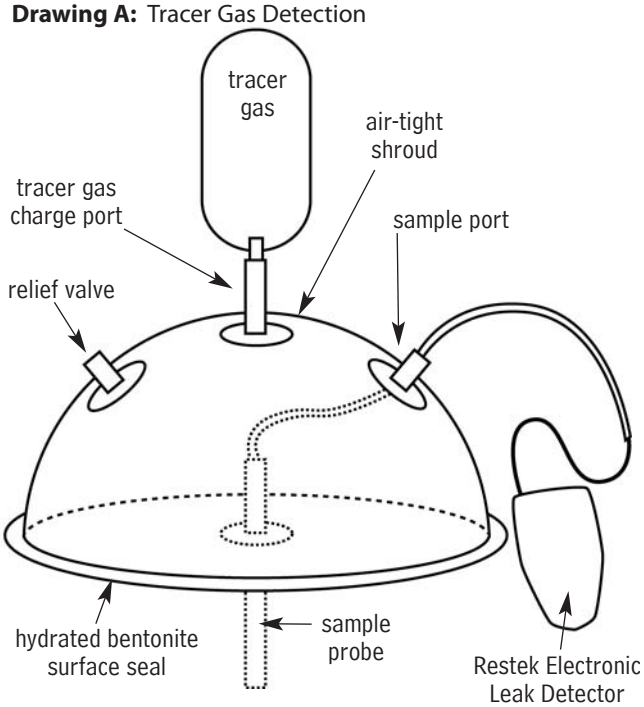
Use a Restek Leak Detector to verify the sample port is sealed (Drawing A).

1. Prepare sampling port by installing sample probe and shroud as described in NY DOH method.
2. Turn on Restek Electronic Leak Detector and allow it to equilibrate for a few seconds prior to use.
3. Insert leak detector probe tip into the open end of the tubing connected to the sealed sample port.
4. Wait 10 seconds and inject a charge of helium into the open space of the shroud.
5. Wait several minutes. An alarm will sound if helium is detected at >10%, indicating a leak in the sample port.

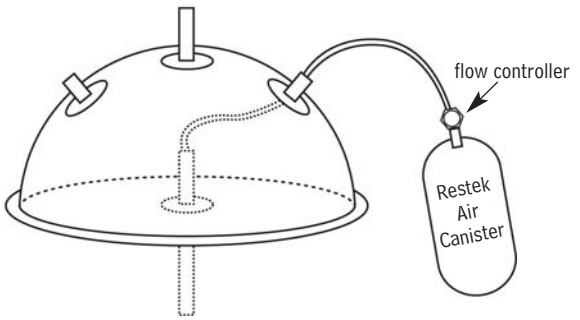
Photos courtesy of Centek Laboratories, LLC

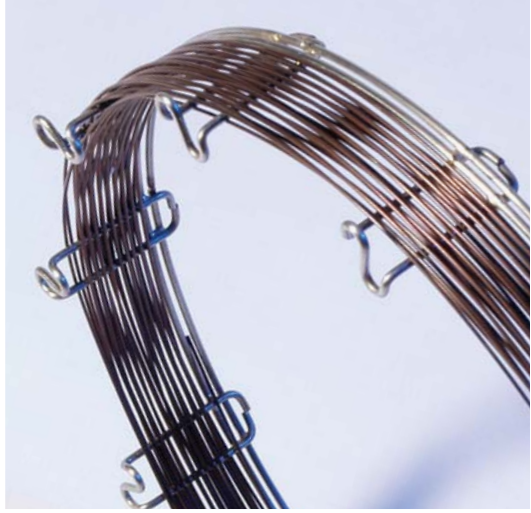
Collect sample using any Restek air canister or mini-can (Drawing B).

Drawing A: Tracer Gas Detection



Drawing B: Sample Collection





New D3606 Column Set

Outperforms TCEP Columns for Benzene Analysis

By Barry Burger, Petroleum Chemist and Jan Pijpelink, Petrochemical Market Development Manager

- Complete resolution of ethanol and benzene allows tighter process control.
- Fully conditioned column set—ready to use out of the box.
- Each column set is tested for method applicability and includes chromatogram.

Demand for finished gasolines containing ethanol continues to increase, as these fuels reduce greenhouse gas emissions and can help control air pollution. Ethanol is a cost-effective additive, but its presence significantly complicates the analysis of benzene, a regulated carcinogen which is added to increase octane levels. A new D3606 column set developed by Restek separates benzene and ethanol completely and more reliably than the 1,2,3-tris(2-cyanoethoxy) propane (TCEP) column listed in ASTM Method D3606.

Independent Testing Shows New D3606 Column Set Outperforms TCEP

It is widely recognized that TCEP columns often fail to adequately separate ethanol and benzene (Figure 1). In contrast, the new D3606 column set from Restek reliably produces resolution values greater than 3.00 for these compounds, allowing easy integration and more accurate quantification of benzene than is typically obtained on TCEP columns (Figure 2). Independent analysis of finished gasoline by beta testers has also produced excellent results (Figure 3). Linearity was assessed and correlations of 0.99999 and 1.00000 were obtained for benzene and toluene calibration curves respectively. Beta testers also reported that repeatability was excellent and that overall reliability exceeded typical TCEP column performance.

Reliable Performance Guaranteed

In addition to inadequate resolution of ethanol and benzene, TCEP columns often show poor thermal stability (max 135 °C). This results in short column lifetimes, making TCEP columns a relatively expensive choice in terms of cost-per-injection and the down-time required for frequent column changes. In comparison, Restek's D3606 column set is stable to 165 °C and exhibits very low bleed, allowing accurate integration and quantification. Reliable performance is assured, as all D3606 column sets are individually tested for method applicability.

Conclusion

Both in-house data and results from independent testers demonstrate that the Restek D3606 column set substantially outperforms TCEP columns and provides more accurate and reliable data for quantifying benzene in finished gasolines.

For the complete version of this condensed article, visit **www.restek.com/adv013**

did you **know**?

The D3606 column set developed by Restek provides accurate, reliable results for benzene and toluene in finished gasoline and will be added to the appendix of ASTM Method D3606. Compared to TCEP columns, this new column set provides better separation of benzene and ethanol.

D3606 Application Column Set

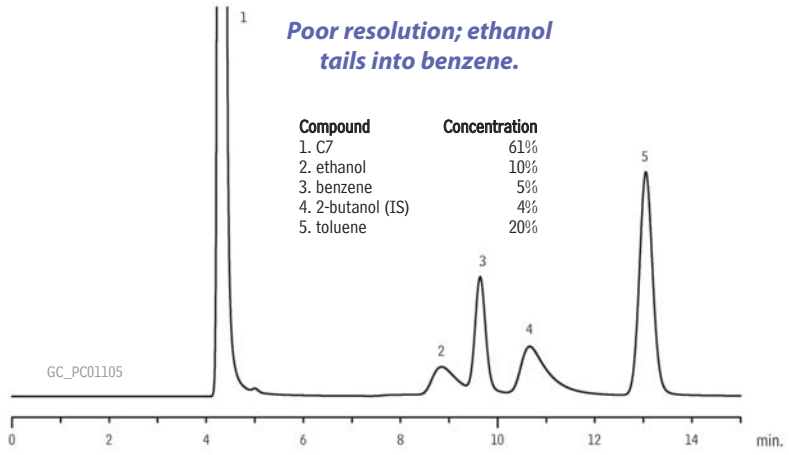
(2 column set)**:
Column 1: 6' (1.8m), 1/8" OD, 2.0mm ID, nonpolar Rtx-1
Column 2: 16' (4.9m), 1/8" OD, 2.0mm ID, proprietary packing material

Description	cat.#*
D3606 Application Column (2 column set)**	83606-

*Please add column configuration suffix number from our catalog to cat.# when ordering—see our catalog or website.
**The column set is designed to accommodate both valve injection and/or syringe injection. Column 1 is configured with a 2" inlet void to facilitate on-column injection. The inlet is identified on both column 1 and column 2. Note: The inlet of column 2 is identified for proper orientation for connection to the valve.

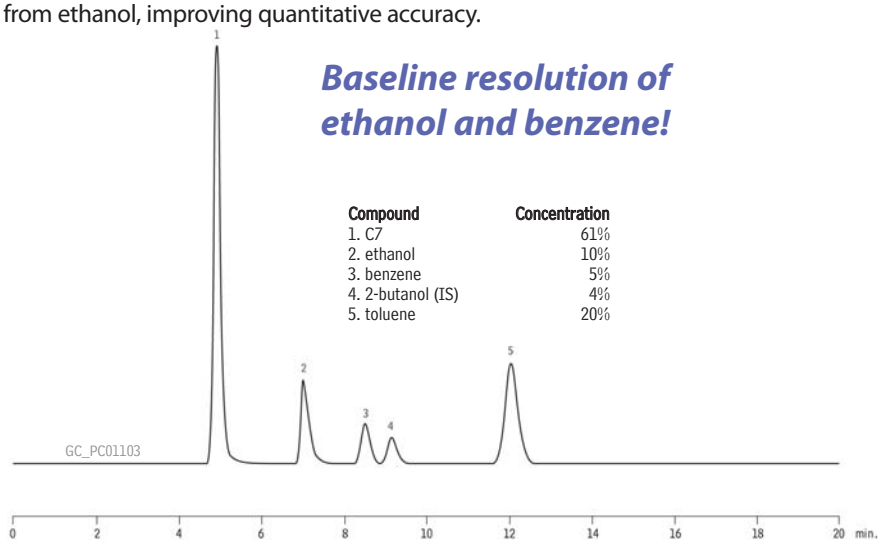
For a complete list of D3606 reference standards, visit **www.restek.com/petro**

Figure 1 TCEP columns often fail to adequately resolve benzene from ethanol, resulting in poor quantitative results



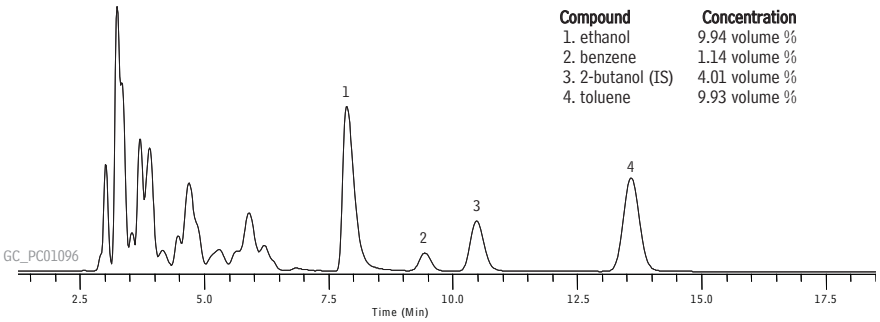
Column: D3606 TCEP Column Set-Up (Column 1: 5', 1/8" OD, 10% OV 101 on Chromosorb PAW 80/100, Column 2: 5', 1/8" OD, 20% TCEP on Chromosorb PAW 80/100, and Column 3: 15', 1/8" OD, 15% Carbowax 1540 on Chromosorb W); **Instrument:** Agilent 6890; **Sample:** mixed standard prepared in C7; **Inj.:** 1 µL, direct; **Inj. temp.:** 200 °C; **Carrier gas:** helium, constant flow; **Flow rate:** 26 mL/min.; **Oven temp.:** 135 °C, isothermal; **Det.:** TCD @ 200 °C

Figure 2 Restek's new D3606 column set accurately and reliably separates benzene from ethanol, improving quantitative accuracy.



Column: D3606 Application Column (2 column set, cat.# 83606) (Column 1: nonpolar Rtx®-1, 6' (1.8 m), 1/8" OD, 2.0 mm ID and Column 2: proprietary packing material, 16' (4.9 m), 1/8" OD, 2.0 mm ID); **Instrument:** Wasson 3606/5599 combo 6890N GC; **Sample:** mixed standard prepared in C7; **Inj.:** 1 µL, direct; **Inj. temp.:** 200 °C; **Backflush time:** 3 min.; **Carrier gas:** helium, constant flow; **Flow rate:** 25 mL/min.; **Oven temp.:** 135 °C, isothermal ; **Det.:** TCD @ 200 °C

Figure 3 Ethanol and benzene are reliably resolved in commercial gasoline by beta testers using the D3606 column set.



Column: D3606 Application Column (2 column set, cat.# 83606-800) (Column 1: 6' (1.8 m), 1/8" OD, 2.0 mm ID, nonpolar Rtx®-1 and Column 2: 16' (4.9 m), 1/8" OD, 2.0 mm ID, proprietary packing material); **Sample:** gasoline; **Inj.**: 1 µL; **Backflush:** 3.0 min.; **Carrier gas**: helium; **Flow rate**: 20.4 mL/min.; **Oven temp**.: 135 °C; **Det.**: TCD; Courtesy of Joaquin Lubkowitz, Separation Systems, Gulf Breeze, Florida

TECH TIP!

Increase column lifetime!

Using Restek gas filters is an easy way to increase GC column lifetime. Our triple gas filters protect your column by removing hydrocarbons, oxygen, and moisture.

Restek Super-Clean Gas Filter Kit

- High-purity output ensures 99.9999% pure gas (at max. flow of 2L/min.).
- "Quick connect" fittings for easy, leak-tight cartridge changes.
- Glass inside to prevent diffusion; polycarbonate housing outside for safety.
- All traps measure 10 5/8" x 1 3/4" (27 x 4.4 cm).
- Each base plate unit measures 4" x 4" x 1 7/8" (10.2 x 10.2 x 4.8 cm).

Description	qty.	cat.#
Carrier Gas Cleaning Kit Includes: mounting base plate, 1/8" inlet/outlet fittings, and oxygen/moisture/hydrocarbon Triple Gas Filter	kit	22019

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Develop Methods Quickly and Easily Using Ultra Selective Chromatography

Ultra Selective Liquid Chromatography™

What is Ultra Selective Liquid Chromatography™? USLC™ is the directed application of selectivity—the most influential factor affecting resolution—to optimize separations and improve method performance. Restek has extensively studied reversed phase selectivity to provide practicing chromatographers with the most effective and widest range of USLC™ stationary phase chemistries available.

RESTEK USLC™
Ultra Selective Liquid Chromatography™

Selectivity Drives Separations

By understanding and controlling selectivity through USLC™, chromatographers have the best opportunity for fast, effective analyte resolution.

One of the most significant challenges in method development is finding the proper stationary and mobile phase chemistry for a particular separation. As sample complexity increases, achieving adequate resolution between matrix components and target analytes becomes more difficult. Despite recent advancements in column format, such as sub-2 micron packings and pellicular particles, resolution can still be difficult to obtain because, while these formats can increase chromatographic efficiency and analysis speed, they do not significantly influence resolution. Selectivity, as shown in Equation 1, is the single most powerful factor affecting resolution, and it is largely dependent upon stationary phase composition.

Real Diversity in Phase Chemistry

Restek columns offer the widest range of selectivities available on a single column line. More choices mean optimized separations and more robust methods.

Equation 1: Selectivity drives resolution—USLC™ considers column selectivity during method development, resulting in fast, effective separations.

$$R = 1/4\sqrt{N} \times (k'/k'+1) \times (\alpha-1/\alpha)$$

Efficiency *Retention capacity* *Selectivity*

While numerous bonded phases are available for reversed phase chromatography, many are similar and offer only moderate changes in retention (e.g. C8 and C18), rather than significant differences in selectivity. Method development is less laborious and time-consuming when using a full range of column selectivities, including orthogonal phase chemistries like polar embedded, phenyl, and fluorophenyl columns. Restek has led the development of unique USLC™ phases across these phase classes in order to provide chromatographers with a more effective range of column selectivities and innovative column chemistries for method development. The phases shown in Figure 1 provide the widest range of reversed phase selectivity available on any column line, and can be used to guide the least understood and most practically significant part of method development—proper column selection.

Evaluating and Extending Selectivity

Restek leads the industry in USLC™ phase diversity because optimal differences in selectivity are built in during the research and development of our bonded phases.

The diversity in selectivity provided by USLC™ columns can be demonstrated empirically using the hydrophobic-subtraction (HS) model [1]. This model is a novel procedure for characterizing selectivity that uses test probes to define the solute and stationary phase interactions in reversed phase separations. Restek is leading the commercial application of this model by implementing it in the research and development of USLC™ bonded phases. To evaluate phase selectivity using the hydrophobic-subtraction model, the retention characteristics of the solute probes are compared across different phases on the same silica base. In this approach, the range of selectivity is indicated by the degree of scatter along the regression line; high correlations indicate similarity and low correlations represent changes in selectivity across phases (Figure 2). The difference in selectivity across columns can then be quantified based on the correlation by calculating the selectivity (S) statistic for the comparison [2].

Figure 1: Restek columns offer the widest range of unique and effective column chemistries to aid the chromatographer in fast and easy method development.

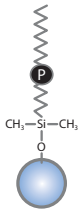
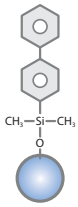
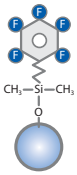
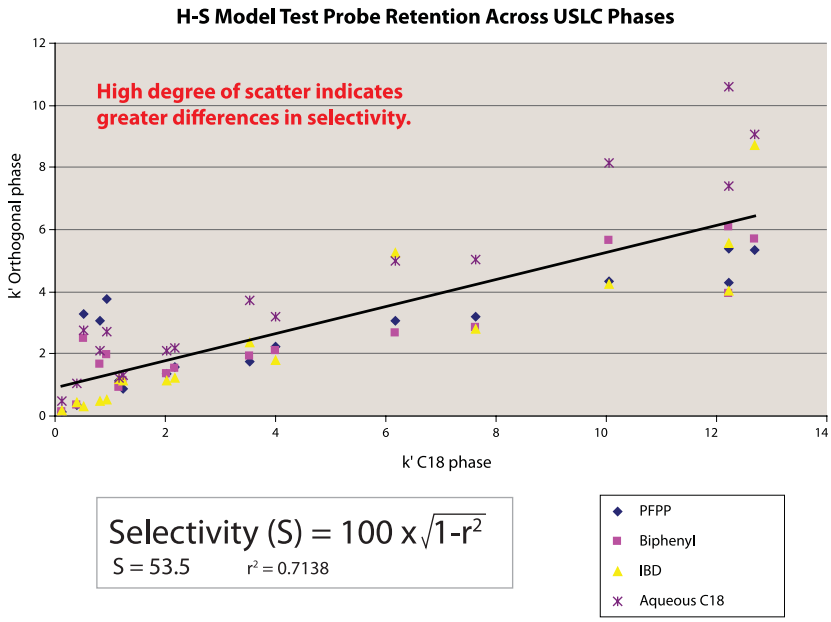
Restek phase (column class)	**Aqueous C18** (alkyl)	**IBD** (polar embedded)	**Biphenyl** (phenyl)	**PFP Propyl** (fluorophenyl)
				
Ligand type	Proprietary polar modified and functionally bonded C18	Proprietary polar functional embedded alkyl	Unique Biphenyl	Proprietary end-capped pentafluorophenyl propyl
Characteristics and uses	• C18 phase for balanced retention of multiple solute types. • Compatible with up to 100% aqueous mobile phases.	• Enhanced retention of polar acids. • Moderate retention of both acidic and basic solutes.	• Increased retention of aromatic, unsaturated, conjugated solutes, or solutes containing an electron withdrawing ring substituent. • Enhanced retention and selectivity when used with methanolic mobile phases.	• Increased retention of protonated bases and solutes containing aromatic moieties.

Figure 2 Restek has extended the selectivity range for reversed phase separations as illustrated by the hydrophobic-subtraction model and corresponding selectivity (S) value.



USLC™ Columns: Selectivity Choices Optimize Separations

Restek USLC™ columns, available in both HPLC and UHPLC formats, offer the widest range of selectivities available and are an integral part of successful method development. Ideal for column switching systems, these columns provide the orthogonal separations needed to create optimal resolution and robust methods. Combining USLC™ phases with a suitable column format gives practicing chromatographers the most powerful tool available for successful method development.

References
1. L.R. Snyder, J.W. Dolan, P.W. Carr, J. Chromatogr. A 1060 (2004) 77.
2. U.D. Neue, J.E O'Gara, A. Mendez, J. Chromatogr. A 1127 (2006) 161.

We're here to help!

To discuss the right selectivity for your separation or to find a comparable column, **contact us at support@restek.com or 800-356-1688.**

Rugged Rxi®-5Sil MS Columns Stand up to Derivatization Reagents, Reducing Downtime and Replacement Costs

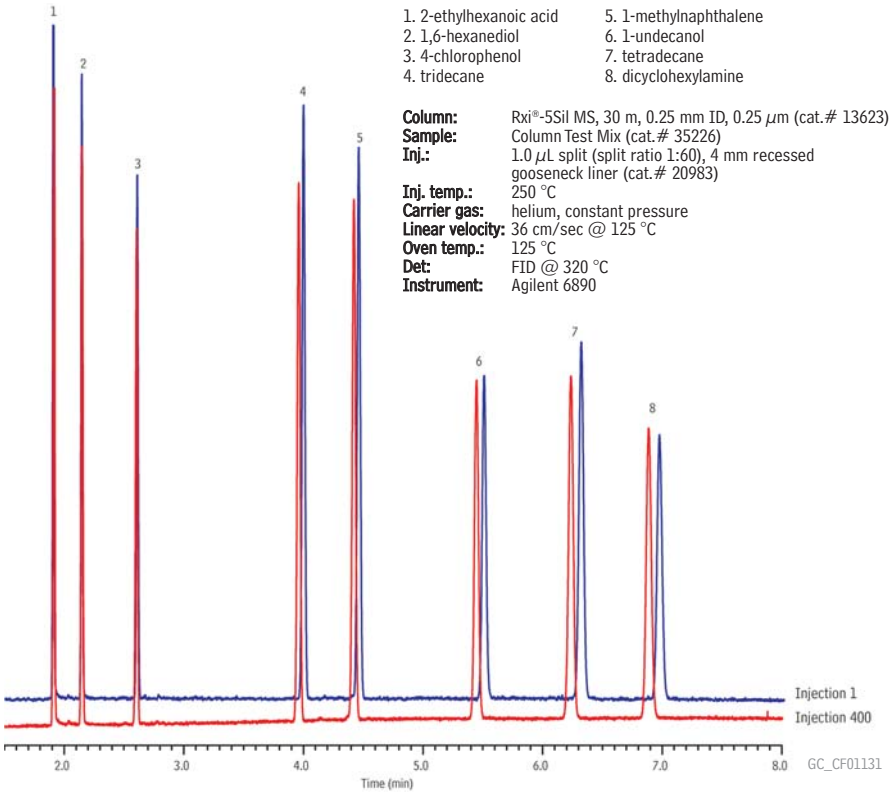
By Amanda Rigdon, Clinical/Forensic Innovations Chemist and Gary Stidsen, GC Columns Product Marketing Manager

- Save costs with long column lifetime.
- Reduce downtime from column trimming and replacement.
- Improve peak shape for active compounds.

When performing GC/MS analysis of drugs, many chemists choose to derivatize samples prior to analysis. Derivatization not only increases the volatility of some drug compounds, but it also reduces activity, resulting in improved peak shape and more accurate quantification. An additional advantage is that derivatized compounds have a higher molecular weight, thus producing more reliable mass spectra than underivatized compounds. Despite these benefits, derivatization reagents are often harsh and can damage analytical columns, leading to high bleed, significant reduction in retention times, and increased tailing for active compounds. Often, this damage is concentrated near the head of the column, so trimming a short length can improve results. However, trimming is a finite solution as repeated clipping ultimately results in decreased efficiency and shorter column lifetimes. Choosing a more rugged column, such as the Rxi®-5Sil MS column, is a better alternative. The Rxi®-5Sil MS column is extremely stable and holds up to harsh treatment, including repeated exposure to derivatization reagents.

The analysis of amphetamine illustrates the ruggedness of the arylene-based Rxi®-5Sil MS polymer. Amphetamine is typically derivatized, because the underivatized form is an active basic compound that produces only a few low molecular weight ions for monitoring. In contrast, upon derivatization, activity decreases, resulting in dramatically improved peak shape and more accurate quantitation. Additionally, several higher molecular weight ions are produced, which can be monitored for definitive identification.

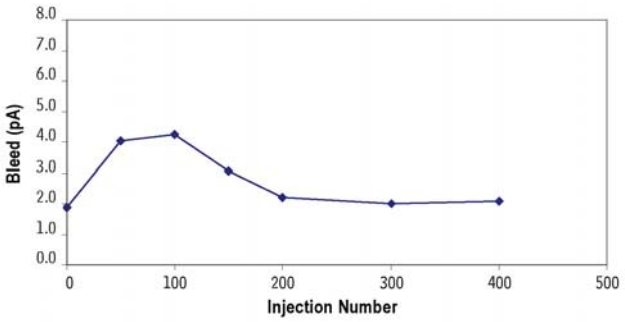
Figure 1 Rugged Rxi®-5Sil MS columns produce consistent retention times, even after 400 injections of derivatization reagent.



Phase Stability Extends Column Lifetime

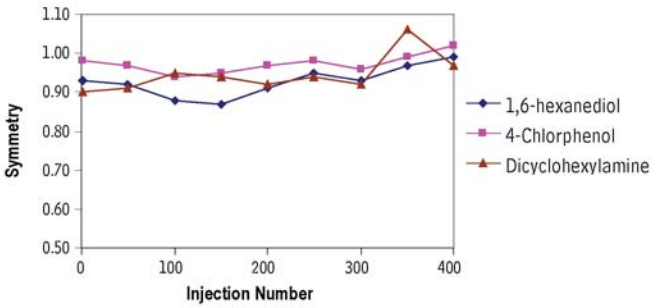
In order to demonstrate the ruggedness of the Rxi®-5Sil MS column, 400 injections of heptafluorobutyric acid anhydride (HFBA) in butyl chloride were performed. HFBA is a very harsh derivatization reagent, and the concentration of reagent in the solvent was equivalent to that of a derivatized sample. Throughout the course of 400 injections, bleed, retention, and peak shape for active compounds were monitored by periodically injecting a column test mix containing active compounds (1,6-hexanediol, 4-chlorophenol, and dicyclohexylamine). Chromatographic results were remarkably consistent, even after 400 injections (Figure 1). Column bleed was monitored over the course of the experiment and remained below 5 pA (Figure 2). The consistency of retention time data and low bleed levels demonstrate phase stability, which results in longer column lifetimes and reduced maintenance and replacement costs.

Figure 2 Low column bleed results in long column lifetimes, saving labs replacement costs.



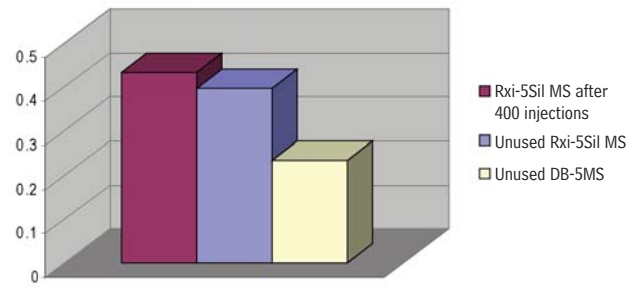
Column bleed over 400 injections of HFBA derivatization reagent.

Figure 3 Active probes show consistent, symmetric peak shape, demonstrating the inertness needed for accurate quantification.



Test probe symmetry over 400 injections of HFBA derivatization reagent.
Symmetry values <1 indicate peak tailing and values >1 indicate fronting.

Figure 4 Peak symmetry for underivatized amphetamine is significantly better on an Rxi®-5Sil MS than on a competitor column, even after 400 injections of HFBA derivatization reagent.



Symmetry values <1 indicate peak tailing and values >1 indicate fronting.

Symmetric Peaks for More Accurate Results

Peak shape was also monitored to ensure column inertness was stable over time—an important factor in maintaining accuracy. Peaks for the active test probes were symmetric even after 400 injections, allowing easy identification and consistent integration (Figure 3). In a second experiment to complement the test probe results, underivatized amphetamine was injected onto a new Rxi®-5Sil MS column, an Rxi®-5Sil MS column after 400 injections of derivatization reagent, and a new competitor column of equivalent phase chemistry. Even though underivatized amphetamine is highly active, peak symmetry on the Rxi®-5Sil MS column was consistent and unaffected by exposure of the column to derivatization reagent. Additionally, peak shape on both the exposed and unexposed Rxi®-5Sil MS column was better than that on the new competitor column (Figure 4).

Conclusion

The rugged arylene phase of the Rxi®-5Sil MS column results in highly stable performance, even under the most demanding of analytical conditions, and its exceptional inertness ensures good peak shape for reproducible quantitation. The stability of the Rxi®-5Sil MS column results in longer column lifetimes, reducing both downtime and replacement costs.

For an online version of this article, visit **www.restek.com/adv018**

Rxi®-5Sil MS Columns (fused silica)

(low polarity Crossbond® silarylene phase; selectivity close to 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	qty.	cat. #
0.10mm	0.10µm	-60 to 330/350°C	10m	ea.	43601
0.18mm	0.18µm	-60 to 330/350°C	20m	ea.	43602
0.18mm	0.36µm	-60 to 330/350°C	20m	ea.	43604
0.25mm	0.10µm	-60 to 330/350°C	15m	ea.	13605
0.25mm	0.10µm	-60 to 330/350°C	30m	ea.	13608
0.25mm	0.25µm	-60 to 330/350°C	15m	ea.	13620
0.25mm	0.25µm	-60 to 330/350°C	15m	ea.	13620-127
0.25mm	0.25µm	-60 to 330/350°C	30m	ea.	13623
0.25mm	0.25µm	-60 to 330/350°C	30m	6-pk.	13623-600
0.25mm	0.25µm	-60 to 330/350°C	30m	ea.	13623-124
0.25mm	0.25µm	-60 to 330/350°C	30m	ea.	13623-127
0.25mm	0.25µm	-60 to 330/350°C	60m	ea.	13626
0.25mm	0.50µm	-60 to 330/350°C	15m	ea.	13635
0.25mm	0.50µm	-60 to 330/350°C	15m	ea.	13635-124
0.25mm	0.50µm	-60 to 330/350°C	30m	ea.	13638
0.25mm	0.50µm	-60 to 330/350°C	30m	ea.	13638-124
0.25mm	0.50µm	-60 to 330/350°C	30m	ea.	13638-127
0.25mm	1.00µm	-60 to 325/350°C	15m	ea.	13650
0.25mm	1.00µm	-60 to 325/350°C	30m	ea.	13653
0.25mm	1.00µm	-60 to 330/350°C	60m	ea.	13697
0.32mm	0.25µm	-60 to 330/350°C	15m	ea.	13621
0.32mm	0.25µm	-60 to 330/350°C	30m	ea.	13624
0.32mm	0.50µm	-60 to 330/350°C	30m	ea.	13639
0.32mm	0.50µm	-60 to 330/350°C	30m	ea.	13639-125
0.32mm	1.00µm	-60 to 325/350°C	30m	ea.	13654
0.32mm	1.00µm	-60 to 325/350°C	30m	ea.	13654-125
0.53mm	1.50µm	-60 to 310/330°C	30m	ea.	13670

Rxi® GC Columns

Innovators in Chromatography

A continuing series of guest editorials contributed by collaborators and internationally recognized leaders in chromatography.

Analytical Chemistry Shapes Response to the Deepwater Horizon Oil Spill

By Ed Overton, Analytical Specialists, Inc.

***Ed Overton** developed the microFAST GC and is the founder of Analytical Specialists, Inc. (ASI). He currently is the principal investigator on a grant to provide NOAA's Office of Response and Restoration with chemical hazard assessments for oil and hazardous chemical spills within US jurisdiction. Prior to retiring in May 2009, Ed held the Claiborne Chair in Environmental Toxicology and Air Quality, an endowed professorship at Louisianna State University.*

The Deepwater Horizon oil spill was an unprecedented event in the annals of US petroleum exploration and development. Much has been made about the comparison to the tragic 1989 Exxon® Valdez spill. Both the current spill and the Alaskan incident are examples of spills that should not have happened and, when all details are known, could have been avoided with more attention to best operational practices and standard safe operating procedures. However, both did occur and we are now faced with trying to mitigate the effects of another major oil spill.

The Exxon® Valdez incident involved the loss of 11,000,000 gallons of oil fairly quickly into the cold waters of Prince William Sound from a floating vessel in close proximity to land. The oil quickly impacted the western islands and shoreline of the sound, and most of the response effort after the first few weeks involved activities to clean these rocky beaches. In the Deepwater Horizon spill, oil was entering the environment at a slower pace, approximately 2,300,000 gallons per day. However, the input was from a leak 1 mile below the Gulf's surface, 50 miles from the closest land, and considerably farther from most shorelines. Unfortunately, the shoreline types most vulnerable to damage from oil spills are marshy, grassy shorelines, like the mostly marshy Louisiana shoreline which represents the open water-land interface for some 40% of our nation's wetlands. Additional coastal marshes were in harm's way along the coasts of Mississippi, Alabama, and Florida.

The Deepwater Horizon spill was a slow moving spill that continued for 87 days and dumped over 200,000,000 gallons of a very volatile, light sweet crude oil into the waters of the Gulf of Mexico. Fresh oil reached the surface each day, and it appears that the oil was mixing with water as it ascended from the depths, stripping mostly aromatic compounds from small droplets. Surface oil formed a water-in-oil emulsion, a mousse, that floated in the water at the surface. Most of the volatile components readily evaporat-

ed. Some percentage of the oil entering the Gulf from the wellhead was both naturally and chemically dispersed at depth, and this very dilute dispersed oil resides and is being degraded in deep water. All of these factors presented scientific and engineering challenges when figuring out how to most effectively mitigate this horrible event.

Human cleanup options consisted mostly of using Corexit® 9500 to disperse the oil on the surface and at the wellhead. *In situ* burning and skimming tactics were also used. Dispersing surface oil in offshore waters certainly speeds up biodegradation, but it also spreads oil within the top 10-20 meters of the water column where marine animal exposure occurs. Dispersing surface oil most certainly mitigated the potential impacts of floating oil on marshy coastlines and sandy beaches along the northern Gulf coastline. It also fueled a massive natural offshore biological treatment process that, as we are now seeing, is rapidly degrading residual spilled oil and allowing the Gulf's environment to recuperate from this massive assault.

Tens of thousands of scientists, engineers, and response personnel worked tirelessly 24/7 to mitigate this spill. Analytical chemists, using techniques like GC/MS, UV fluorescence, and HPLC, played a critical role in guiding response efforts, following the environmental impacts, and ensuring the safety of the seafood harvested from the Gulf region. Analytical chemists are essential in responding to massive environmental disasters, like oil spills, and in monitoring environmental damage and ecological recovery. Thank God for analytical chemists, their impressive technologies, and all the supply companies that support high quality chemical analysis.

Bringing Back the Bluebirds

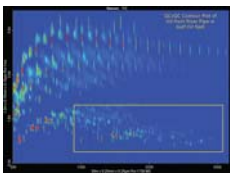
Restek chemist Mike Wittrig, a life-long bird enthusiast, has been supplying local bluebirds and tree swallows with nesting habitat for the past 9 years. What started as a backyard hobby quickly expanded to local parks, and also to the Restek campus where he works with the facility maintenance team to locate nesting boxes in prime locations. "Over the years I've learned the importance of both nest box location and spacing in improving reproductive success rates by limiting competition from sparrows," says Mike. Local initiatives like Mike's project have helped bring Eastern Bluebird populations back to healthy levels, following a critical midcentury decline due to habitat destruction and nesting competition.

HOT TOPICS in Chromatography

ChromaBLOGraphy www.restek.com/blog

GCxGC-TOFMS of Riser Pipe Oil from BP Gulf Oil Spill

posted by Jack Cochran



I recently analyzed an oil sample collected by an ROV from the riser pipe at the BP Gulf oil spill site using GCxGC-TOFMS, a powerful multidimensional technique capable of characterizing complex samples that defy one-dimensional GC-MS. The column setup was a 30 m x 0.25 mm x 0.25 µm Rxi-17Sil MS in the first dimension with a 1.2 m x 0.15 mm x 0.15 µm Rxi-1ms in the second dimension. This arrangement puts the highly aromatic compounds (e.g. PAHs) at the bottom of the contour plot while the aliphatics are retained by the Rxi-1ms in the second dimension, eluting away from the aromatics. Given that PAHs are considered the "toxic" compounds in crude oil, this is an efficient arrangement for their interference-free determination. Having a full mass range TOFMS allows spectral fingerprinting of the resolved components, including PAHs. Read full blog or post comments at **www.restek.com/blog**

See page 4 for a GCxGC analysis of PAHs in mussels!

New NJ-EPH Method

The New Jersey Department of Environmental Protection began phasing in a new analytical method for extractable petroleum hydrocarbons on Sept. 1, 2010. **Restek has all the standards, GC columns, SPE tubes needed for this new method**—contact us for assistance getting set up.

Flip Seal Doubles Lifetime

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See page 7 for details.

FPRW QuEChERS Session

The 47th Florida Pesticide Residue Workshop took place July 18-21 in St. Pete Beach, Florida. As usual, this meeting featured an excellent technical program, which included sessions on the Gulf oil spill, multiclass/multiresidue analyses, veterinary drug residues, global chemical contaminant conflicts/resolutions, and US government residue programs.

In addition to the formal presentations, there was a lively evening discussion on developing a unified QuEChERS method that harmonizes the two current official methods (AOAC and EN 15662), which differ slightly in their approach. No consensus was reached, but attendees enjoyed a vigorous debate moderated by QuEChERS inventors Steve Lehotay (USDA) and Michelangelo Anastassiades (CVUA-Stuttgart). Much discussion centered on efficiencies for just a few pesticides, which is ironic, in a way, considering the effectiveness of QuEChERS for hundreds of pesticides!

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