





# WHAT'S NEW?

Restek 2011

## GC Columns GC Accessories Sample Preparation

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# Rxi-624Sil ms (fused silica)

- -20 to 300/320 deg C.
- 6% cyanopropyl phenyl/ 94% dimethyl arylene polysiloxane
  - Low bleed, high thermal stability
- Inert-excellent peak shape for a wide range of compounds including acidic and basic compounds
- Selective-highly selective for residual solvents, great choice for USP <467>
  - Manufactured for column to column reproducibility-great for validated methods.

# Rxi-1HT (fused silica)

- Columns processed for high-temperature applications.
  - Temperature range: -60 to 400 deg C.
- Column capable of 430 deg C, but will have reduced lifetime

# Rtx-Volatile Amine Column (fused silica)

- -60 to 290 deg C
- Unique selectivity for baseline resolution of all volatile amines.
- Excellent inertness assures accuracy and sensitivity for volatile amines including free ammonia.
- Highly robust phase withstands repeated water injections, resulting in longer column lifetime.
- High temperature stability(290 deg C) ensures elution of amines up to C16 and allows contaminants to be removed by “baking out” the column.

# Rtx-DHA Columns (fused silica)

- -60 to 340 deg C.
- 100% dimethyl polysiloxane
- Columns meet or exceed all ASTM D6730-01 and Can/CGSB 3.0 No.14.3-99 method guidelines.
- Excellent response and peak symmetry for polar oxygenates.

# Rt-Alumina Bond/CFC Columns (fused silica PLOT)

- To 200 deg C.
- Improved inertness for halogenated compounds such as CFC's
- Highly selective alumina based column, separates most CFC's.
- High retention and capacity for CFC's

# Siltek-stainless steel PLOT Columns

## MXT-Alumina Bond/NaSO<sub>4</sub> Columns

- Can be made in small coil diameters.
- Will not spontaneously break, making them ideal for rugged environments.
- Designed for robust performance in process GC's and field instruments.
  - To 200 deg C.

## MXT-Q-Bond Columns (New 0.25mm also)

- To 300/320 deg C.

## MXT-S-Bond Columns (New 0.25mm also)

- To 250 deg C.

# MXT-1HT SimDist Column (Siltek-stainless steel)

- Stable to 450 deg C. –Lowest bleed for longest column lifetime.
  - Reliably meet all ASTM D6352, D7169, and D7500 specifications.
- 100% dimethyl polysiloxane phase allows easy comparison of historical data.
  - -60 to 430/450 deg C.

# 150 micron ID Columns

- Reduces analysis time up to 2 times, while maintaining resolution.
- Can be installed in all existing instrumentation.
  - Low bleed maximizes sensitivity.
- Available in most selectivity's and also thick films eliminating sample capacity issues.
- OD similar to 0.25mm for easy installation

# Rxi-1ms

- 0.15mm X 0.15um X 10M
  - -60 to 330/350 deg C.

## Rxi-5Sil ms

- 0.15mm X 0.15um X 10M and 20M
  - 0.15mm X 2.0um X 20M
  - -60 to 330/350 deg C.

## Rxi-17Sil ms

- 0.15mm X 0.15um X 10M and 20M
  - 40 to 340/360 deg C.

# Rtx-200

- 0.15mm X 0.15um X 10M and 20M
  - -20 to 320/340 deg C.
  - Rxi-624Sil ms
- 0.15mm X 0.15um X 10M and 20M
  - -60 to 300/320 deg C.

## Stabilwax

- 0.15mm X 0.15um X 10M and 20M
  - 40 to 250/260 deg C.

# New Sky™ Inlet Liners



- Great secure source
- Blue is Beautiful
- New packaging



*Choose blue – the best choice for dependable results.*



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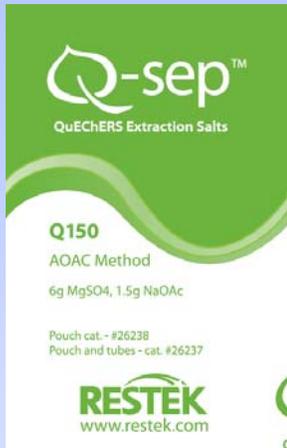
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# QuEChERS Extraction Salts



Why pouched salts?

- Method required
- Customer convenience
- Reduced cost
- First in a series of products to improve and expand our QuEChERS line

# Centrifuge

- Meets requirements of AOAC and European QuEChERS methodology.
- Supports 50mL, 15mL, and 2mL 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 3000g force required by the European method.



Q-sep 3000 Centrifuge  
26230-26235

# Use a smaller bore capillary..

## Reducing Column Internal Diameter...

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# Influence of column diameter on separation

50 m x 0.53 mm => 100.000 plates  
25 m x 0.25 mm => 100.000 plates  
15 m x 0.15 mm => 100.000 plates  
10 m x 0.10 mm => 100.000 plates



*All these columns will generate the same separation*

# Influence of diameter on analysis time \*

50 m x 0.53 mm => 300 seconds

32 m x 0.32 mm => 170 seconds

25 m x 0.25 mm => 120 seconds

15 m x 0.15 mm => 60 seconds

10 m x 0.10 mm => 40 seconds

5 m x 0.05 mm => 25 seconds

*Shorter columns of smaller ID will generate a significant reduction in analysis time*

# Practical Challenges with smaller bore columns..

- **Not for on-column, direct and valve**
  - » Has to be coupled, which is difficult and critical
- **Not ideal for trace analysis**
  - » Column flow is low: need long splitless times/small injection volumes and small volume liners required: pressure Pulse;
- **Loadability is limited**
  - » For traces no problem; Higher level components will require thicker films
- **High inlet pressures required**
  - » Have to watch for leaks: leakage of septum and needles
- **Smaller bore columns are not as “Forgiving”**
  - » Need thicker films here or use guard columns
- **Need fast injection**
  - » To create smallest possible injection band

Is there an other solution for speeding up analysis time that is easier to implement and is less risky?

0.15-0.18 mm ID Fused silica columns

*A good intermediate diameter capillary that provides many advantages for implementing faster GC which is easily achievable with the standard GC*

# Scope of implementation of 0.15-0.18 mm ID columns

Reduction of analysis time by minimal a factor 2

- Same GC
- Same samples
- Same injection system and injection amount
- Same detection system
- Generating the same elution order, only 2 times faster

By:

- 1 Replacing the existing 0.32 or 0.25 mm capillary for a 0.15mm ID column..
- 2 Adjust the carrier gas Flow and the Split Flow..
- 3 Use a recommended faster Oven temperature program..

## OLD METHOD

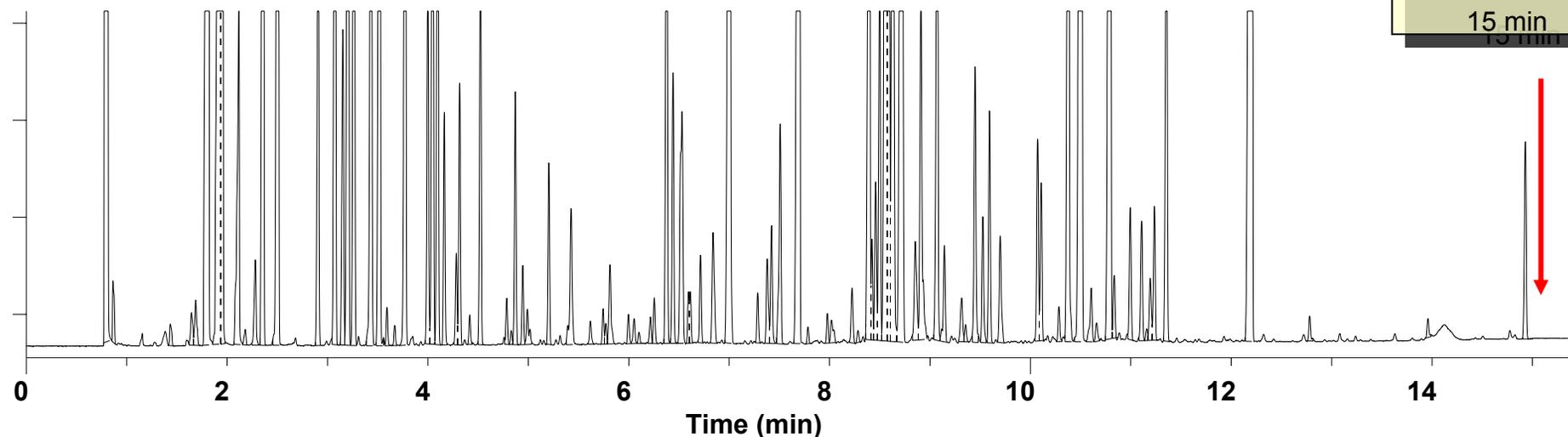
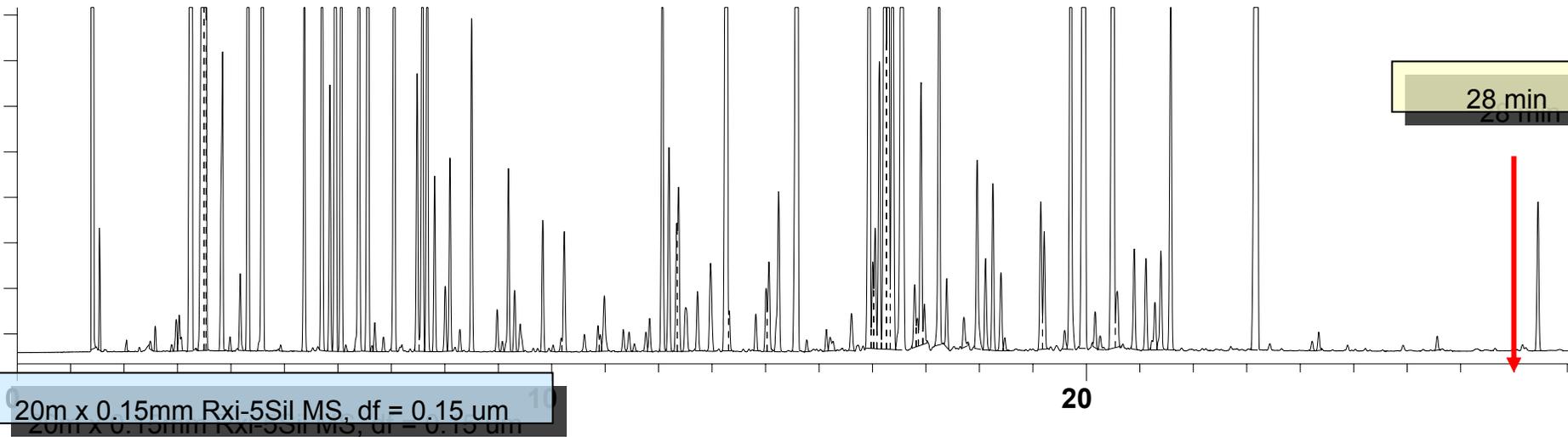
Column : Rxi-5Sil MS, 30m x 0.25mm, df = 0.25  $\mu$ m  
Carrier : H<sub>2</sub>, 1.2 mL/min, u = 36 cm/s constant flow  
Spit inject : 1:100; 1.0  $\mu$ l  
Oven : 100°C, 2 min, 5°C/min  $\rightarrow$  250 °C  
GC : Agilent 6890

## NEW METHOD

Column : Rxi-5Sil MS, 20m x 0.15mm, df = 0.15 $\mu$ m  
Carrier : H<sub>2</sub>, 0.5 mL/min, u = 50 cm/s constant flow  
Spit inject : 1:100; 1.0  $\mu$ l  
Oven : 100°C, 0.9 min, 9.75°C/min  $\rightarrow$  250 °C  
GC : Agilent 6890

30m x 0.25mm Rxi-5Sil MS, df = 0.25  $\mu$ m  
30m x 0.25mm Rxi-5Sil MS, df = 0.25  $\mu$ m

# Analysis of Perfume "Eternity Moment"



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# Sample Preparation

## 2011 Restek Overview



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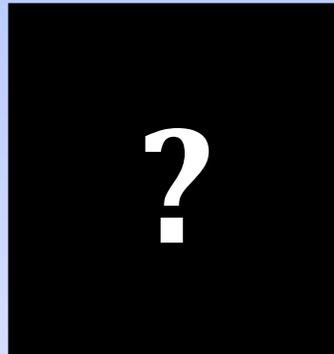
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# Outline

- What is Sample Preparation?
- Key Markets Overview
  - Product Offering
  - New Products
  - Products and applications in development
- Current Sample Prep Objectives
- Promotions

# What is Sample Preparation?



Sample Collection

Sample Analysis

# The Sample Prep Market



- Worldwide extraction market is project to be 370 million dollars in 2012.
- Two thirds of this (\$247 million) is aftermarket
- 22% (\$81 million) is Asian with a growth rate more than double ROW (about 6%)
- No single manufacturer has more than 11% of the market



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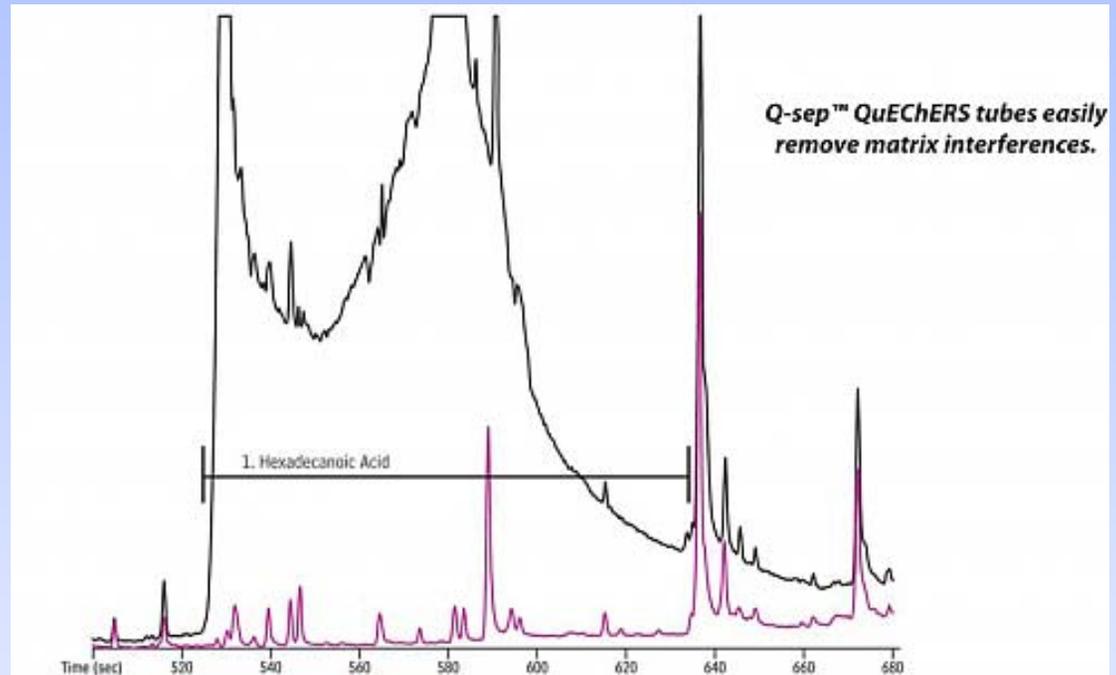
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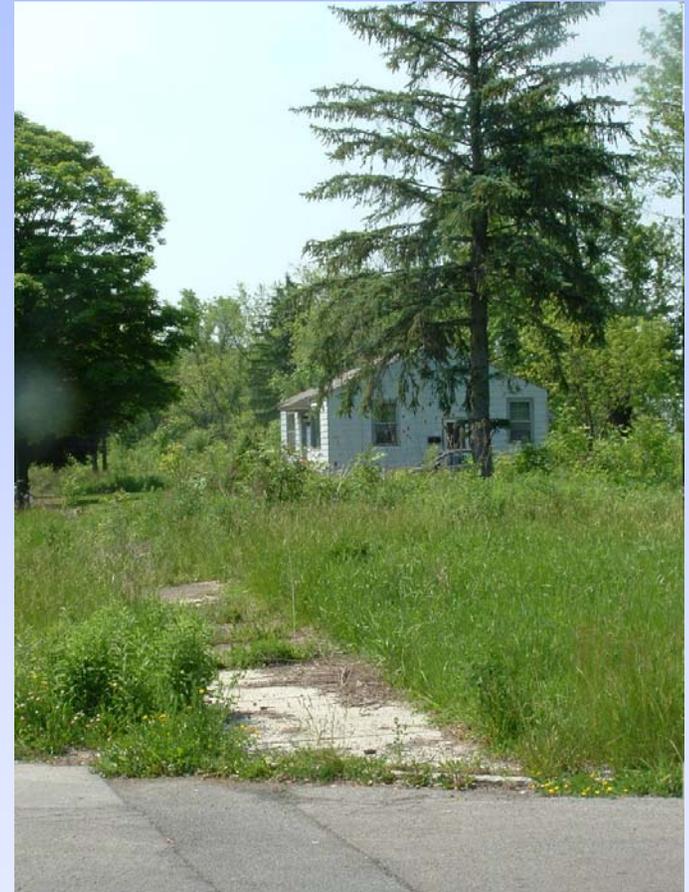
# We want a sample that is:

- Clean
- Concentrated
- And Detectable



# Environmental Market

- Historically a strong market for Restek
- Original sample prep product offering was built to fit the environmental analysis market



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# Solid Phase Extraction (SPE) Concepts

- Liquid/solid chromatography
- “LPLC”
- Disposable extraction column filled with a variety of chromatographic grade sorbents
- Alternative to traditional liquid-liquid extraction

# Why SPE?

- Reduce solvent costs due to smaller amounts of solvents used
  - Buying
  - And disposing!
- Lab supply cost savings
  - Most SPE components are relatively inexpensive
  - Extend life of analytical hardware and consumables due to cleaner samples

# SPE Tubes

- Normal phase – silica, Florisil
- CarboPrep®
- Method Specific
  - EPH Fractionation
  - Coconut charcoal
  - Resins
- Reverse phase – C18, C8
- Ion Exchange – WAX, SCX



# SPE Disks

- Higher reproducibility
- Faster throughput
- Higher recoveries
- Primarily used for large volume water samples
- C18 or C8 embedded in glass fiber disks



# More SPE Products



Bulk Sorbents



Manifolds and replacement parts

# Pressurized Fluid Extraction (PFE)



- Commonly known as ASE<sup>®</sup> (Accelerated Solvent Extraction)
- EPA 3545A
- High temperature, high pressure automated extraction
- Used to remove organics from various solid matrices
- Faster and less solvent usage than Soxhlet



# ASE® Products

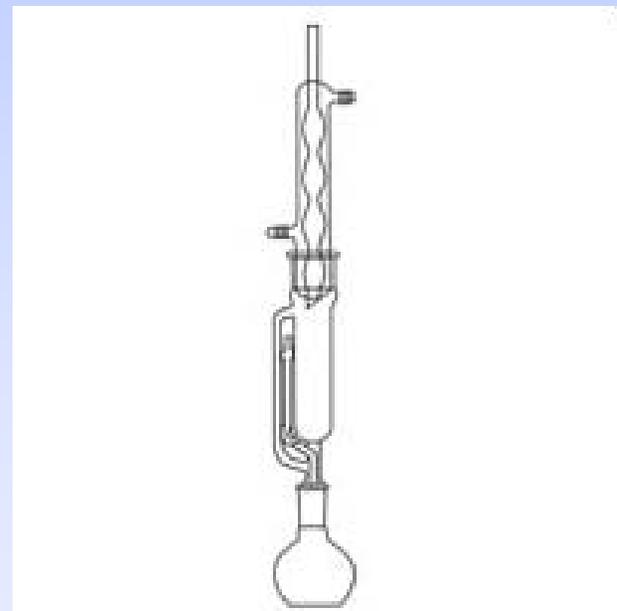


- Full line of cells, caps, and replacement parts for ASE 100, 200, and 300 systems
- Bulk package quantities for high-use items like PEEK washers, glass and cellulose filters, replacement frits
- Currently investigating ASE® 150 and 350 for replacement parts

# Environmental Glassware



- New in 2011 is our line of technical glassware.
- For the environmental market:
  - Soxhlet Extraction Glassware
  - Kuderna-Danish Evaporators



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# Environmental Positioning

- Priced competitively with industry leaders like Agilent, JT Baker, Supelco
- Targets more common, larger volume sample preparation applications
- SPE tubes and ASE consumables part of Restek's core focus on high volume consumables
- Complements Restek's industry leading GC columns and reference standards

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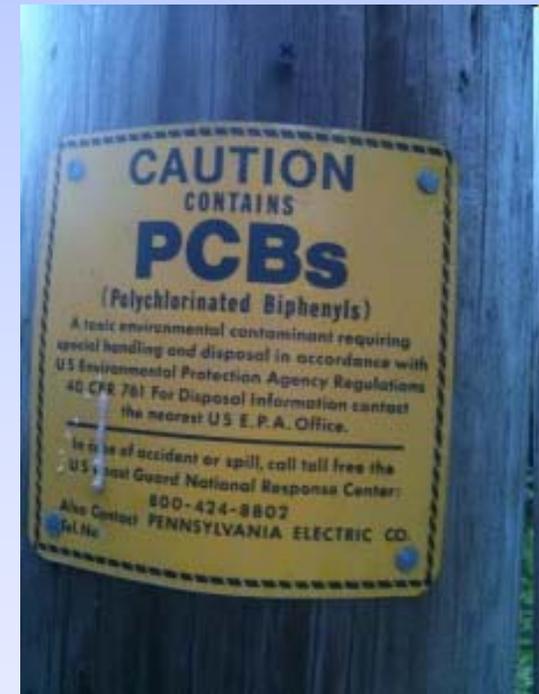
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# Environmental Developments

- New and updated applications for
  - Extractable petroleum hydrocarbons
  - CarboPrep for pesticide extract clean-up
  - Environmental sample prep application resource
- New products in development
  - Larger selection of bulk sorbents
  - Additional SPE materials and formats
  - New ASE parts
  - New glassware



# Food Safety Market

- Smaller market, but rapidly growing especially in Asia and South America
- Restek has a large selection of related products and technical resources
- More details in our **Food Safety Market** overview



# Restek's Total Solution for Food Safety

- GC Columns
- LC Columns
- Analytical Standards
- QuEChERS Sample Preparation
- Related Lab Equipment

**Q-sep™**  
QuEChERS Products

**Your Total Solution for QuEChERS Methods**

Tubes • Standards • Columns • Accessories

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# Food Safety Expertise

- Restek.com/QuEChERS
- ChromaBLOGraphy
- Jack, Jason, Julie, Michelle
- Worldwide tradeshows

Home > Restek Q-sep™ QuEChERS Products

Restek Q-sep™ QuEChERS Products

**Related Links**  
[www.quechers.com](http://www.quechers.com)  
 AOAC Official Method 2007.01  
 CEN European Standards  
 EN 15662 Method  
 More Chromatograms

**Tired of throwing away all those tubes?**

**GO GREEN!**

New Reusable Teflon QuEChERS Centrifuge Tubes

[Shop now](#)



1 2 3

Products Resources Chromatograms

**Q-sep™ QuEChERS Extraction Salts**  
 Fast, Simple Sample Prep for Multi-Residue Pesticide Analysis

- Salt packets eliminate the need for a separate extraction solvent.
- Go green by using packets with reusable tubes.
- Convenient and easy to use.
- Ready-to-use tubes, no glassware required.
- Preweighed, ultra-pure sorbents.
- Support original unbuffered, AOAC (2005) method.



**ChromaBLOGraphy** Visit Restek.com

Topical and timely insights from top chromatographers.

◀ Some answers on the Chromatography challenge posted before: Does a GC-capillary column produce different retention times when installed in the opposite direction? LC/MS/MS Analysis of Synthetic Cannabinoid Metabolites in Urine – The Saga Begins ▶

## First QuEChERS Extraction of Marijuana with GCxGC-TOFMS Analysis, dudes...

April 20th, 2011 by Jack Cochran

My Restek colleagues Julie Kowalski, Michelle Misselwitz, and Amanda Rigdon, along with Professor Frank Dorman from The Pennsylvania State University (PSU), report here what we believe is the first QuEChERS extraction of marijuana, with subsequent analysis using GCxGC-TOFMS. With the help of Randy Hoffman, a Police Officer Specialist/Evidence Technician at PSU, who very kindly donated the samples confiscated from a suspect, probably should have had their minds on class, not grass.

Our interest in this topic is mainly about medicine, since at least 15 states (Pennsylvania is not one of them) and Washington DC have legalized medical marijuana. When you fill your prescription, how do you know your remedy is active (potency, or cannabinoid content), pesticide free, or mold or fungus? Well, you probably don't, but eventually FDA might get involved and we'll need good, robust analytical methods for pesticide analysis. We think that you might be able to do one extraction for both potency and pesticide determinations and we're high on that.

First, the potency work, or cannabinoids determination. Although you don't need GCxGC for the BIG THREE (cannabinoid, Δ9-THC, and CBD), Restek has a reference material containing these compounds... we used it to illustrate one of the benefits of that technique, the structure of the first figure below, the GCxGC contour plot (or chromatogram), you can see that compound classes position themselves in certain areas, and makes discovery of new compounds within classes a bit easier (e.g. perhaps there are undiscovered cannabinoids out there). Zooming in, we can see the terpenoid classes, which are thought to have therapeutic effects. Finally, you can see the cannabinoid, one of much interest given that it has been shown to relieve convulsion, inflammation, anxiety, and nausea, as well as its role in the endocannabinoid system (<http://en.wikipedia.org/wiki/Cannabinoid>).

We quantified cannabinoid (CBD), Δ9-THC (THC), and cannabidiol (CBN) for 4 marijuana samples using QuEChERS and GCxGC-TOFMS and the results are presented in the table below. Since the samples had been stored in an evidence locker for over a year in some cases, the CBN content is relatively high versus fresh marijuana. CBN increases as THC degrades. The THC content is in line with what is typically reported for higher grade illicit marijuana.

Stay tuned for a report on pesticide analysis of marijuana using QuEChERS and GCxGC-TOFMS. As you might imagine, the extracts are extremely complex, similar to what we saw in our dietary supplements work.



cannabinoid



[Click here for more information on our QuEChERS](#)

[Click here for an audio slideshow.](#)

**Q-sep™ 3000 Centrifuge**  
for QuEChERS

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# Universal Products

- Syringe filters
- Filter vials
- Technical Glassware



# Filtration

- Membrane choices
  - Chemical stability
- Pore size
- Surface area
  - Larger sizes can accommodate larger flow rates
  - Larger sizes have more dead volume



# Syringe Filters

- Current Promotion – **10% increased discount** on top of **quantity break** discounts for all of 2011



- Current line includes
  - Cellulose, PVDF, Teflon, and Nylon membranes
  - 13 and 25mm diameter
  - 0.22 and 0.45  $\mu\text{m}$  porosity
- Just released
  - PES membranes
  - 4 and 30mm diameters for all membranes



**NEW!**

# Thomson Filter Vials

- Polypropylene vials with PTFE, Nylon, PVDF, or PES filters
- Simplifies sample filtration – put vial straight in the autosampler.



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# Technical Glassware



- In 2011, we have introduced a line of technical glassware including:
  - Erlenmeyer Flasks
  - Volumetric Flasks
  - Soxhlet Extraction Glassware
  - Kuderna-Danish Evaporators
  - Separatory Funnels
  - And more!



# Technical Glassware

- In 2010, Restek acquired Glastron, a manufacturer of scientific glassware
- Glastron has been making technical glassware for over forty years
- Our current offering is just a limited selection of the most common items
- Market competitive pricing

# QuEChERS Objectives

- QuEChERS products for the Food Safety market
- Focus on the environmental market
- Low price, large volume universal products like syringe filters and glassware

# Food Safety

- Leverage our deep technical knowledge with applications
- Continue to expand our “Total Solution”
- Focus on environmentally friendly “Green” solutions

# Environmental

- Provide the products and applications to support the environmental market
- Focus on key sample preparation products to support our columns and standards
- Increase our application footprint in the market

# Universal Products

- Support the universal consumable products found in analytical labs
- Our acquisition of Glastron strongly positions us to serve the technical glass market
- Offer high-quality, high-volume consumables like syringe filters at competitive pricing

# 2011/12 Promotions

Tired of throwing away all those tubes?

## GO GREEN!

New Reusable Teflon  
QuEChERS Centrifuge Tubes

Shop now



- Purchase one package of reusable Teflon centrifuge tubes and get one box of QuEChERS Extraction Salts

# Pharmaceutical

## Restek Chromatography Products

### 2011 Overview



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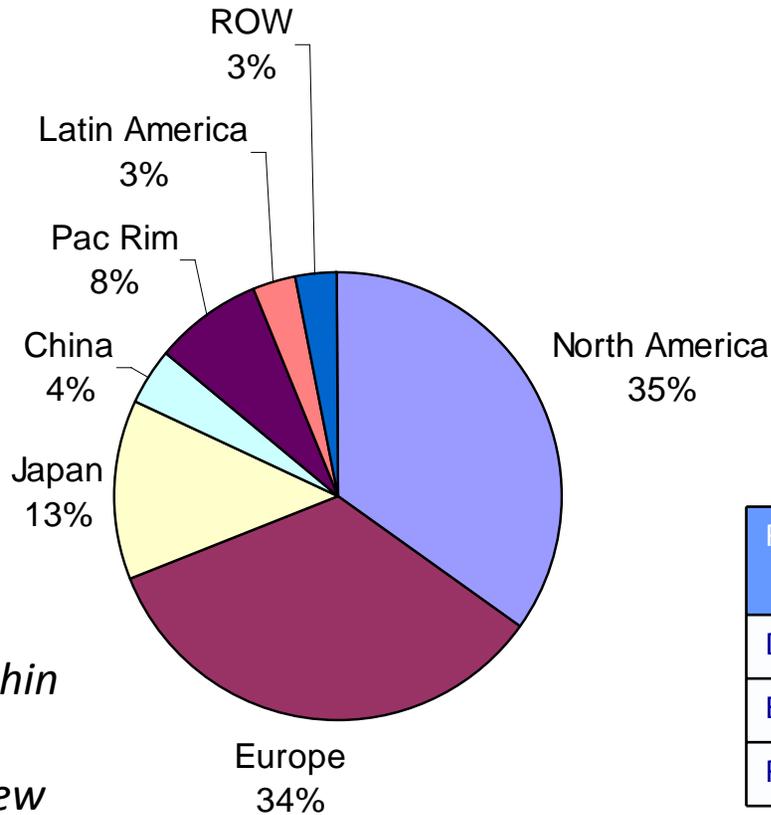
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# Outline

- Market trends
- Pharmaceutical Products for HPLC
- Pharmaceutical Products for GC

# Liquid Chromatography Consumables Market by Geography



Region	Percentage	Estimated TAM
Domestic US	35%	245
Europe	34%	238*
ROW	31%	217

\* Potentially inflated by currency translation



*Large market opportunity within Restek reach, especially for new technology*

2007 SDI Market Forecast

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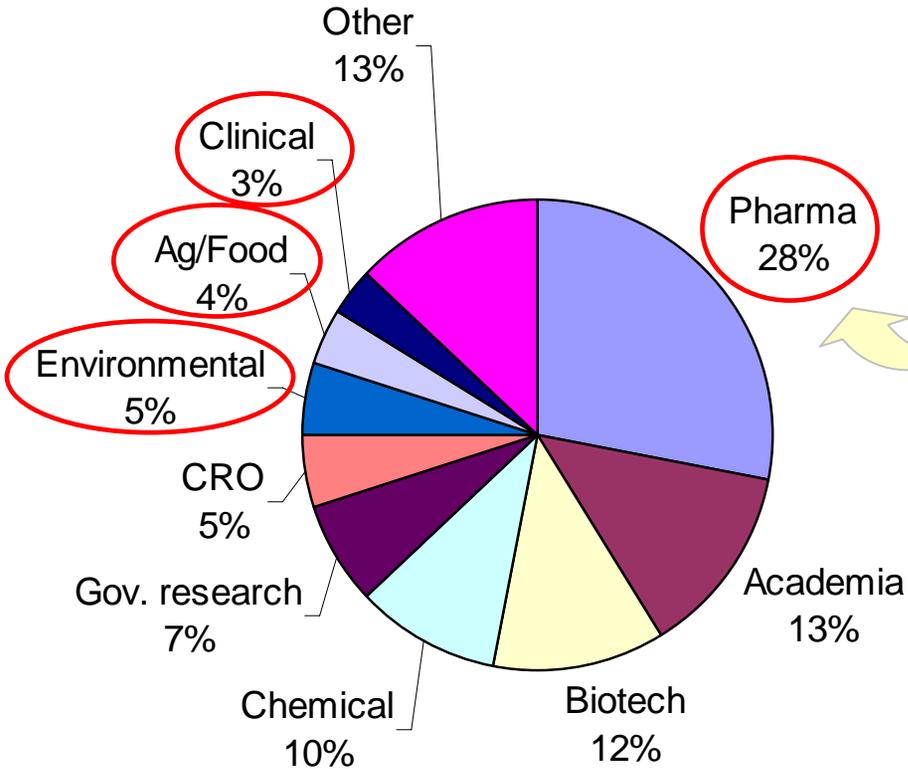
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# Liquid Chromatography Consumables Market by Industry

2007 HPLC Demand by Industry



Pharma is a major user of LC



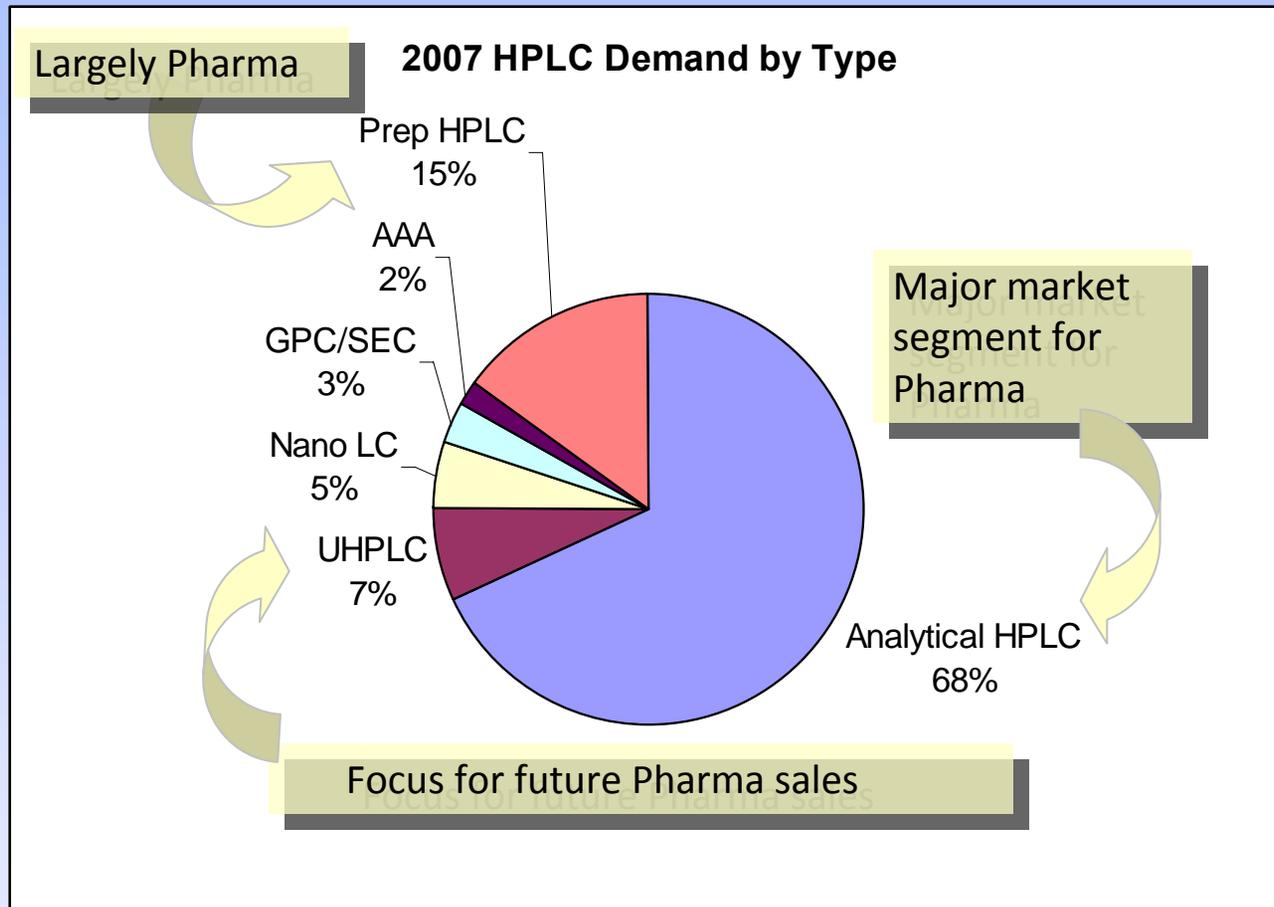
Opportunity to expand our industry reach and focus –

- Ag/Food
- Clinical
- Environmental

2007 SDI Market Forecast

\* Potentially inflated by currency translation

# Market Assessment – HPLC Demand



2007 SDI Market Forecast

# Technology Focus - Mass Spectrometry

SDI data on analytical instrument technology shows mass spectrometry is a **\$3B** market and predicts...

**Table V-2: Demand by Analytical Instruments Technology, 2005-2010**

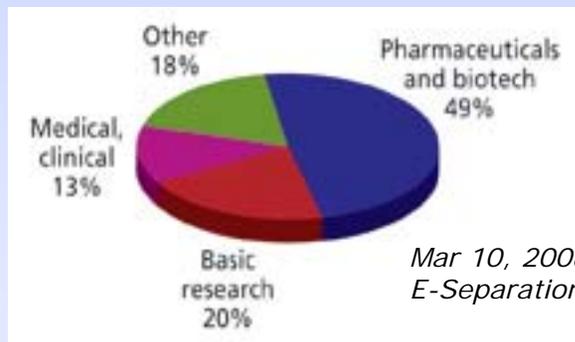
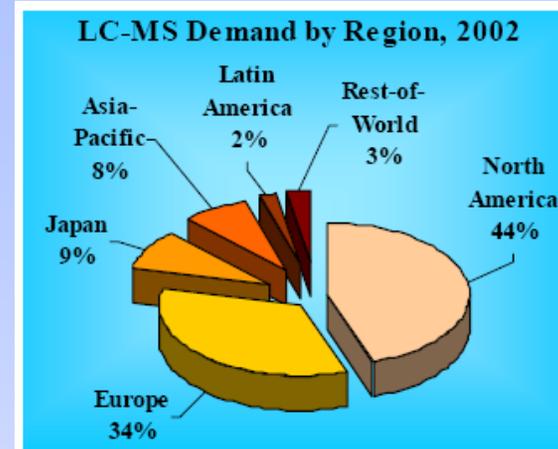
	2005		2006		2007		2010		'05-'10 CGR
	\$ Mil	Percent							
Separations	5,248	17%	5,529	17%	5,812	17%	6,666	16%	4.9%
Life Science Instrumentation	7,943	26%	8,529	26%	9,119	26%	10,815	26%	6.4%
Mass Spectrometry	1,809	6%	1,976	6%	2,146	6%	2,700	7%	8.3%
Molecular Spectroscopy	2,745	9%	2,908	9%	3,087	9%	3,700	9%	6.2%
Atomic Spectroscopy	2,207	7%	2,364	7%	2,495	7%	2,786	7%	4.8%
Surface Science	3,771	12%	4,019	12%	4,274	12%	5,198	13%	6.6%
Materials Characterization	1,803	6%	1,910	6%	2,023	6%	2,368	6%	5.6%
Laboratory Automation	2,669	9%	2,873	9%	3,082	9%	3,770	9%	7.2%
General Lab Instrumentation	2,610	8%	2,702	8%	2,798	8%	3,102	8%	3.5%
<b>Total</b>	<b>30,805</b>	<b>100%</b>	<b>32,810</b>	<b>100%</b>	<b>34,836</b>	<b>100%</b>	<b>41,106</b>	<b>100%</b>	<b>5.9%</b>

...that it will have fastest growth rate (8.3%) of any analytical instrument technology

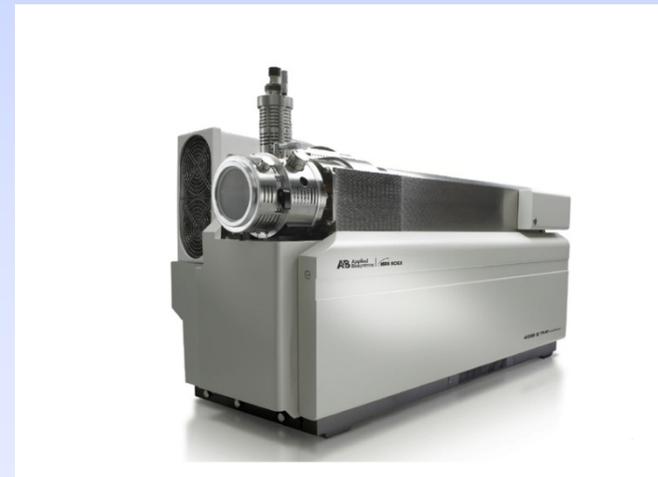
# Technology Focus - Mass Spectrometry

The use of Mass Spectrometry is creating industry growth opportunities

Industry	Percentage	Trending
Pharmaceutical and Biotech	49%	↓
Clinical	13%	↑↑
Basic Research	20%	↓
Environmental	18%	↑
Petroleum		
Forensics		
Food		



Mar 10, 2008  
E-Separation Solutions



*Focus product development and applications on MS*





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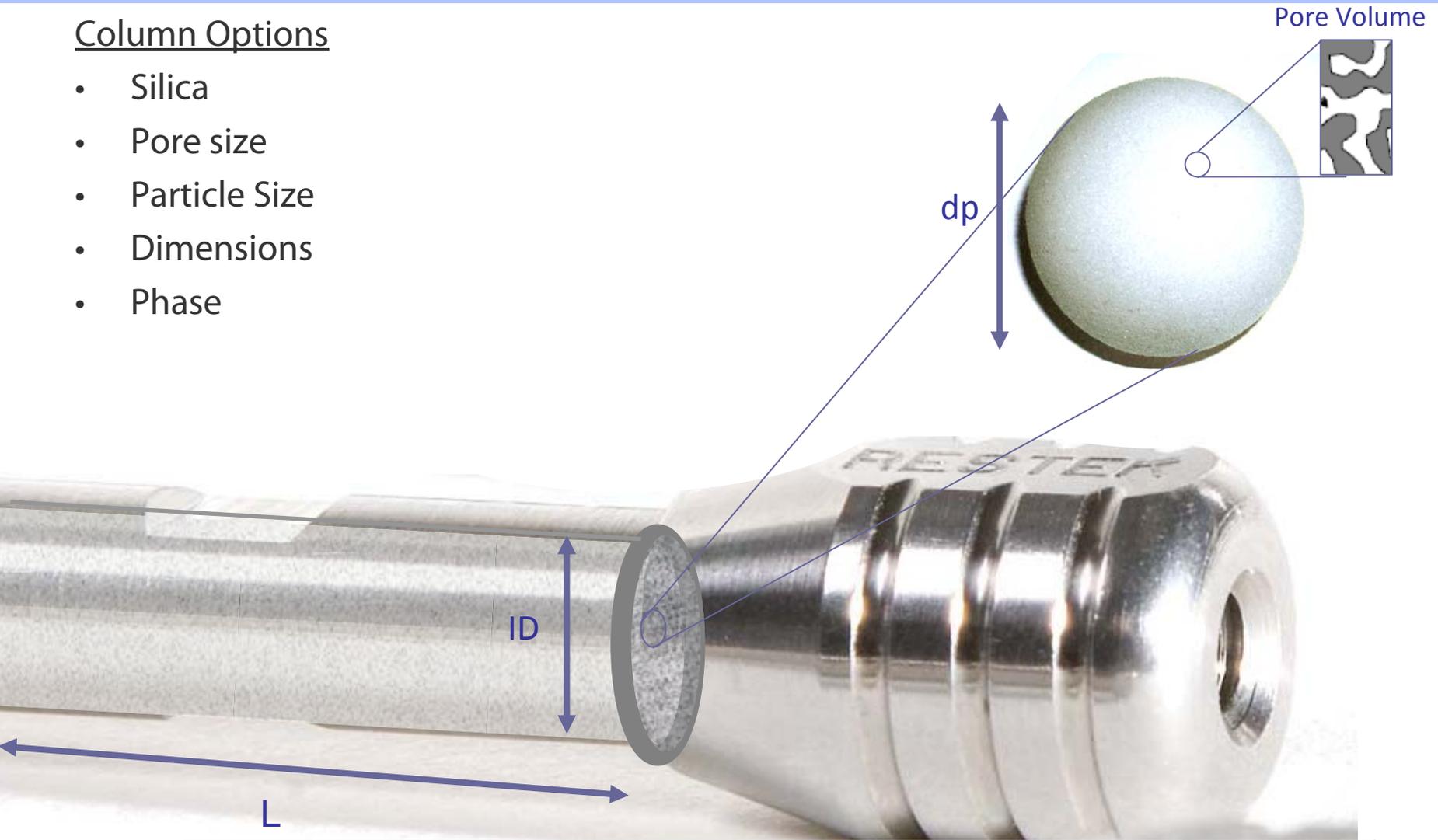
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# Determining Column Format

## Column Options

- Silica
- Pore size
- Particle Size
- Dimensions
- Phase



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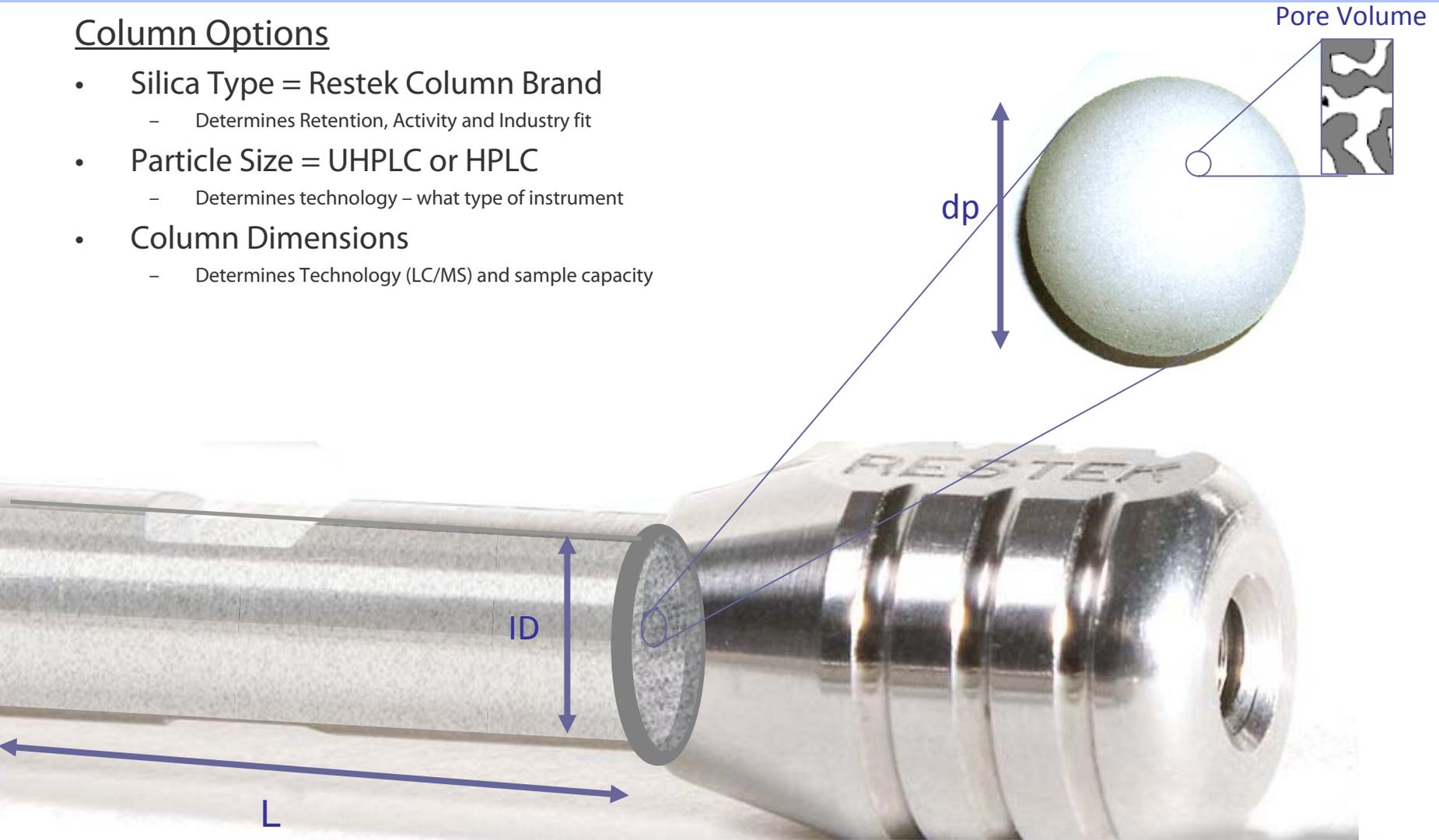
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# Determining Column Format

## Column Options

- Silica Type = Restek Column Brand
  - Determines Retention, Activity and Industry fit
- Particle Size = UHPLC or HPLC
  - Determines technology – what type of instrument
- Column Dimensions
  - Determines Technology (LC/MS) and sample capacity



# Restek High Purity LC Column Lines

Column Line	Pore Size (Å)	Surface Area (m <sup>2</sup> /g)	Carbon Load (%)	Usage
Allure	60	450	27	<u>Very high retention</u> (highest convectional C18 in market) High Purity Type B (super low metal content) 5 um particle size only (type B)
Ultra**	100	300	20	<u>High Retention</u> High Purity Type B (super low metal content) 3 and 5um particle size only
Pinnacle DB**	140	150	11	Mid- Retention Base Deactivated 1.9, 3 and 5um particle sizes <a href="#">UHPLC and HPLC</a>

Good recommendations for all markets, including pharmaceutical

# Restek LC Column Lines

Column Line	Pore Size (Å)	Surface Area (m <sup>2</sup> /g)	Carbon Load (%)	Usage
Pinnacle II	110	180	13	Mid – Retention Acidic Silica <b>(not for Reversed Phase in Pharma)</b> 3 and 5µm particle size only

Type A Economical Silica – NOT intended for sale in the pharmaceutical market

# Particle Size to Instrument Fit Chart

Manufacturer	Model	Recommended Particle Size (µm)	Recommended ID (mm)
Waters	Alliance	5 and 3	4.6 and 3.2
Waters	Acquity	1.9	2.1
Agilent	1100	5 and 3	4.6 and 3.2
Agilent	1200	5,3 (and 2.2)	4.6, 3.2 (and 3.0)
Agilent	1290	1.9	2.1
Shimadzu	Prominence	5 and 3	4.6 and 3.2
Shimadzu	UFLC XR	2.2	3.0
Shimadzu	Nexera	1.9	2.1
Jasco	XLC	1.9	2.1
Hitachi	La Chrom Elite	5 and 3	4.6 and 3.2
Hitachi	La Chrom Ultra	1.9	2.1
Dionex	Ultimate 3000	5 and 3	4.6 and 3.2
Dionex	Ultimate 3000 RS	1.9	2.1
Perkin Elmer	Flexar	1.9	2.1
Thermo	Accela	1.9	2.1

# Capillary Columns Available

Becoming popular for Bio Analytical and Bio Pharma applications

Phase	Pore Size (Å)	ID (mm)	Length (mm)
Ultra Aqueous C18	100	0.3	50
Ultra Aqueous C18	100	0.3	150
Ultra C18	100	0.3	50
Ultra C18	100	0.3	150
Viva C18	300	0.3	50
Viva C18	300	0.3	150



# Restek Reversed Phase Column Listing

Phase Type	Phase Type	Usage
C1 through C18	Conventional Alkyls	General Purpose Alkyl Phases
Aqueous C18 ★	"Aqueous"	<ul style="list-style-type: none"> <li>• <u>Balanced Retention</u> (retains the widest range of compounds)</li> <li>• 100% compatibility with aqueous mobile phases – no de-wetting</li> <li>• Great for drug discovery and LC/MS/MS C18</li> <li>• <u>Rugged</u></li> </ul>
AK ★	Combined Alkyl and Phenyl Properties	Intermediate C18 and Phenyl Selectivity
Phenyl	Conventional Phenyl	General purpose phenyl
Biphenyl ★	Advanced "special" Phenyl	<ul style="list-style-type: none"> <li>• <u>Higher retention and selectivity than phenyl</u></li> <li>• Great for aromatic or unsaturated compounds</li> <li>• Increased selectivity when compared to phenyl hexyl and diphenyl</li> </ul>
Aromax ★	Maximum Aromatic Selectivity	<ul style="list-style-type: none"> <li>• Specialty phenyl for maximum aromatic selectivity</li> <li>• High retention for LC/MS/MS in DMPK</li> </ul>

★ Unique

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# Restek Reversed Phase Column Listing

Phase Type	Phase Type	Usage
IBD ★	Polar Embedded	<ul style="list-style-type: none"> <li>•RP and HILIC Modes</li> <li>•Great for DMPK (retention of polar acids) and QA/QC (excellent peak shape of bases)</li> <li>•Lessens needs for mobile phase modifiers</li> </ul>
PFP	Fluorinated	<i>Not to be recommended over PFP Propyl unless PFP Propyl shows too much retention</i>
PFP Propyl	Fluorinated	<ul style="list-style-type: none"> <li>•RP and HILIC Modes</li> <li>•Great for DMPK (retention of protonated bases and halogenated compounds)</li> <li>•Typically more versatile than Cyano columns in pH range</li> <li>•Lessens need for mobile phase modifiers</li> </ul>
Amino	Amino	Traditional chemistry, highly used for the analysis of saccharides and sugars
Cyano	Cyano	Traditional column chemistry for the increased retention and selectivity of protonated bases

★ Unique

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# Multi Modal Analysis of 4-Methylimidazole (4MI or 4-MEI)

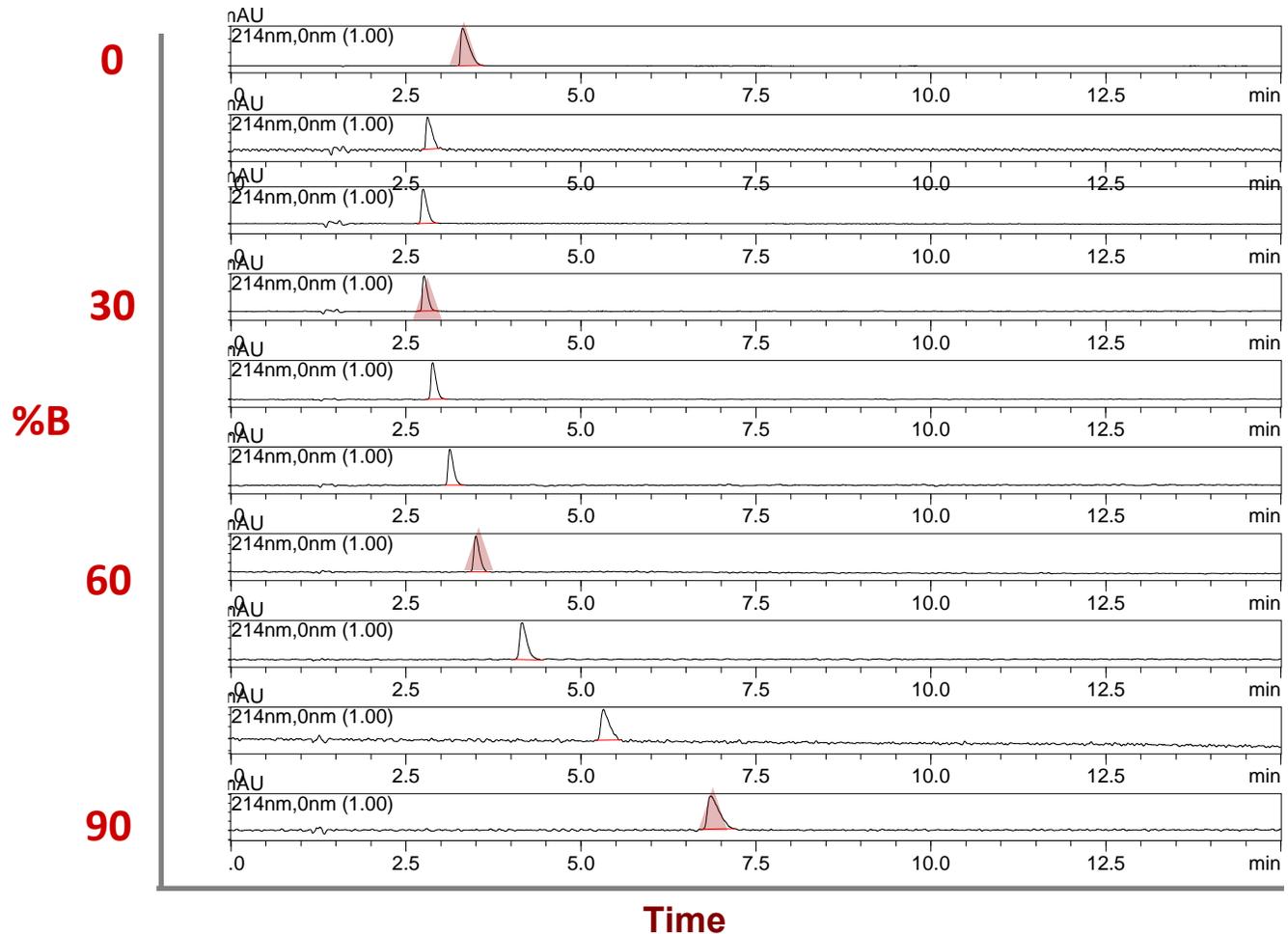
Ultra PFP Propyl  
4.6 x 150mm,  
5µm

1.5 mL/min,

30°C

A: 0.1% formic  
acid in water

B: 0.1% formic  
acid in Methanol



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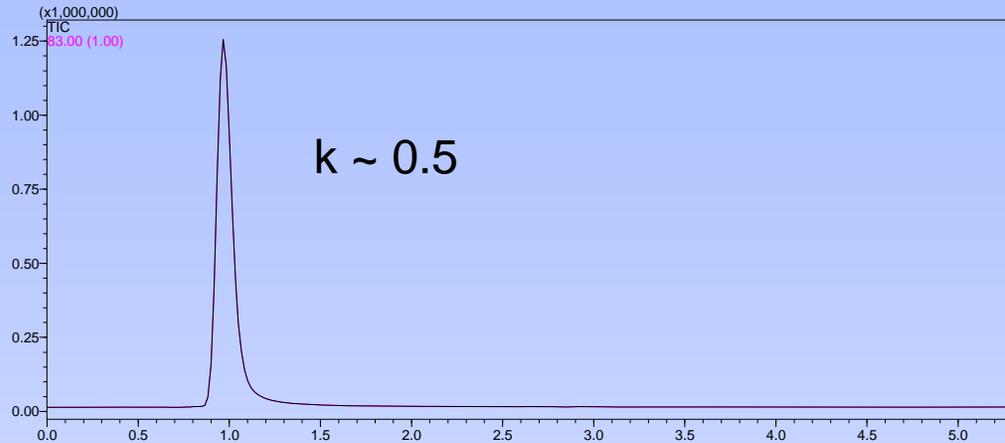
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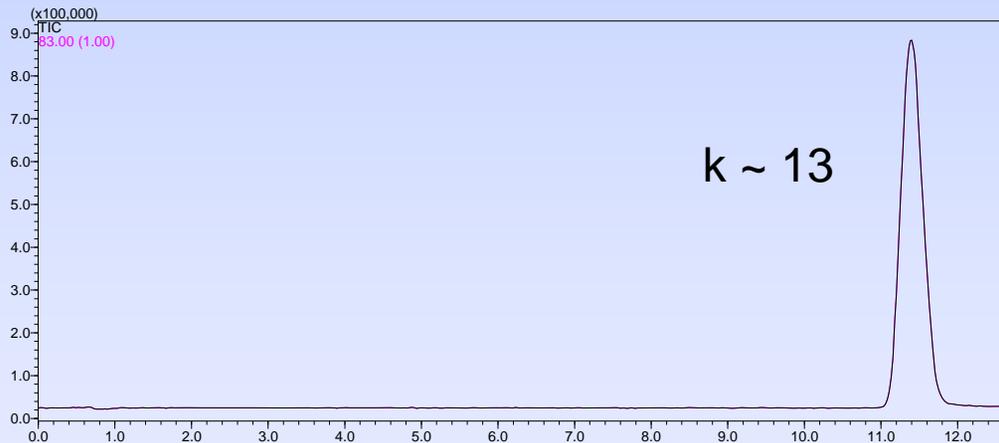
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# HILIC Retention

Ultra Silica  
2.1 x 100mm, 3 $\mu$ m  
0.4mL/min  
80% MeOH  
20% 10mM ammonium formate in water  
30°C  
1 $\mu$ L injection  
10 $\mu$ g/mL in 0.1% formic acid in water  
SIM m/z=83



Ultra PFPP  
2.1 x 100mm, 3 $\mu$ m  
0.4mL/min  
30% Water w/ 0.02% formic acid  
**70% MeOH** w/ 0.02% formic acid  
30°C  
1 $\mu$ L injection  
10 $\mu$ g/mL in 0.1% formic acid in water  
SIM m/z=83



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# Restek Normal Phase / HILIC Column Listing

Phase Type	Phase Type	Usage
IBD	Polar Embedded	<ul style="list-style-type: none"> <li>•Can be used on NPC and HILIC modes</li> <li>•Excellent selling point for DMPK (RP and HILIC modes)</li> </ul>
PFP	Fluorinated	<i>Not to recommended over PFP Propyl unless PFPP shows too much retention</i>
PFP Propyl	Fluorinated	<ul style="list-style-type: none"> <li>•Can be used on NPC and HILIC modes</li> <li>•Excellent selling point for DMPK (RP and HILIC modes)</li> </ul>
Amino	Amino	General purpose NPC / HILIC
Cyano	Cyano	General purpose NPC / HILIC
Silica	Silica	General purpose NPC / HILIC

# Restek UHPLC

## •Features

- Widest Variety of Stationary Phases
- Cost – Effective
- Improved Lifetime – innovative hardware design
- Restek Manufactured Silica – total control



## Actual E-mail Customer response :

*" Darrel,*

*Just wanted to let you know we retired one of your uplc columns today. The column had over 13,000 injections on it and still passed all system suitability requirement. Just getting a little close. Just thought I'd give you a shout. Feel free to use that as an example to any potential customers if you want*

*- Manager of Quality Control,  
Mylan Pharmaceuticals "*

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# Pinnacle DB UHPLC Columns

COLUMNS TESTED: UPLC® HSS T3 1.8 mm, 2.1/50 MM (Waters Corporation),  
Acquity UPLC® BEH C18 1.7 mm, 2.1/100 mm (Waters Corporation)  
 **Pinnacle DB C18 1.9 mm, 2.1/50 mm (Restek U.S.)**  
VisionHT C18-P 1.5 mm, 2.0/50 mm (Grace)  
Hypersil GOLD 1.9 mm, 2.1/50mm (Thermo Scientific)

using a validated (Bioanalytical method validation by FDA 2001) in GLP environment and OECD audited method.

Title of article is:

APPLICATION OF ULTRA PERFORMANCE COLUMNS IN HIGH PERFORMANCE LIQUID CHROMATOGRAPHY FOR ALBENDAZOLE AND ITS METABOLITES DETERMINATION IN TURKEYS

Tomasz Grabowski a, Jerzy Jan Jaroszewski b , Anna Świerczewska a, Renata Sawicka a, Tomasz Maślanka b, Włodzimierz Markiewicz b, Hubert Ziółkowski b

a Centre of Pharmacokinetics Research FILAB, Ravimed Sp. z o.o., Łajski, (Poland)

b Department of Pharmacology and Toxicology, Faculty of Veterinary Medicine, University of Warmia and Mazury, Olsztyn, (Poland)

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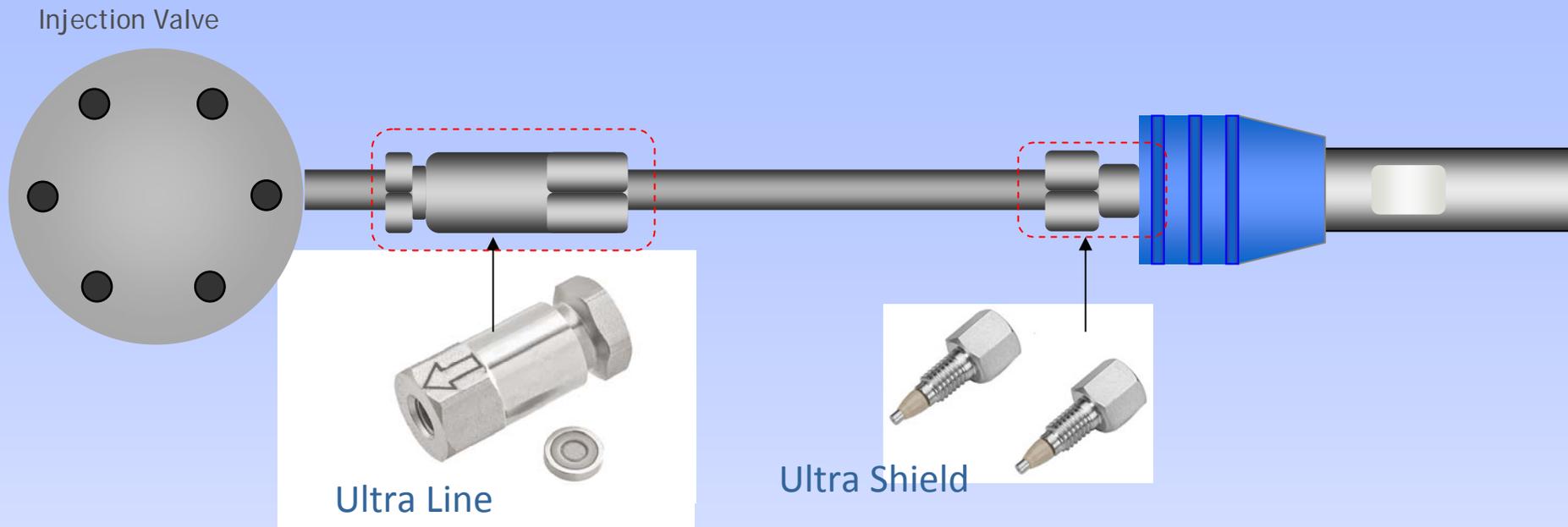
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# Proper Filtration for UHPLC



**Install 0.5um Ultra Line or  
0.5um Ultra Shield**

- Will not stop matrix
- Guard columns add volume and losses in efficiency
- 0.00625mm tubing compared to 0.175mm tubing for HPLC

## Product Focus



- **USLC is the use of *Selectivity* on any instrument platform**
- **USLC phases offer the widest variety of selectivity for effective and fast method development**
- **USLC phases define a simple approach to column selection**
- **USLC offers a simplified product offering for column recommendations**

# Restek Product Positioning



**Discover Restek USLC™**  
**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.

**This is independent of column line. It differentiates our phases, not our column lines**

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# Competitive Column Offerings

**Figure 3:** Restek offers the widest range of phase selectivities for HPLC and UHPLC.

Column Type	Column Line								
	Restek			Waters			Phenomenex	Agilent	
	Pinnacle DB	Ultra	Viva	Acquity CSH	Acquity HSS	Acquity BEH	Kinetex	Zorbax RRHD	Poroshell 120
Alkyl (C18 and C8)	●	●	●	●	●	●	●	●	●
Phenyl	●	●	●	●		●			
Polar Embedded Alkyl	●	●	●						
Fluorophenyl	●	●	●	●			●		

*We have them all!*

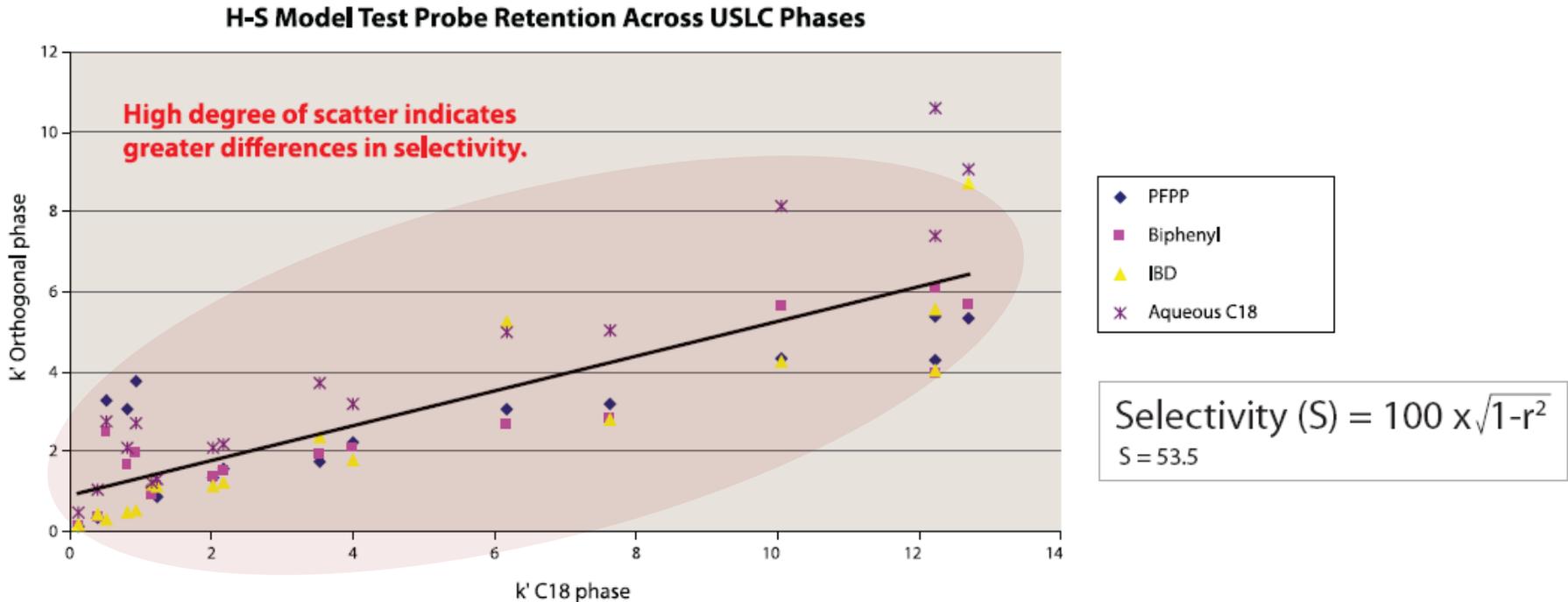
# Selectivity on any Instrument Format

**Figure 4:** Restek USLC™ phases are available on several silica lines to accommodate any instrument platform or chromatographic application.

Column line (silica)	Pinnacle DB (Base deactivated, moderate retention silica for scalable UHPLC and HPLC analyses)	Ultra (Highest purity silica for traditional HPLC analyses)	Viva (Wide pore silica for biological separations using HPLC)
Particle diameters (µm)			
• UHPLC	1.9		
• HPLC		3 and 5	5
<b>Stationary phases</b>			
C18	●	●	●
C8	●	●	●
Aqueous C18	●	●	
Biphenyl	●	●	●
PFP Propyl	●	●	●
IBD	●	●	
Silica	●	●	●

# The Value of Selectivity

## Correlation of Orthogonal Phase Selectivity Relative to C18

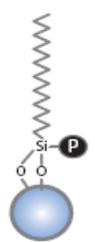
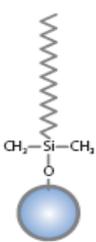
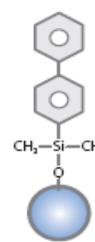
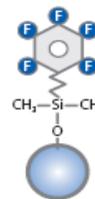


[2] U.D. Neue, J.E. O’Gara, A. Mendez, J. Chromatogr. A 1127 (2006) 161.

Restek currently has the largest range of selectivity in HPLC and UHPLC

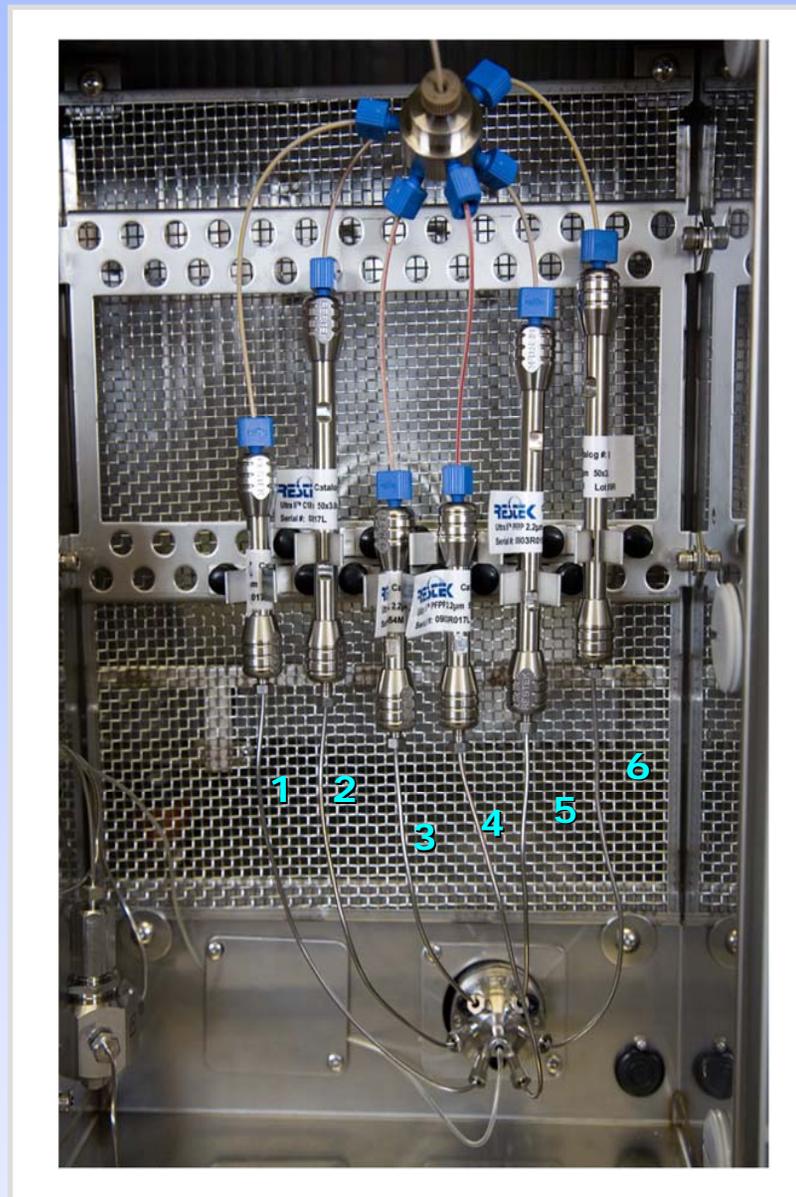
# USLC Phases have the highest range of selectivity for RPC

**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)	PPF Propyl (fluorophenyl)
	 <p>Aqueous C18</p>	 <p>C18</p>	 <p>Biphenyl</p>	 <p>PPF Propyl</p>
Ligand type	Proprietary polar modified and functionally bonded C18	Proprietary polar functional embedded alkyl	Unique Biphenyl	Proprietary end-capped pentafluorophenyl propyl
Characteristics and uses	<ul style="list-style-type: none"> <li>• C18 phase for balanced retention of multiple solute types.</li> <li>• Compatible with up to 100% aqueous mobile phases.</li> </ul>	<ul style="list-style-type: none"> <li>• Enhanced retention of polar acids.</li> <li>• Moderate retention of both acidic and basic solutes.</li> </ul>	<ul style="list-style-type: none"> <li>• Increased retention of aromatic, unsaturated, conjugated solutes, or solutes containing an electron withdrawing ring substituent.</li> <li>• Enhanced retention and selectivity when used with methanolic mobile phases.</li> </ul>	<ul style="list-style-type: none"> <li>• Increased retention of protonated bases and solutes containing aromatic moieties.</li> </ul>

# Great for Column Switching

- Automated Column Selection
- Attach multiple mobile and stationary phases to system and systematically scout gradients
- Fast and effective method development
- Most Effective with Widest Variety of Stationary Phases



*Courtesy of Shimadzu*

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# Biphenyl Advantage

Biphenyl can provide improved selectivity and retention over competitive and traditional phenyls as well as phenyl hexyls and diphenyls

★ **Biphenyl columns are more retentive for drug compounds than conventional phenyl and C18 phases, and are an excellent choice for LC/MS or the analysis of complex biological matrices.** ★

★ **The versatility of a Biphenyl column makes it an invaluable tool for the practicing method developer and a great addition to column screening systems.** ★

Biphenyl has been, and continues to be, very successful in the domestic market

## The Evolution of Resolution

**Figure 1** The Biphenyl phase provides higher retention of both hydrophobic and hydrophilic aromatic compounds and is capable of stronger pi-pi interactions than other phenyl phases.



### BIPHENYL

- Incorporates aryl linker to the surface silica
- Highest hydrophobic retention of all phenyl phases
- Highest aromatic selectivity of all phenyl phases



### PHENYL HEXYL

- Incorporates hexyl linker to the surface silica
- Increased hydrophobic retention
- Increased aromatic selectivity



### DIPHENYL

- High density phenyl stationary phase
- Higher hydrophobic retention than conventional phenyl phases
- Capable of more aromatic selectivity than conventional phenyl phases



### PHENYL PROPYL

- Conventional phenyl stationary phase
- Weak to moderate hydrophobic retention
- Capable of aromatic selectivity



### ODS (OCTADECYLSILANE) OR C18

- Most popular reversed phase column
- Strong hydrophobic retention (dispersive interactions)
- Not selective towards aromatic compounds

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# Viva - Wide-Pore Silica for Bio Pharma



**Viva Wide Pore HPLC Columns**  
Spectacular performance for peptides, proteins, or other large molecules.

[Find out more](#)

For the separation of compounds of MW greater than approximately 2000Da – Proteins and Bio Pharma

- Restek manufactured and tightly controlled for effective chromatography and reproducibility - **Accurate and precise pore volume**
- Wide Variety of stationary phases available
  - C4, C8, C18, Silica, **Biphenyl, PFP Propyl, IBD**

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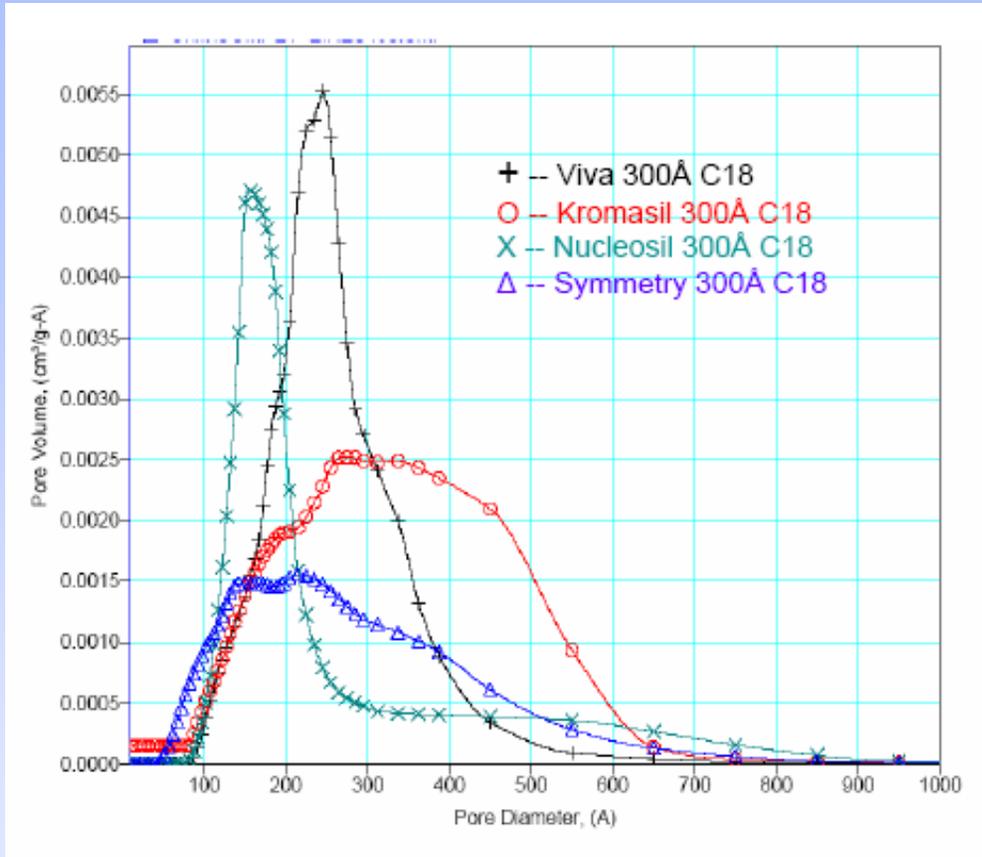
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# BJH Desorption $dV/dD$ Pore Volume



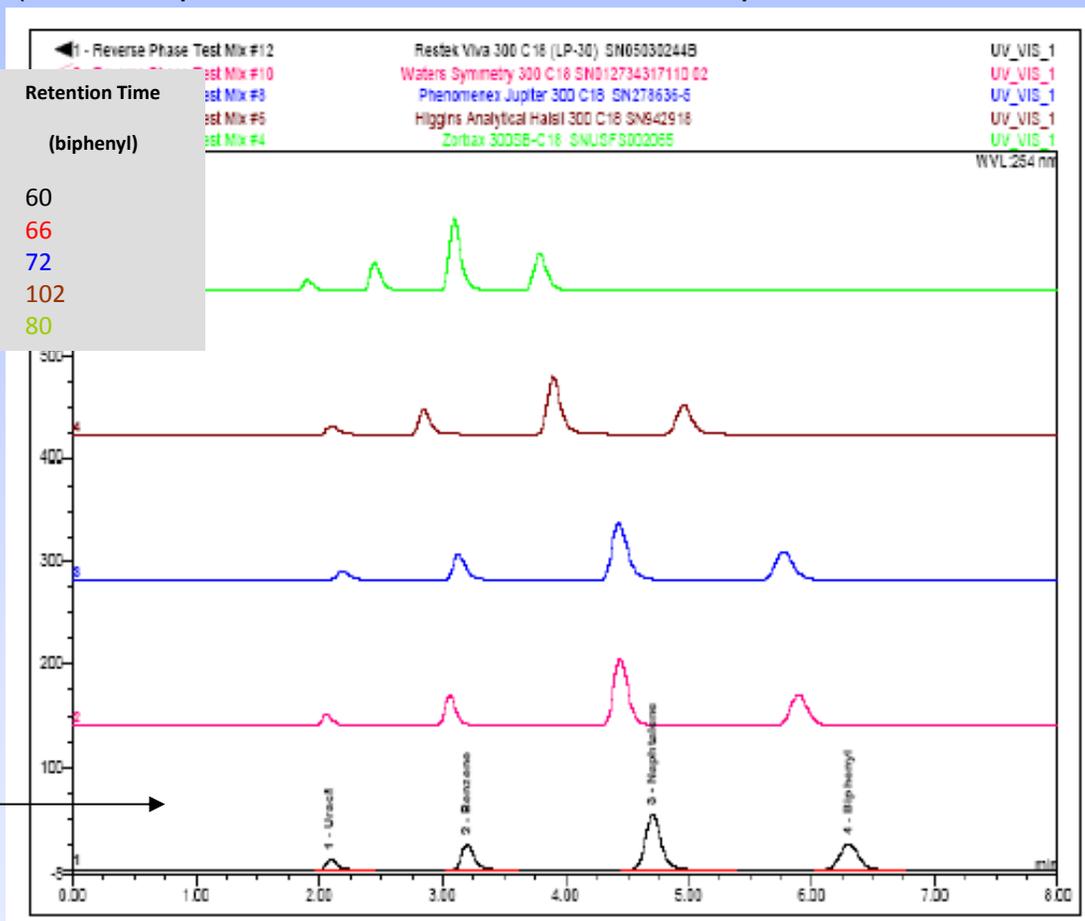
- Restek manufactured for control
- If they wont fit, they wont separate
  - Wide distribution means molecules may not fit

Only Viva™ silica has a narrow distribution with the majority of pores falling within the 250–350Å range.

# Viva Competitive Comparison

Viva™ wide pore C18 columns provide the best overall performance among five tested columns (reversed phase test mix; 150 x 2.1mm C18 phase columns, 5µm particles).

	Pressure (bar)	Efficiency (plates/meter)	Asymmetry (biphenyl)	Retention Time (biphenyl)
Viva™ 300 C18	52,146	1.16	6.30	60
Waters C18	57,326	1.30	5.89	66
Phenomenex C18	49,720	1.46	5.77	72
Higgins C18	51,813	1.46	4.96	102
Zorbax C18	41,666	1.49	3.79	80



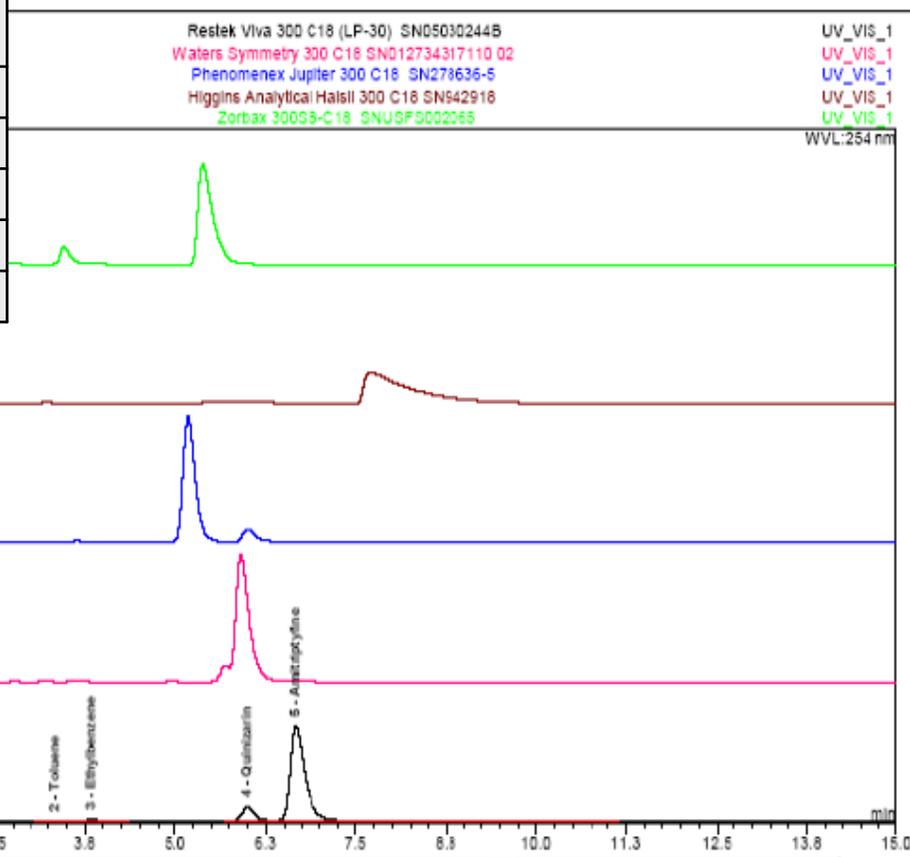
9514562) Column: Viva™ C18, 150 x 2.1mm, 5µm (cat.#  
 Mobile Phase: water:methanol, 25:75  
 Flow Rate: 0.2mL/min.  
 Detector: UV @ 254nm  
 Sample: 10µL Reversed Phase Test  
 Mix (cat.# 35005)

**Widest spread, most symmetrical peaks**

# Viva Competitive Comparison

Viva™ wide pore C18 columns provide low silanol activity and low metals activity

	Silanol Activity	Metal Activity	Embedded Polar Functionality
Viva 300 C18	Low	Low	No
Waters 300 C18	Low	Low	No
Phenomenex 300 C18	Low	Low	Yes
Higgins 300 C18	High	High	No
Zorbax 300 C18	Low	Low	No



Column: Viva™ C18, 150 x 2.1mm, 5µm  
 (cat.# 9514562)  
 Mobile Phase: A=20% 5mmol K<sub>x</sub>H<sub>y</sub>PO<sub>4</sub>, pH = 7.0  
 B=80% Methanol  
 Flow Rate: 0.2ml/min.  
 Detector: UV, 254nm  
 Injection: 1ml SRM870 test mix

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# Viva Competitive Comparison

Analysis of a four-protein test mix shows the superior performance of the Viva™ C18 column

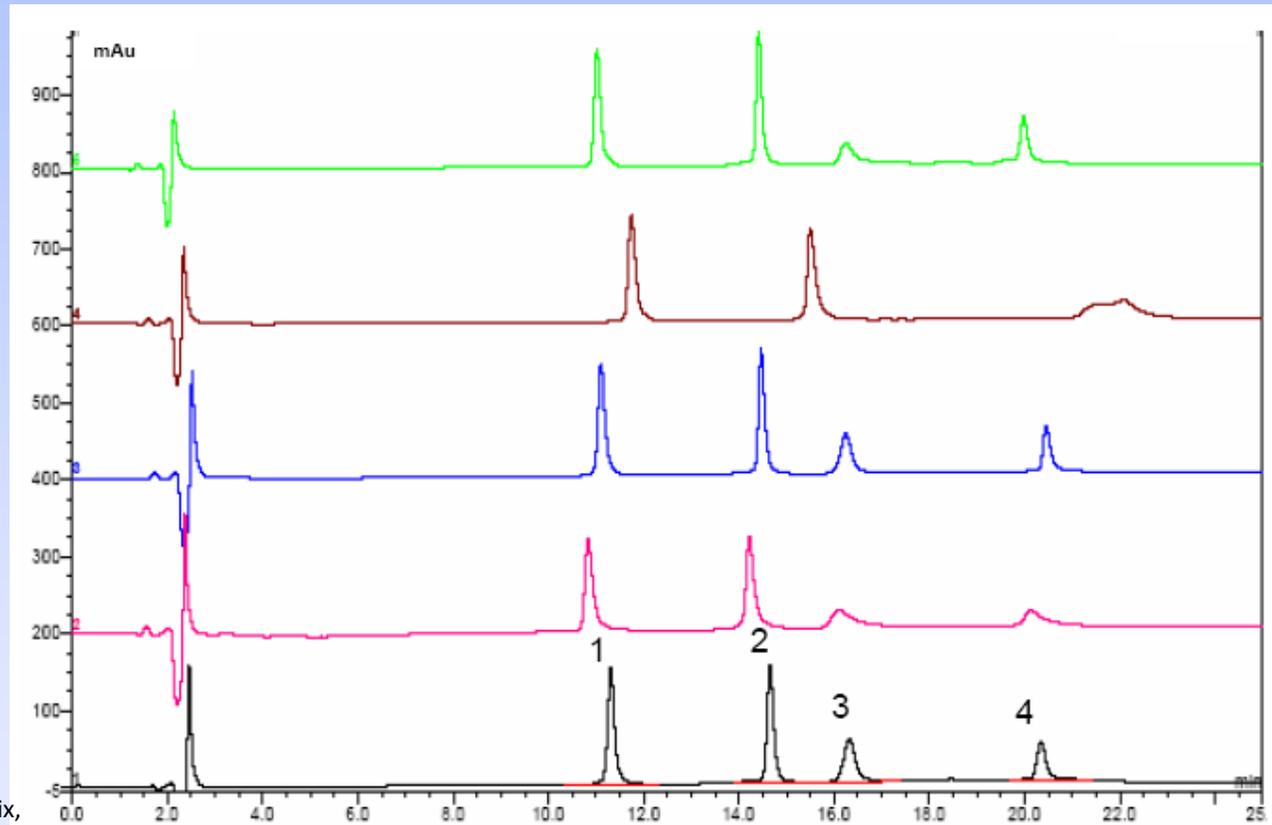
Waters Symmetry 300 C18,  
150mm x 2.1mm, 5µm

Phenomenex Jupiter 300 C18,  
150mm x 2.1mm, 5µm

Higgins Analytical Haisil C18,  
150mm x 2.1mm, 5µm

Zorbax 300SB-C18,  
150mm x 2.1mm, 5µm

Column: Viva™ C18, 150 x 2.1mm, 5µm  
(cat.# 9514562)  
Mobile Phase: A: 0.1% TFA in water, (pH 2.0)  
B: 0.1% TFA in acetonitrile  
20% B to 70% B in 30 min.  
Flow Rate: 0.2mL/min.  
Detector: UV @ 214nm  
Injection: Four Protein Standard Test Mix,  
Sigma Aldrich #H2899



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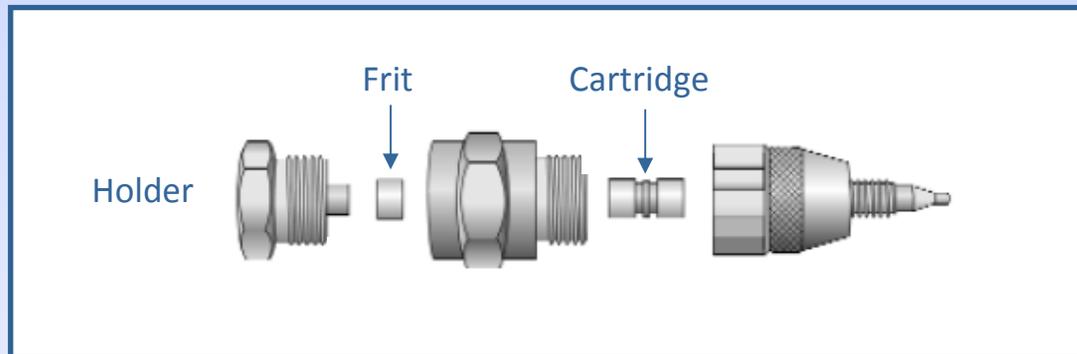
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# Guard Columns - a necessity

- Usually same packing material as analytical column
- Inexpensive
- Sample compatibility
- Low sample dispersion
- Adequate capacity
- Minimal loss of efficiency



# Guard Column Options



Trident Direct Guard Cartridge System



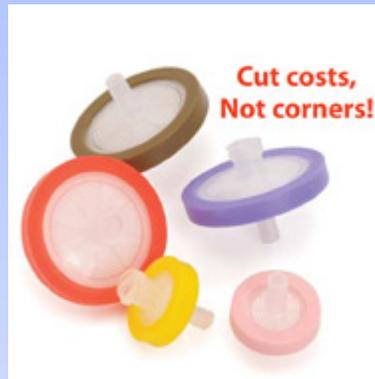
Restek's Exclusive Trident Integral System



Trident HPLC In-Line Guard Cartridge Holders

# Other LC interesting products

Individual product line promotions that can be easily applied for cross-selling opportunities



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# GC in Pharmaceutical Market

- Organic volatile impurities
  - Widely used to verify contaminants in solvents used to make pharmaceutical products
- Restek developed new column phase
  - Rxi-624Sil MS
- Column selectivity has been carefully tuned to give the best performance for analysis of solvents



# 3-IN-1 TECHNOLOGY

Highest Inertness • Lowest Bleed • Exceptional Reproducibility

- Literature

- Promotional Brochures
  - Rxi Flyer – GNFL1173
  - Rxi-5Sil MS – GNFL1061
  - Rxi-624Sil MS – PHFL1245
  - USP <467> - PHFL1018A
- Application Notes and Technical Articles
  - Outsourcing Solvent Methods – PHTS1262
  - USP <467> - On-line
  - How Column Inertness Improves...PHAN1259
  - PGI Analysis – On-Line

[http://www.restek.com/aoi\\_pharm\\_main.asp](http://www.restek.com/aoi_pharm_main.asp)

- Webinars

Practical Method Development for gas Chromatography

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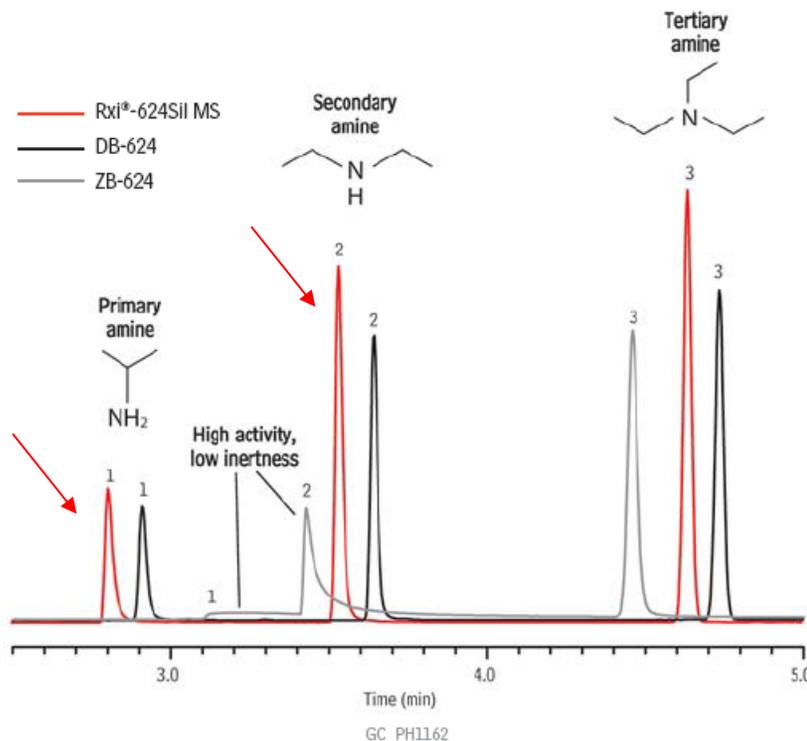
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# Balanced Inertness

## Balanced Inertness Gives Higher Data Quality— Excellent Peak Symmetry and Reproducibility for Active Compounds

**Figure 2** Innovative Rxi<sup>®</sup> deactivation technology provides excellent peak symmetry at 5 ng on-column for primary, secondary, and tertiary amines, which is not possible on competitive columns.



*Highly inert Rxi<sup>®</sup>-624Sil MS columns give excellent peak symmetry.*

Peaks	Conc. (µg/mL)
1. Isopropylamine	100
2. Diethylamine	100
3. Triethylamine	100

<b>Column</b>	30 m, 0.32 mm ID, 1.8 µm (cat.# 13870)
<b>Sample</b>	Isopropylamine
<b>Diluent:</b>	DMSO
<b>Conc.:</b>	100 µg/mL
<b>Injection</b>	
<b>Inj. Vol.:</b>	1 µL split (split ratio 20:1)
<b>Liner:</b>	5mm Single Gooseneck with Wool (cat.# 22973-200.1)
<b>Inj. Temp.:</b>	250 °C
<b>Oven</b>	
<b>Oven Temp:</b>	50 °C (hold 1 min.) to 200 °C at 20 °C/min. (hold 5 min.)
<b>Carrier Gas</b>	He, constant flow
<b>Linear Velocity:</b>	37 cm/sec.
<b>Detector</b>	FID @ 250 °C
<b>Instrument</b>	Agilent/HP6890 GC

# Balanced Inertness -

## Balanced Inertness Gives Higher Data Quality— Excellent Peak Symmetry and Reproducibility for Active Compounds

**Figure 3** Primary amines, such as isopropylamine, can be more accurately integrated on an Rxi<sup>®</sup>-624Sil MS column, lowering levels of quantification (LOQs) and increasing method accuracy.

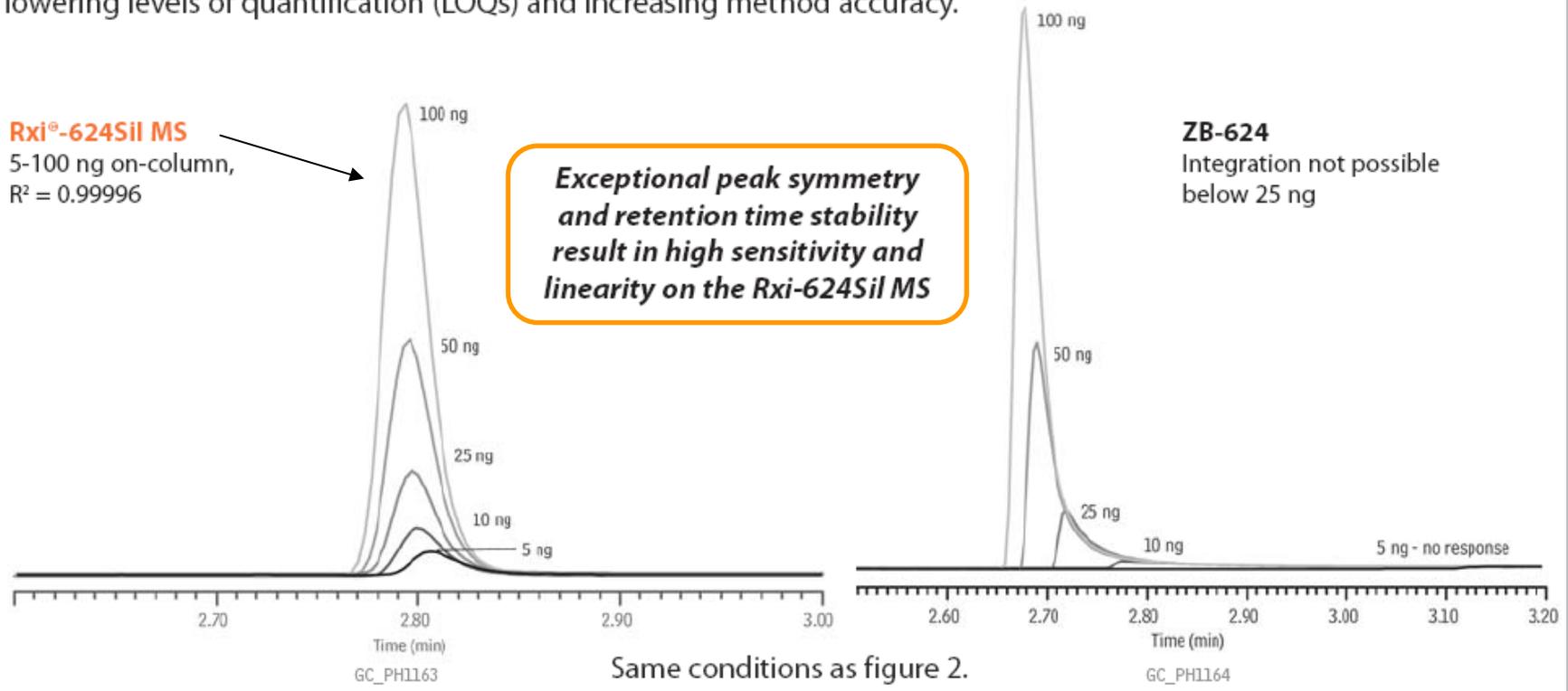
**Rxi<sup>®</sup>-624Sil MS**

5-100 ng on-column,  
 $R^2 = 0.99996$

*Exceptional peak symmetry  
and retention time stability  
result in high sensitivity and  
linearity on the Rxi-624Sil MS*

**ZB-624**

Integration not possible  
below 25 ng



# Highest Thermal Stability of its Class

## High Thermal Stability and Low Bleed for GC/MS Compatibility

**Table I** The Rxi®-624Sil MS column has the highest thermal stability of any 624 column.

Column	Manufacturer	Highest Temperature Limit (Isothermal)
Rxi®-624Sil MS	Restek	320 °C
VF-624ms	Varian	300 °C
DB-624	Agilent J&W	260 °C
ZB-624	Phenomenex	260 °C

Data obtained from company website or literature for a 30 m x 0.25 mm x 1.4 µm df column.



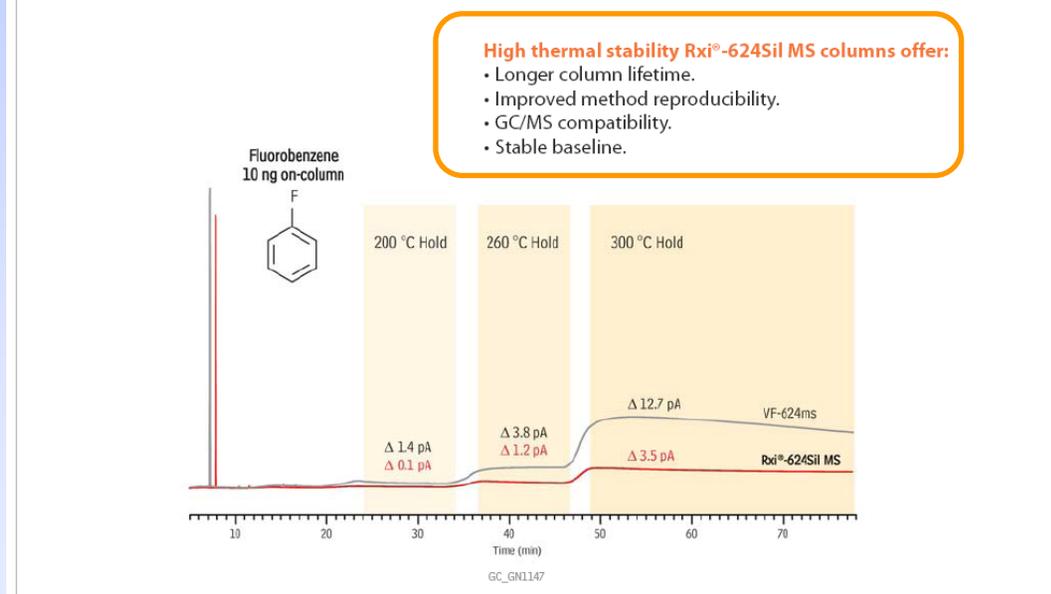
- Low Bleed, high thermal stability polymer

- MS compatibility – no need to change columns when converting FID to MS

- Stable baselines for consistent results

- Longer column lifetimes

**Figure 5** The Rxi®-624Sil MS column has the lowest bleed of any column in its class, providing true GC/MS capability.



# Rxi Essentials for Pharma Use



Pharmaceutical

Go To...the Right Column First

## NEW! Rxi®-624Sil MS

The **Go To** GC Column for Fast, Effective Volatile Impurities Method Development

**Go To...** the right column first

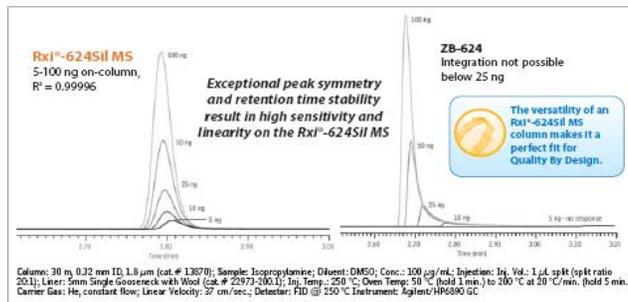
For better retention of polar analytes and improved accuracy, peak shape, and response for active compounds.

**Go To...** mass spec directly

With the lowest bleed 624 column available; stable up to 320 °C, for easy transfer of methods to GC/MS.

**Go To...** the next batch faster

With the best-in-class G43 for USP methods.



### Rxi®-624Sil MS Columns (fused silica)

- Low bleed, high thermal stability column—maximum temperatures up to 320°C.
- Inert—excellent peak shape for a wide range of compounds, including acidic and basic compounds.
- Selective—highly selective for residual solvents, great choice for USP<467>.
- Manufactured for column-to-column reproducibility—well-suited for validated methods.

(Inid polarity Crossbond® silylene phase; equivalent to 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	d <sub>f</sub> (µm)	Temp. Limits	20-Meter	30-Meter	60-Meter
0.18mm	1.00	-20 to 300/320°C	13865	\$405	
0.25mm	1.40	-20 to 300/320°C		13868	\$500
0.32mm	1.80	-20 to 300/320°C		13870	\$540
0.53mm	3.00	-20 to 280/300°C		13871	\$615

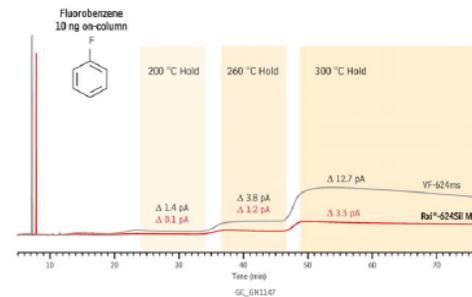


## Not all "624s" are Equivalent

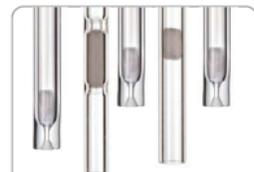
High thermal stability Rxi®-624Sil MS columns offer:

- Longer column lifetime.
- Improved method reproducibility.
- GC/MS compatibility.
- Stable baseline.

Column	Manufacturer	Highest Temperature Limit (Isotherm)
Rxi®-624Sil MS	Restek	320 °C
VF-624ms	Varian	300 °C
DB-624	Agilent J&W	260 °C
ZB-624	Phenomenex	260 °C



Ideal for use with FID and MS detectors.



Couple the right column with the right liner. Visit [www.restek.com/liners](http://www.restek.com/liners) for a complete selection.



## Custom Residual Solvent Mixes

A perfect match for validated residual solvent methods

Save time and money with mixes prepared to your specific solvent set and concentrations. The more you buy the less you pay per ampul!

Easy Online Order Form!  
[www.restek.com/customusp](http://www.restek.com/customusp)



Make the Switch to Rxi® columns!

Experience what Rxi® did for many others

- Lower detection limits
- Better peak shape
- Accurate results

[www.restek.com/rxi](http://www.restek.com/rxi)

# Rxi Updated Brochure

Lit. Cat.# PHFL1018A

## Residual Solvent Analysis

Implementing USP <467>

- Satisfy USP <467> requirements—detailed procedures inside.
- Improve system suitability pass rates with an optimized system.
- Easy technical tips ensure successful implementation.

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Updated brochure for the USP <467>

This is the technical flyer meant to assist customers in implementing the method

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# USP <467> Standards



## USP-equivalent Standards

### Residual Solvents - Class 1

benzene	10mg/mL	1,1-dichloroethene	40
carbon tetrachloride	20	1,1,1-trichloroethane	50
1,2-dichloroethane	25		

In dimethyl sulfoxide, 1mL/ampul  
cat. # 36279 (ea.) \$30

### Residual Solvents Class 2 - Mix A

acetonitrile	2.05mg/mL	methylcyclohexane	5.90
chlorobenzene	1.80	methylene chloride	3.00
cyclohexane	19.40	tetrahydrofuran	3.45
<i>cis</i> -1,2-dichloroethene	4.70	toluene	4.45
<i>trans</i> -1,2-dichloroethene	4.70	<i>m</i> -xylene	6.51
1,4-dioxane	1.90	<i>o</i> -xylene	0.98
ethylbenzene	1.84	<i>p</i> -xylene	1.52
methanol	15.00		

In dimethyl sulfoxide, 1mL/ampul  
cat. # 36271 (ea.) \$30

### Residual Solvents Class 2 - Mix B

chloroform	60µg/mL	nitromethane	50
1,2-dimethoxyethane	100	pyridine	200
<i>n</i> -hexane (C6)	290	tetralin	100
2-hexanone	50	trichloroethene	80

In dimethyl sulfoxide, 1mL/ampul  
cat. # 36280 (ea.) \$30

### Residual Solvents Class 2 - Mix C

2-ethoxyethanol	800µg/mL	2-methoxyethanol (methyl	
ethylene glycol	3,100	Cellulosolve)	250
formamide	1,100	N-methylpyrrolidone	2,650
N,N-dimethylacetamide	5,450	sulfolane	800
N,N-dimethylformamide	4,400		

In dimethyl sulfoxide, 1mL/ampul  
cat. # 36273 (ea.) \$30

### USP <467> Singles

Volume is 1mL/ampul.

Compound	Solvent	Conc.	cat.# (ea.)	price
acetonitrile	DMSO	2.05mg/mL	36281	\$26
benzene	DMSO	10mg/mL	36282	\$26
carbon tetrachloride	DMSO	20mg/mL	36283	\$26
chlorobenzene	DMSO	1.8mg/mL	36284	\$26
chloroform	DMSO	0.3mg/mL	36285	\$26
cyclohexane	DMSO	19.4mg/mL	36286	\$26
1,1-dichloroethene	DMSO	40mg/mL	36287	\$26
1,2-dichloroethane	DMSO	25mg/mL	36288	\$26
<i>cis</i> -1,2-dichloroethene	DMSO	4.67mg/mL	36289	\$26
<i>trans</i> -1,2-dichloroethene	DMSO	4.67mg/mL	36290	\$26
1,2-dimethoxyethane	DMSO	0.5mg/mL	36291	\$26
N,N-dimethylacetamide	DMSO	5.45mg/mL	36292	\$26
N,N-dimethylformamide	DMSO	4.4mg/mL	36293	\$26
1,4-dioxane	DMSO	1.9mg/mL	36294	\$26
2-ethoxyethanol	DMSO	0.8mg/mL	36295	\$26
ethylbenzene	DMSO	1.84mg/mL	36296	\$26
ethylene glycol	DMSO	3.1mg/mL	36297	\$26
formamide	DMSO	1.1mg/mL	36298	\$26
hexane	DMSO	1.45mg/mL	36299	\$26
methanol	DMSO	15mg/mL	36401	\$26
2-methoxyethanol	DMSO	0.25mg/mL	36402	\$26
methylbutylketone	DMSO	0.25mg/mL	36400	\$26
methylcyclohexane	DMSO	5.9mg/mL	36403	\$26
methylene chloride (dichloromethane)	DMSO	3mg/mL	36404	\$26
N-methylpyrrolidone	DMSO	2.65mg/mL	36405	\$26
nitromethane	DMSO	0.25mg/mL	36406	\$26
pyridine	DMSO	1mg/mL	36407	\$26
sulfolane	DMSO	0.8mg/mL	36413	\$26
tetrahydrofuran (THF)	DMSO	3.6mg/mL	36408	\$26
tetralin	DMSO	0.5mg/mL	36409	\$26
toluene	DMSO	4.45mg/mL	36410	\$26
1,1,1-trichloroethane	DMSO	50mg/mL	36411	\$26
trichloroethene	DMSO	0.4mg/mL	36412	\$26
<i>m</i> -xylene	DMSO	6.51mg/mL	36414	\$26
<i>o</i> -xylene	DMSO	0.97mg/mL	36415	\$26
<i>p</i> -xylene	DMSO	1.52mg/mL	36416	\$26

DMSO=dimethyl sulfoxide

# USP < 467 > Standards



## Custom Standards

Restek is your #1 source for custom reference standards!

- **Save time!** Let us prepare your standards, blended precisely to your specifications.
- **Save money!** Custom mixes for your processes; don't pay for solvents you don't need.
- **Great value!** Volume discounts on fully-qualified standards.

Order form available on-line

### BIG SAVINGS

## Custom Residual Solvents Mixes

- **Save time!** Let us prepare your standards, blended precisely to your specifications.
- **Save money!** Custom mixes for your processes; don't pay for solvents you don't need.
- **Great value!** Volume discounts on fully-qualified standards.

**Buy More, Save More!**  
Restek Custom Residual Solvent Mixes are a great way to save money and time in the laboratory. You can buy exactly what you need—mixed to your specifications—and not pay for components you don't need.

*Savings vary based on the size and how much you purchase. Here's an example custom order and the associated savings relative to list price.*

**Example Order**  
This customer saved 94% off list price!

Solvent	Cat #	Conc. (vol/vol)
Acetone	47-04-1	0.01
Acetonitrile	75-01-8	0.01
2-Butanone (MEK)	78-09-3	0.01
Cyclohexane	109-62-7	0.01
Ethanol	44-05-2	0.01
Ethyl acetate	141-78-4	0.01
Methanol	65-50-1	0.01
n-Butyl-2-pyrrolidone (NMP)	106-10-1	0.01
Methyl tert-butyl ether (MTBE)	1634-04-4	0.01
Methylene chloride	75-09-2	0.01
2-Methyltetrahydrofuran	36-47-9	0.01
2-Propanol	67-63-0	0.01
Tetrahydrofuran	209-99-3	0.01
Toluene	108-88-3	0.01

Number of Amps/Bottle	Price/List Price	Year Save
5	\$170	49%
10	\$110	69%
25	\$40	77%
100	\$12	90%
500	\$1	94%
1000	\$0	94%

**Buy more, SAVE MORE!**

Order today at [www.restek.com/customusp](http://www.restek.com/customusp) or use the convenient form on the reverse.

### Custom Solvent Mix Order Form

**Send**  **Print**  **Download**  
 **PDF**  **HTML**  **Excel**

Name: \_\_\_\_\_ Title: \_\_\_\_\_  
 Company: \_\_\_\_\_  
 Address: \_\_\_\_\_  
 Phone: \_\_\_\_\_ Fax: \_\_\_\_\_  
 E-mail: \_\_\_\_\_

We'll bill you:  Annual Volume (AV)  Number of Amps  
 Preparer's name: \_\_\_\_\_

Select contents and concentrations. Note that not all residual solvents are compatible. Our chemists will review your order and advise if separate orders are recommended or required.	Concentration (vol/vol)	Select	Cat #	Concentration (vol/vol)
Acetone	0.01	Acetone	47-04-1	0.01
Acetonitrile	0.01	Acetonitrile	75-01-8	0.01
2-Butanone (MEK)	0.01	2-Butanone (MEK)	78-09-3	0.01
Cyclohexane	0.01	Cyclohexane	109-62-7	0.01
Ethanol	0.01	Ethanol	44-05-2	0.01
Ethyl acetate	0.01	Ethyl acetate	141-78-4	0.01
Methanol	0.01	Methanol	65-50-1	0.01
n-Butyl-2-pyrrolidone (NMP)	0.01	n-Butyl-2-pyrrolidone (NMP)	106-10-1	0.01
Methyl tert-butyl ether (MTBE)	0.01	Methyl tert-butyl ether (MTBE)	1634-04-4	0.01
Methylene chloride	0.01	Methylene chloride	75-09-2	0.01
2-Methyltetrahydrofuran	0.01	2-Methyltetrahydrofuran	36-47-9	0.01
2-Propanol	0.01	2-Propanol	67-63-0	0.01
Tetrahydrofuran	0.01	Tetrahydrofuran	209-99-3	0.01
Toluene	0.01	Toluene	108-88-3	0.01

Please complete a separate form for each mix to expedite processing.

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Lit. Cat.# PHTS1212

# GC Column Mix for Pharma Method Development

Rxi-5Sil MS



Rxi-17Sil MS



Rxi-624Sil MS



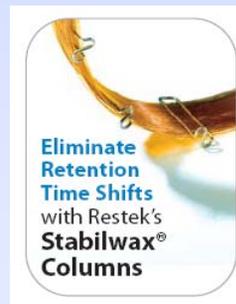
Stabilwax



Phase:	Non-polar	—————→		Polar
Thermal Stability:	High (360)	350	320	Low (260)
Inst. Compat.	GC/MS			GC/FID only
Deactivation:	Balanced Amine (follow with same deactivation for liners)			DA vs DB option

Four GC column phases recommended for organic volatile impurities

- Wide variation in selectivity
- Highly thermally stable columns
- Very inert
- Reproducible from column to column



# Summary

- HPLC
  - USLC
    - Ultra Selective Liquid Chromatography
  - Focus on:
    - Ultra (Type B silica, more inert)
      - 3um and 5um particles
    - Pinnacle DB (Type A silica)
      - 1.9um particles
    - Viva (large molecules, Proteins and Bio Pharma )
- GC
  - Organic volatile impurities
    - Rxi-624Sil MS
    - Best selectivity choices
      - Rxi-5Sil MS, Rxi-17Sil MS, Rxi-624Sil MS, Stabilwax

# Petrochemical APPLICATIONS

## - 2011 Restek Overview

Market Focus:

### Petrochemical

Gary Stidsen  
Jaap de Zeeuw

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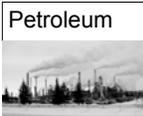
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### Who are the End-users

**Petroleum**  
  
Shell, ESSO, bp, ExxonMobil, Syncrude, Schlumberger, IP, HAZOPROM

**Chemicals**  
  
DOW, BASF, WACKER, EASTMAN, eif arochem, ATO, Ineos

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### Products analyzed in Segment

**Petroleum**  
  
Oil, Natural gas, gasoline, kerosene, diesel, Naphtha  
Hydrocarbons, Sulfur, Biomarkers, Aromatics, Alcohols  
Bio-Fuels, Biodiesel, Bioethanol  
glycerides, FAME, alcohols

**Chemicals**  
  
Intermediates and final product  
C2-C5 Alkenes, Aromatics, Amines, Glycols, Acids, Solvents, Halogenated compounds, Alcohols, Aldehydes

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- MXT-1HT Sim Dist
- MXT-2887
- Rtx-DHA
- D3606 packed column set
- Rt-XL Sulfur packed column
  - And Siltek deactivation for sulfurs
- PLOT columns
- Appendix
  - Products for biodiesel and chemical analysis

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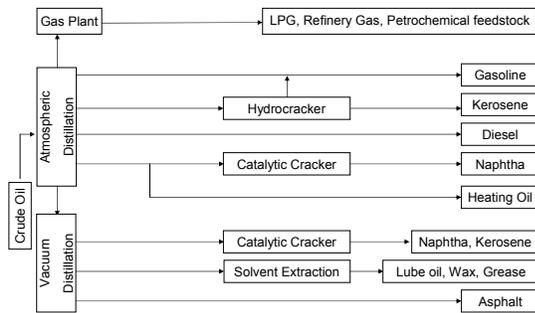
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### Refinery Process Map



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### What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene /Toluene
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions does Restek offer?

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### Simulated Distillation

The chromatogram displays a series of peaks labeled A<sub>1</sub> through A<sub>10</sub> across a retention time range from 1 to 10. The y-axis is labeled 'Response' and the x-axis is labeled 'Retention time'.

- The chromatogram of the sample is divided into time slices
- The software determines the area count for each slice

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### Challenges

- Columns Break (fused silica)
  - Need to recalibrate often (time)
  - Replace columns (price per analysis, time)
  - Reduced accuracy (reliability challenge)
- Columns are not efficient
  - Do not meet resolution requirements

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### Needs for Accurate Simulated Distillation Analysis

- High temperature stable columns
- Low Bleed phases

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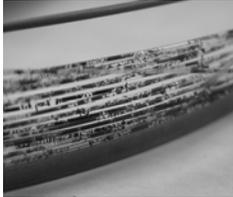
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### Sim Dist with fused silica: Big Problem area..

- FS Standard fused silica column polyimide coated, type Inferno..



Polyimide outside coating seriously damaged

This will happen with ALL fused silica columns



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### Optimize strength/robustness of capillary Application of MXT Metal capillary tubing



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### MXT Tubing is Extremely Strong



- Virtually unbreakable
  - Stainless steel with Siltek coated surface
- Can be bent / coiled to small radius
- Siltek surface stabilizes the stationary phase
- Sim Dist columns wound on standard 11-pin cage
- MXT columns can be cut with standard scoring wafer
  - Special tools also available

MXT columns provide reliability:  
No column breakage possible!



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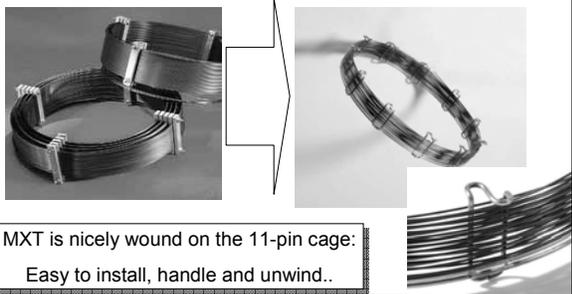
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## New, easy accessible column cage



MXT is nicely wound on the 11-pin cage:  
Easy to install, handle and unwind..

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## Simulated Distillation of Crude oil

Analysis: Simulated Distillation

C5-C110 D6352 High Temp

C5-C44 D2887

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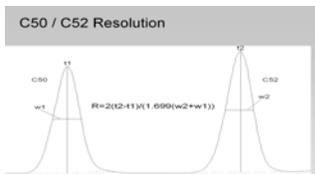
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## ASTM Spec for High Temperature Sim Dist

- In order to be approved for ASTM D86 and 6352, 7169 and 7500:
  - Resolution between C50 and C52 > 2.0, running under ASTM conditions



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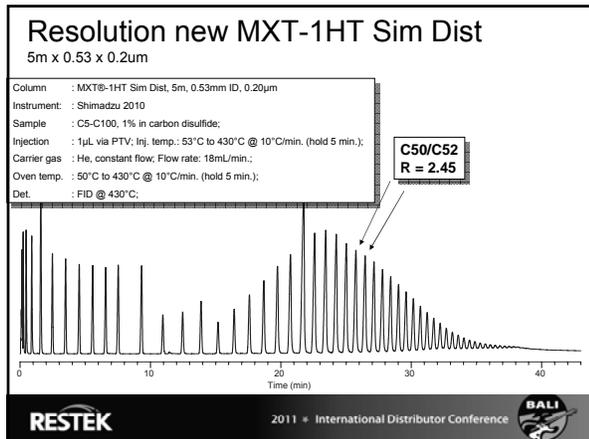
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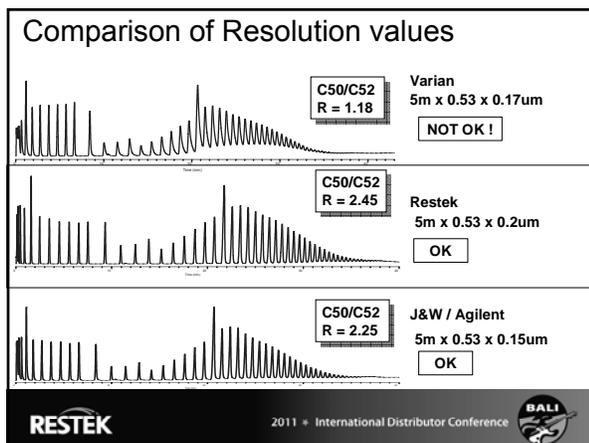
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### Resolution values

The higher resolution value indicates the Restek MXT-1HT Sim Dist:

- Has a smoother coated surface of the PDMS phase over the length of the column
- The higher resolution value allows for a longer column lifetime

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## Column Stability / Lifetime

- Column lifetime in Sim Dist applications are also in direct proportional with the level of the column bleed
- The LOWER the bleed, The LONGER the life time

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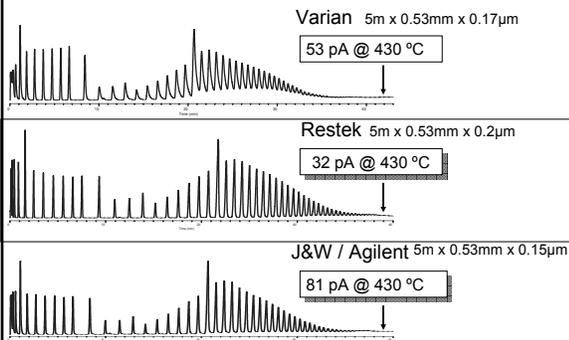
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## Comparison of Bleed values



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## Performance-ASTM D7169

- Customer feedback on Beta testing, done by Joaquin Lubkowitz, Separation systems.
- Authority in petro – analysis field and ASTM

 Separation Systems, Inc.  
Experts In Gas Chromatography

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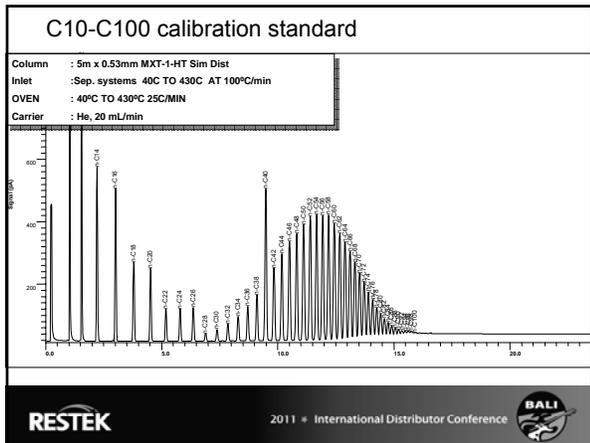
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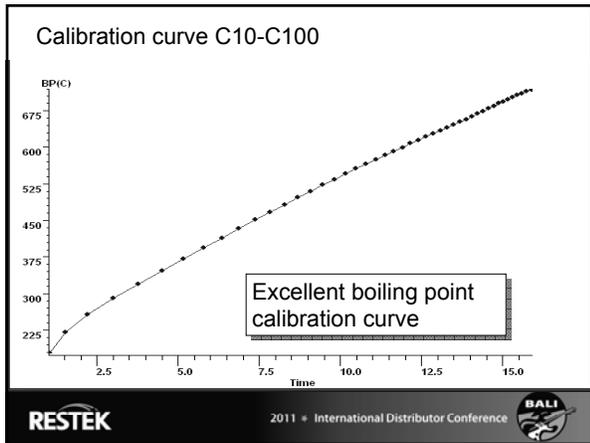
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## Column Cutting

For installation in a PTV or on-column injector, the column inlet must be well-cut..

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## MXT Column Cutting

- A special tube "scorer" has been developed..



Makes a perfect "cut" required for  
"on-Column injection"..  
For 0.25 - 0.53mm MXT



tubing scorer : 20523  
replacement wheel: 20522

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Carrier gas considerations  
Avoid introduction of oxidizers

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Impact of Oxygen/Moisture

- Increase of bleed
- Retention times drop fast
- Peaks will broaden
- Peaks will tail

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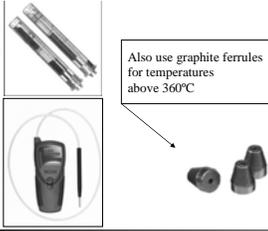
### Carrier Gas purification

- Degradation reaction of stationary phase + water/oxygen develops exponentially with higher temperature..

Need: Pure gases 

Need: Elimination of leaks 

Also use graphite ferrules for temperatures above 360°C



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### Simulated Distillation of Gasoline Range Hydrocarbons

Analysis: Simulated Distillation

C5-C110 D6352 High Temp

C5-C44 D2887

Also Called "gasoline-range" Sim Dist

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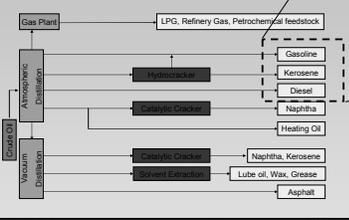
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### D2887 Used for:

**Refinery Process Map**



Gasoline

Kerosene

Diesel

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## Gasoline Range Sim Dist

- C5-C44 D2887
- Column: 10m x 0.53mm x 2.65µm Rtx-2887 (Fused silica or MXT)

Uses a THICK film because the sample is injected on the column with no dilution;  
Need LOADABILITY !!

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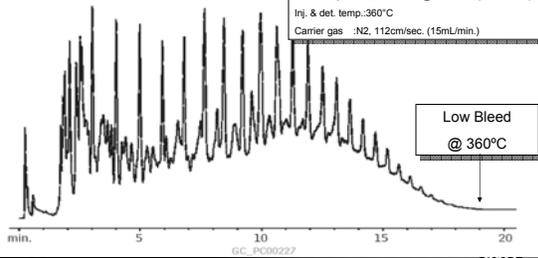
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## D-2887 Sim Dist

Column :10m, 0.53mm ID, 2.65µm Rtx®-2887  
Injection :Direct injection, 1.0µL of a 0.1 to 0.01 wt % hydrocarbon standard in carbon disulfide.  
Oven temp. :35°C to 360°C @15°C/min. (hold 5 min.)  
Inj. & det. temp.:360°C  
Carrier gas :N<sub>2</sub>, 112cm/sec. (15mL/min.)



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## Restek New Generation MXT Sim Dist Columns

- Features: Low bleeding, Temperature Stable, Better Efficiency
- Benefit: Longer Column Lifetime, Less Replacement, less calibrations, Saving \$\$\$\$\$

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### Product information for MXT Sim Dist

- Sim Dist sales companion + presentation
- Restek Refined brochure - PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Simdist brochure - PCFL1201A: <http://www.restek.com/restek/images/external/PCFL1201A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp>; and [http://www.restek.com/info\\_chromatograms.asp](http://www.restek.com/info_chromatograms.asp)
- Technical Literature: [http://www.restek.com/aoi\\_lit\\_main.asp](http://www.restek.com/aoi_lit_main.asp)
- Article: **Tighten Control of Distillation Processes with the New MXT®-1HT Sim Dist Column :** [http://www.restek.com/aoi\\_petro\\_A022.asp](http://www.restek.com/aoi_petro_A022.asp)



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### What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene /Toluene
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions does Restek offer?

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### What products come from most refineries?

- Gasoline
- Diesel
- Jet Fuel
- Refinery Gas

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### Analysis: Gasoline

#### Detailed Hydrocarbon Analysis (DHA) D6730

- Column: 100m x 0.25mm x 0.50um Rtx-DHA 100
- Tuning column: 5m x 0.25mm x 1.0um Rtx-5
- Tuning column is for adjusting selectivity for aromatics



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### Biggest Challenges for Customers Running ASTM D6730

- Have good peak shape for alcohols
- Have sufficient theoretical plates to separate the complex samples
- Coupling of "tuning" capillary
- Analysis time too long, wish for faster methods
  
- Reproducibility of column-column quality parameters



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### Why do the Refiners Run DHA Analysis?

- At the end a certain gasoline must have a certain octane number..
- It is very important for the refiner to monitor the blending process carefully to avoid too much yield.



Different octane numbers are available

- Accurate DHA analysis can save a lot of money ..



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## Important Quality Parameter for DHA Analysis

- The column must not only elute hydrocarbons
- Also need to elute **alcohols**



This is a big challenge and that's why Rtx-DHA 100 columns are preferred

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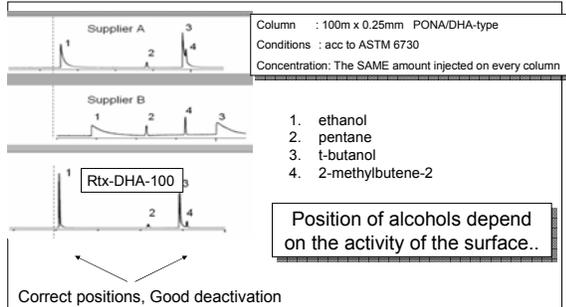
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## Elution of Alcohols on the DHA-type Columns ASTM Method D6730



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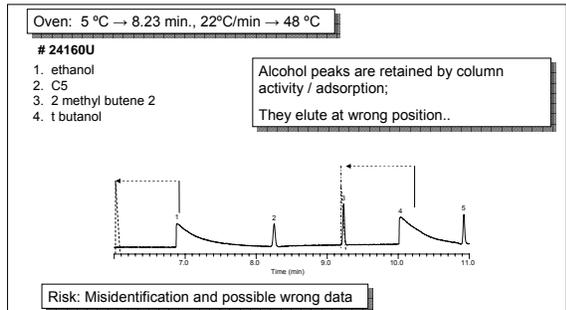
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## Impact of a Tailing Alcohol Peak

Supplier 1 type DHA column



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## Reproducibility of Rtx-DHA-100 Columns

Following graphs show the typical performance of the chronological production of 250 Rtx-DHA 100 columns

- Efficiency /Resolution
  - Retention
  - Inertness
- } Set by ASTM
- Bleed

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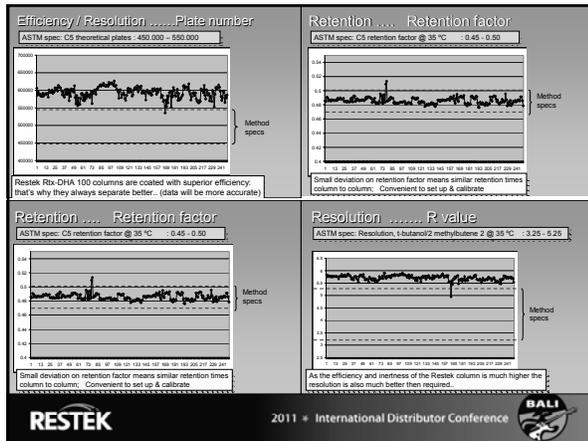
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## ASTM D 6730 Requires a "tuned" Column...

ASTM wants general purpose methods  
Columns from different suppliers will show small differences in selectivity..  
By coupling a short piece of Rtx-5, every column can be "tuned" to fulfill ASTM specs on selectivity..

Tuning pre-column: Rtx-5, 2-5 meters  
Length to be determined empirically

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There will be always a wish from customers to  
**SPEED UP** the analysis

Using Helium and different temperature program

Using Hydrogen

Using shorter, smaller ID columns

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### Using Helium and a different program

Using the same column, same GC

Column : 100m x 0.25mm Rtx-DHA-100, df = 0.5  $\mu$ m coupled with  
2.6m x 0.25mm Rtx-5DHA tuning column, df = 1.0  $\mu$ m

- Carrier gas : He, Flow 2.3 mL/min (Constant Flow), 28 cm/s
- Oven temp. : 5 °C → 15min., 5°C/min → 50°C, 50min.  
8°C/min → 200°C, 10 min

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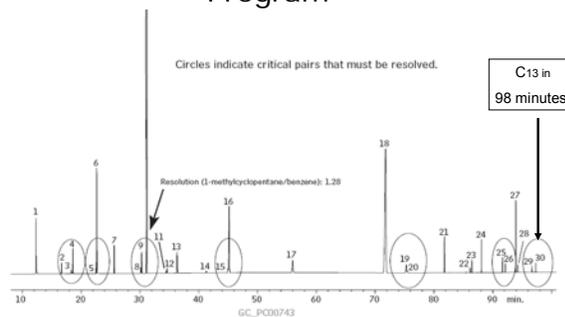
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### Using Helium and a Different Program



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## Speeding up the Application using HYDROGEN Carrier Gas

- Using hydrogen we can operate at near 2 times the linear velocity as used for helium
  - This results in significant shorter run time
- To get the same chromatogram/separation, we need to adjust the oven temperature program

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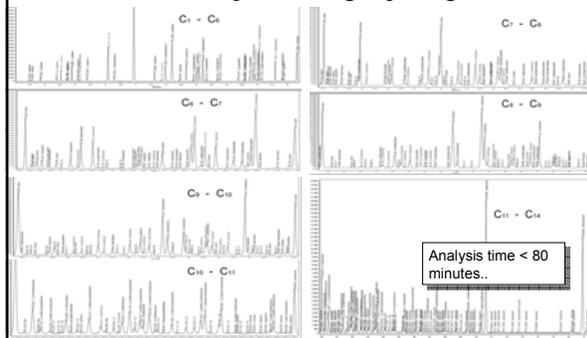
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## Fast DHA Analysis using Hydrogen...



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## Fast DHA using Hydrogen

- Same GC
- Same method
- Same Injection/detection techniques
- Same column
- No issues with overloading, peak shifts, tailing, high inlet pressures, discrimination and reduced life time..



Neil Johansen

All retention data and identification is generated by Neil Johansen, developer of the D6730 DHA method.

Available on the Restek website  
: [http://www.restek.com/promo\\_gc\\_pona.asp#004](http://www.restek.com/promo_gc_pona.asp#004)

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Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel: 03 9762 2034 . . . in AUSTRALIA

## Hydrogen : Safety Issues

Need to make sure there is no accumulation of H2 possible in the oven..

Hydrogen is combustible over a concentration range of 4% to 74% by volume;  
Diffusion VERY fast;

- Use of Hydrogen generators: limited amount of hydrogen
- Hydrogen Flow restriction to set with GC inlet (electronic flow setting)
- Hydrogen detection systems (sniffer)

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## Rtx-DHA Columns for Other ASTM Methods

For ASTM D6733 : Rtx-DHA-50 50m x 0.25mm, df = 0.5 µm

For ASTM D6729 : Rtx-DHA-100 100m x 0.25mm, df = 0.5 µm

For ASTM D5501 : Rtx-DHA-150 150m x 0.25mm, df = 1.0 µm

### NOTE:

This column was called earlier Rtx-1 PONA, which is a WRONG name as the application is DHA, not PONA.

Competing products are still called: HP-PONA, DB-PONA, CP-Sil PONA CB, BP-1PONA and Petrocol

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## Biggest Challenges for customers running ASTM D6730

Have good peak shape for alcohols.....



Have sufficient theoretical plates .....



Coupling of "tuning" capillary .....



Analysis time too long, wish for faster methods ..



Reproducibility of column quality parameters .....



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## Customer referral

I just wanted to relay to you how pleased I am with the performance of the Restek DHA PONA bonded to the 5 meter RTX-5 pre-column. We've found that the optimal tuning length is 2.05 meters  $\pm$  0.5 meters, which means less time finding the optimal length as compared to the prior Supelco columns.

The peaks look great – far superior to the prior supplier. I am especially pleased with the critical separation of t-butanol and 2-methylbutene-2. The t-butanol peak only tails minimally with the temperature programming, and it keeps us from having to scrap failed columns. This improvement process of switching columns has improved our production rate by nearly half and cut our waste costs as well.

Terry Osenbach  
PerkinElmer Health Science  
GC Application and Engineered Solutions  
Chemist  
710 Bridgeport Ave, Shelton, CT 06614

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## Product information Rtx DHA columns

- Rtx-DHA sales companion + presentation
- Restek Refined brochure PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>  
Brochure Rtx-DHA 100 PCFL1007A: <http://www.restek.com/restek/images/external/PCFL1007A.pdf>
- Recorded WEBINAR
- Applications see: <http://www.restek.com/restek/class/5004088.asp>; and [http://www.restek.com/info\\_chromatograms.asp](http://www.restek.com/info_chromatograms.asp)
- Technical Literature: [http://www.restek.com/aoi\\_lit\\_main.asp](http://www.restek.com/aoi_lit_main.asp)
- Article: **Faster DHA Analyses Using Helium or Hydrogen:** [http://www.restek.com/aoi\\_petro\\_A025.asp](http://www.restek.com/aoi_petro_A025.asp)
- Article: **How Good is Your PONA Column?:** [http://www.restek.com/aoi\\_petro\\_A006.asp](http://www.restek.com/aoi_petro_A006.asp)



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## What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions  
does Restek offer?

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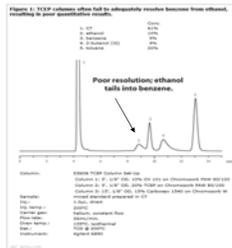
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Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel: 03 9762 2034 . . . in AUSTRALIA

## Benzene / Toluene/ Ethanol in Gasoline

ASTM Method D3606



Present solution is problematic:

Big issues with separations and peak shapes

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## Modified ASTM D3606 Method using the D3606 Column Set

Uses a SET of packed columns:

Material	Siltek deactivated SS
ID / OD	2mm / 1/8"
Lengths	6' and 16' long
Phases	Rtx-1 and proprietary

- First part of eluting peaks is sent to the second, highly selective column, where the alcohols and aromatics are separated
- Higher boiling compounds are back-flushed
- Unique column set, only from Restek

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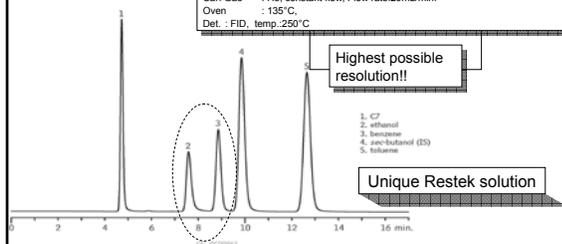
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## New D3606 Method

Columns :RT™-D3606 Column Set (cat.# 83606)  
 column 1 : RT™-D3606-1: 6x 1/8"OD (1.8m x 2mm ID), nonpolar Rtx®-1  
 column 2 : RT™-D3606-2: 16x 1/8"OD (4.9m x 2mm ID), proprietary packing  
 Injection : 0.05µL, direct injection; Inj. temp.: 200°C  
 Car. Gas : He, constant flow; Flow rate: 20mL/min.  
 Oven : 135°C  
 Det. : FID, temp.: 250°C



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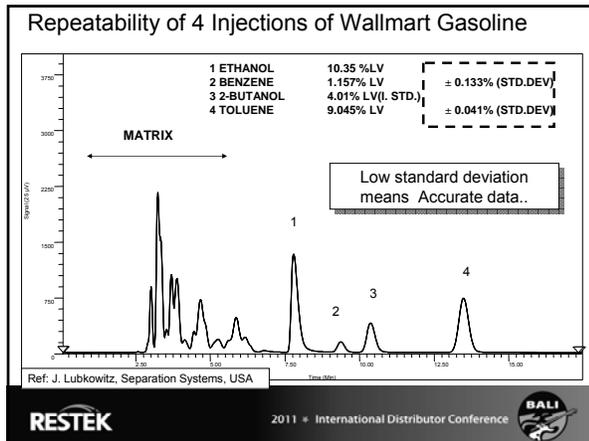
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### Product Information for D3606

- Restek Refined brochure: PCFL1195A; <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Applications see : <http://www.restek.com/restek/class/5004088.asp>
- Technical Literature: [http://www.restek.com/gol\\_lit\\_main.asp](http://www.restek.com/gol_lit_main.asp)
- Article: New D3606 Column Set Outperforms TCEP Columns for Benzene Analysis: [http://www.restek.com/gol\\_petro\\_A020.asp](http://www.restek.com/gol_petro_A020.asp)

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### What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions does Restek offer?

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## Refinery Gases

- Refinery Gas, what is it?
- Gas produced in petroleum refineries by cracking, reforming, and other processes;
- Mainly Light hydrocarbons and permanent gases

Separation done with PLOT columns

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## Restek PLOT Columns Versus Competitor Columns

The PLOT columns supplied by competition have issues with:

- Particle stabilization
- Reproducibility of retention
- Reproducibility of Flow

- All Restek PLOT columns have unique bonding chemistry to eliminate particle generation and are ALL tested for Flow-Restriction.
- The Flow Restriction Factor (F) is printed on each test report

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**RESTEK** Quality Assurance Report  
110 Beaver Creek • Bechtelsville, PA 19522 • 610-353-1332 • 800-355-1888 • Fax: 610-353-1338 • www.restek.com

CATALOG No. 19752    **RESTEK Chromatography**    SERIAL No. 97126

REMARKS: 10% Hydrocarbon  
 INLET: 100    INLET: 200°C  
 INLET: 100    INLET: 200°C  
 BREAK POINTS: 17 and 18/19/20/21

Retention	Name	Area	Height	Width	Area %	Retention
1.00	Hydrocarbon	11200	0.170	0.13	99.97	1.00
1.02	Hydrocarbon	2880	0.270	0.20	0.03	1.02
1.04	Hydrocarbon	4000	0.470	0.35	0.07	1.04
1.06	Hydrocarbon	1000	0.970	0.73	0.01	1.06
1.08	Hydrocarbon	1000	1.970	1.47	0.01	1.08
1.10	Hydrocarbon	1000	2.970	2.22	0.01	1.10
1.12	Hydrocarbon	1000	3.970	2.97	0.01	1.12
1.14	Hydrocarbon	1000	4.970	3.72	0.01	1.14
1.16	Hydrocarbon	1000	5.970	4.47	0.01	1.16
1.18	Hydrocarbon	1000	6.970	5.22	0.01	1.18
1.20	Hydrocarbon	1000	7.970	5.97	0.01	1.20
1.22	Hydrocarbon	1000	8.970	6.72	0.01	1.22
1.24	Hydrocarbon	1000	9.970	7.47	0.01	1.24
1.26	Hydrocarbon	1000	10.970	8.22	0.01	1.26
1.28	Hydrocarbon	1000	11.970	8.97	0.01	1.28
1.30	Hydrocarbon	1000	12.970	9.72	0.01	1.30
1.32	Hydrocarbon	1000	13.970	10.47	0.01	1.32
1.34	Hydrocarbon	1000	14.970	11.22	0.01	1.34
1.36	Hydrocarbon	1000	15.970	11.97	0.01	1.36
1.38	Hydrocarbon	1000	16.970	12.72	0.01	1.38
1.40	Hydrocarbon	1000	17.970	13.47	0.01	1.40
1.42	Hydrocarbon	1000	18.970	14.22	0.01	1.42
1.44	Hydrocarbon	1000	19.970	14.97	0.01	1.44
1.46	Hydrocarbon	1000	20.970	15.72	0.01	1.46
1.48	Hydrocarbon	1000	21.970	16.47	0.01	1.48
1.50	Hydrocarbon	1000	22.970	17.22	0.01	1.50
1.52	Hydrocarbon	1000	23.970	17.97	0.01	1.52
1.54	Hydrocarbon	1000	24.970	18.72	0.01	1.54
1.56	Hydrocarbon	1000	25.970	19.47	0.01	1.56
1.58	Hydrocarbon	1000	26.970	20.22	0.01	1.58
1.60	Hydrocarbon	1000	27.970	20.97	0.01	1.60
1.62	Hydrocarbon	1000	28.970	21.72	0.01	1.62
1.64	Hydrocarbon	1000	29.970	22.47	0.01	1.64
1.66	Hydrocarbon	1000	30.970	23.22	0.01	1.66
1.68	Hydrocarbon	1000	31.970	23.97	0.01	1.68
1.70	Hydrocarbon	1000	32.970	24.72	0.01	1.70
1.72	Hydrocarbon	1000	33.970	25.47	0.01	1.72
1.74	Hydrocarbon	1000	34.970	26.22	0.01	1.74
1.76	Hydrocarbon	1000	35.970	26.97	0.01	1.76
1.78	Hydrocarbon	1000	36.970	27.72	0.01	1.78
1.80	Hydrocarbon	1000	37.970	28.47	0.01	1.80
1.82	Hydrocarbon	1000	38.970	29.22	0.01	1.82
1.84	Hydrocarbon	1000	39.970	29.97	0.01	1.84
1.86	Hydrocarbon	1000	40.970	30.72	0.01	1.86
1.88	Hydrocarbon	1000	41.970	31.47	0.01	1.88
1.90	Hydrocarbon	1000	42.970	32.22	0.01	1.90
1.92	Hydrocarbon	1000	43.970	32.97	0.01	1.92
1.94	Hydrocarbon	1000	44.970	33.72	0.01	1.94
1.96	Hydrocarbon	1000	45.970	34.47	0.01	1.96
1.98	Hydrocarbon	1000	46.970	35.22	0.01	1.98
2.00	Hydrocarbon	1000	47.970	35.97	0.01	2.00
2.02	Hydrocarbon	1000	48.970	36.72	0.01	2.02
2.04	Hydrocarbon	1000	49.970	37.47	0.01	2.04
2.06	Hydrocarbon	1000	50.970	38.22	0.01	2.06
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2.10	Hydrocarbon	1000	52.970	39.72	0.01	2.10
2.12	Hydrocarbon	1000	53.970	40.47	0.01	2.12
2.14	Hydrocarbon	1000	54.970	41.22	0.01	2.14
2.16	Hydrocarbon	1000	55.970	41.97	0.01	2.16
2.18	Hydrocarbon	1000	56.970	42.72	0.01	2.18
2.20	Hydrocarbon	1000	57.970	43.47	0.01	2.20
2.22	Hydrocarbon	1000	58.970	44.22	0.01	2.22
2.24	Hydrocarbon	1000	59.970	44.97	0.01	2.24
2.26	Hydrocarbon	1000	60.970	45.72	0.01	2.26
2.28	Hydrocarbon	1000	61.970	46.47	0.01	2.28
2.30	Hydrocarbon	1000	62.970	47.22	0.01	2.30
2.32	Hydrocarbon	1000	63.970	47.97	0.01	2.32
2.34	Hydrocarbon	1000	64.970	48.72	0.01	2.34
2.36	Hydrocarbon	1000	65.970	49.47	0.01	2.36
2.38	Hydrocarbon	1000	66.970	50.22	0.01	2.38
2.40	Hydrocarbon	1000	67.970	50.97	0.01	2.40
2.42	Hydrocarbon	1000	68.970	51.72	0.01	2.42
2.44	Hydrocarbon	1000	69.970	52.47	0.01	2.44
2.46	Hydrocarbon	1000	70.970	53.22	0.01	2.46
2.48	Hydrocarbon	1000	71.970	53.97	0.01	2.48
2.50	Hydrocarbon	1000	72.970	54.72	0.01	2.50
2.52	Hydrocarbon	1000	73.970	55.47	0.01	2.52
2.54	Hydrocarbon	1000	74.970	56.22	0.01	2.54
2.56	Hydrocarbon	1000	75.970	56.97	0.01	2.56
2.58	Hydrocarbon	1000	76.970	57.72	0.01	2.58
2.60	Hydrocarbon	1000	77.970	58.47	0.01	2.60
2.62	Hydrocarbon	1000	78.970	59.22	0.01	2.62
2.64	Hydrocarbon	1000	79.970	59.97	0.01	2.64
2.66	Hydrocarbon	1000	80.970	60.72	0.01	2.66
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2.96	Hydrocarbon	1000	95.970	71.97	0.01	2.96
2.98	Hydrocarbon	1000	96.970	72.72	0.01	2.98
3.00	Hydrocarbon	1000	97.970	73.47	0.01	3.00
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3.26	Hydrocarbon	1000	110.970	83.22	0.01	3.26
3.28	Hydrocarbon	1000	111.970	83.97	0.01	3.28
3.30	Hydrocarbon	1000	112.970	84.72	0.01	3.30
3.32	Hydrocarbon	1000	113.970	85.47	0.01	3.32
3.34	Hydrocarbon	1000	114.970	86.22	0.01	3.34
3.36	Hydrocarbon	1000	115.970	86.97	0.01	3.36
3.38	Hydrocarbon	1000	116.970	87.72	0.01	3.38
3.40	Hydrocarbon	1000	117.970	88.47	0.01	3.40
3.42	Hydrocarbon	1000	118.970	89.22	0.01	3.42
3.44	Hydrocarbon	1000	119.970	89.97	0.01	3.44
3.46	Hydrocarbon	1000	120.970	90.72	0.01	3.46
3.48	Hydrocarbon	1000	121.970	91.47	0.01	3.48
3.50	Hydrocarbon	1000	122.970	92.22	0.01	3.50
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3.54	Hydrocarbon	1000	124.970	93.72	0.01	3.54
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3.78	Hydrocarbon	1000	136.970	102.72	0.01	3.78
3.80	Hydrocarbon	1000	137.970	103.47	0.01	3.80
3.82	Hydrocarbon	1000	138.970	104.22	0.01	3.82
3.84						

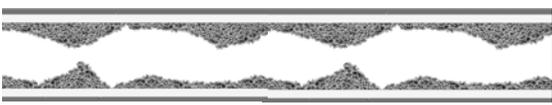
PLOT Layer is often deposited as non homogeneous film

**FLOW Restriction**

Ideal "open Path: Flow-restriction factor = 1.0



PLOT column with Flow-Restrictions: Flow restriction factor "F" = 0.2-0.8



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This was one of the reasons why we could replace Varian and Agilent PLOT columns for:

- Siemens
- Agilent micro GC
- C2V (Thermo)
- Schlumberger
- Many companies that supply Analyzer solutions (PAC, Interscience,..)

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**Transportation**



**Transportation of PLOT columns**

During transport columns are subjected to uncontrolled:

- Vibrations
- Pressure changes
- Temperature changes
- Shocks



Which all can destabilize the adsorption layer..

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## Dow Chemical, J . Luong

One of the concerns involves the use of PLOT column technology is adsorbent particle shedding. The shedding of adsorbent particles has the potential of plugging the flow channel of the capillary flow Deans switch device. This in turn can cause retention time shift.

Prior to connecting the columns to the capillary flow Deans switch device, we highly recommend the columns be purged with carrier gas at a flow of 50 mL/min for 15 min to remove any particles that might have been dislodged during shipment of the columns from the manufacturers.

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## Refinery Gas Analysis

- C1-C5
  - Rt AluminaBOND KCl or Na2SO4
- Permanent gases, C1-C2 and CO2:
  - Rt Msieve 5A / Rt-Q BOND
  - ShinCarbon

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## C1-C5 Hydrocarbons Rt-Alumina BOND

KCl : Non polar Al2O3 surface

Na2SO4 : Polar Al2O3 surface

Deactivation is necessary to reduce the activity of the alumina..

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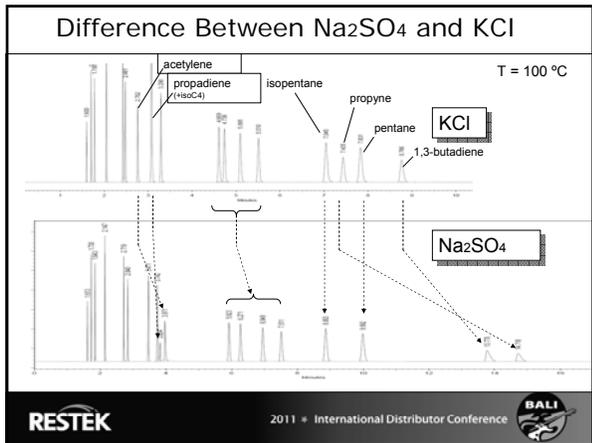
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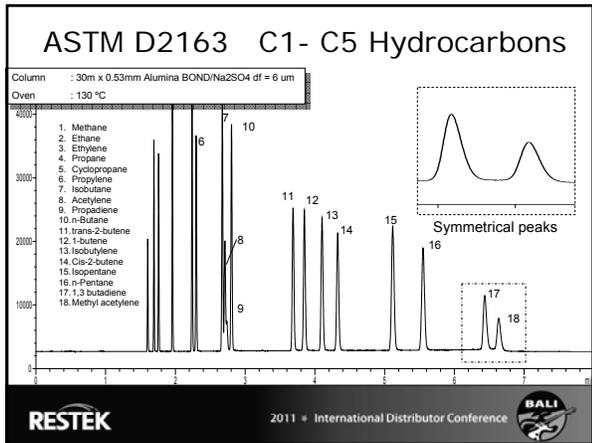
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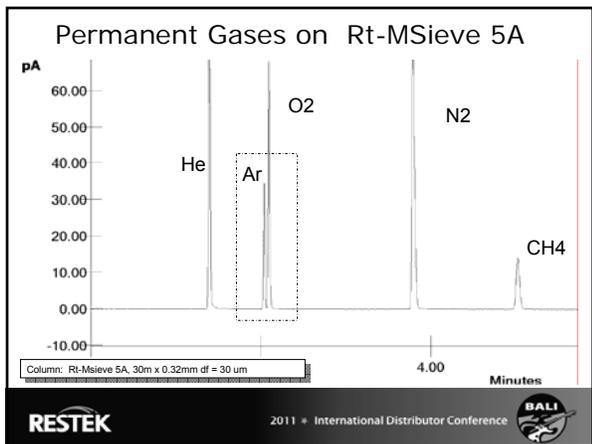
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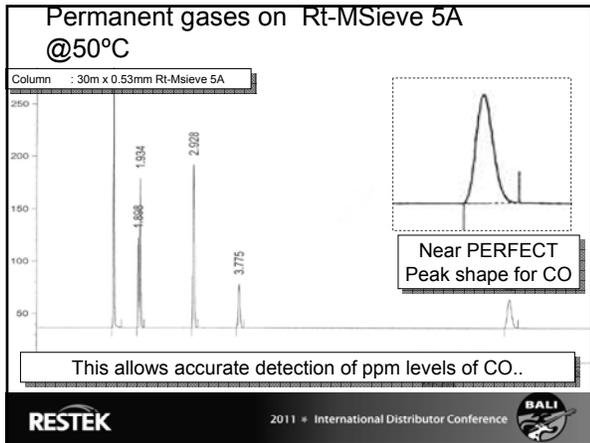
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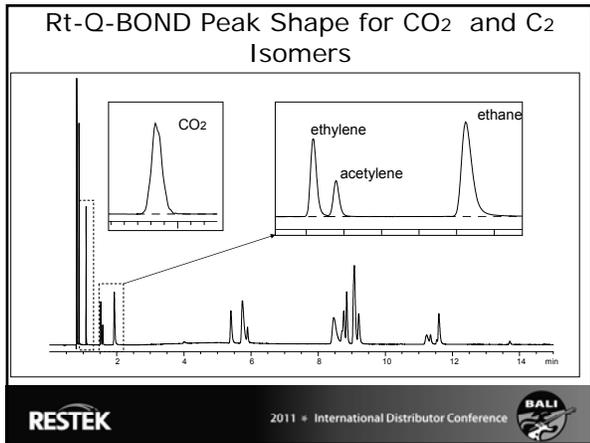
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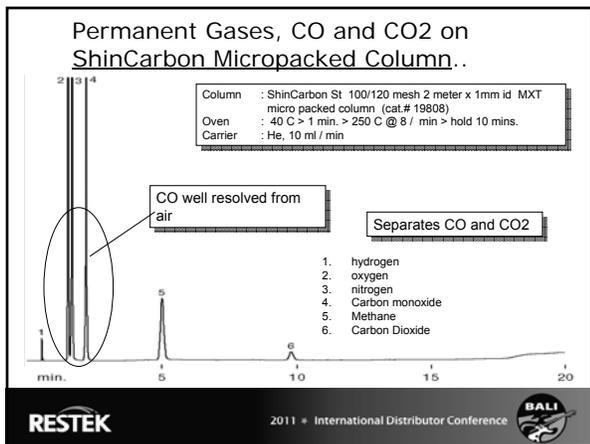
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## Product Information for PLOT

- Restek Refined brochure PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Next generation PLOT columns PCFL1163A: <http://www.restek.com/restek/images/external/PCFL1163A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp>
- Technical Literature: [http://www.restek.com/aci\\_lit\\_main.asp](http://www.restek.com/aci_lit_main.asp)
- Article: New PLOT Column Technology Stabilizes Flow and Improves Analysis of Light Hydrocarbons, Solvents, and Permanent Gases : [http://www.restek.com/aci\\_petro\\_A023.asp](http://www.restek.com/aci_petro_A023.asp)
- Article: Advances in Porous Layer Open Tubular Columns : [http://www.iscrops.com/Media%20Library/files/972new%20layout\\_0.pdf](http://www.iscrops.com/Media%20Library/files/972new%20layout_0.pdf)



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## Additional UNIQUE Restek Development

Making PLOT and LIQUID PHASE columns available in MXT..

In PROCESS type applications, customers look for:

RELIABILITY..

MXT columns will offer extra security and value..

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MXT-MSieve 5A

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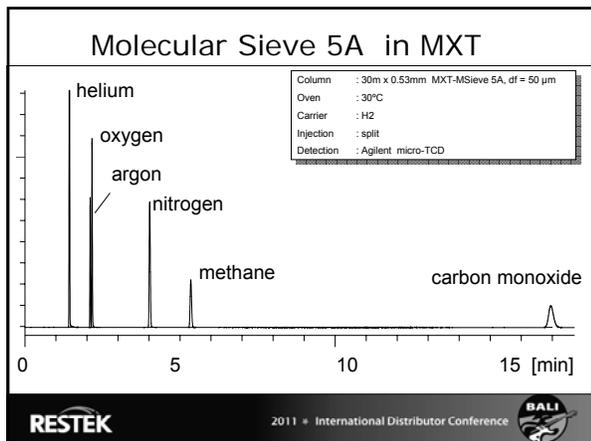
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**HRM**alytic +61(0)3 9762 2034  
ECHnology Pty Ltd

Australian Distributors  
Importers & Manufacturers  
[www.chromtech.net.au](http://www.chromtech.net.au)

12/13

Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel: 03 9762 2034 . . . in AUSTRALIA




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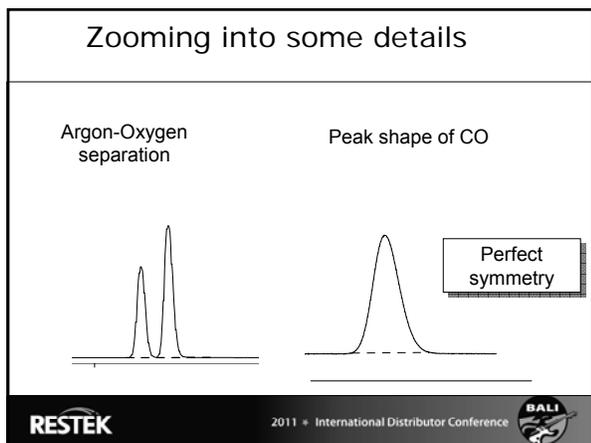
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MXT-Alumina BOND  
(Na<sub>2</sub>SO<sub>4</sub> deactivation)

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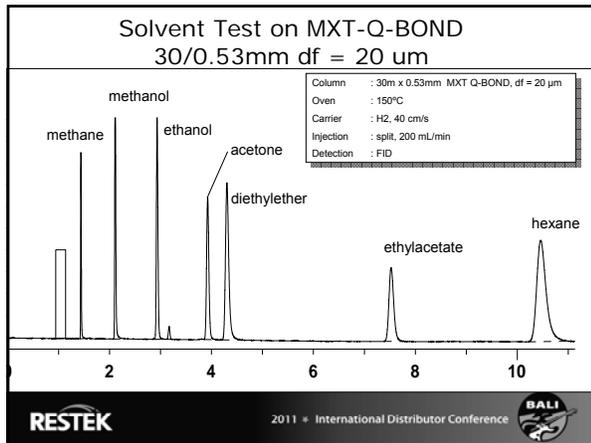
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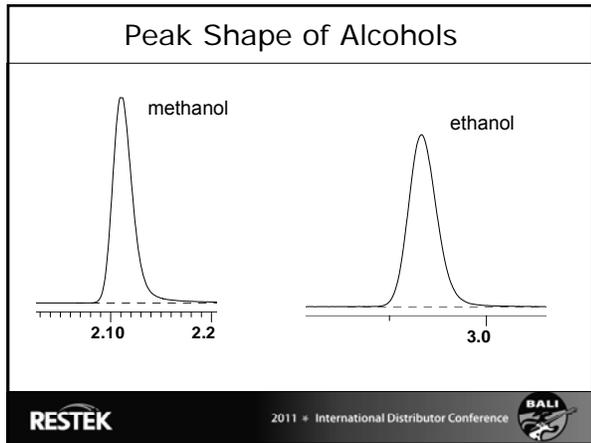
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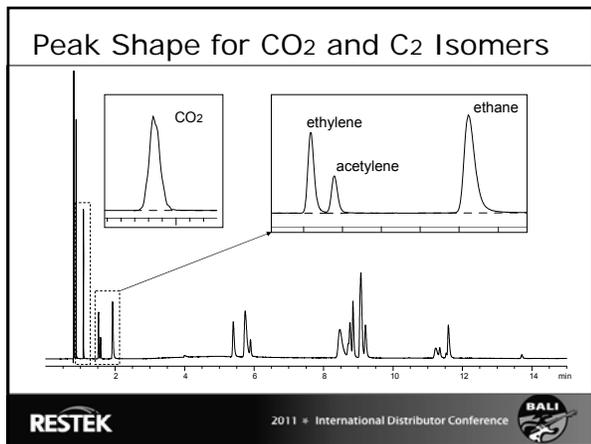
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## Product information MXT PLOT

- Brochure Restek Refined PCFL1195A: <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Brochure Next generation PLOT columns PCFL1163A: <http://www.restek.com/restek/images/external/PCFL1163A.pdf>
- Applications see: <http://www.restek.com/restek/class/5004088.asp> and [http://www.restek.com/info\\_chromatograms.asp](http://www.restek.com/info_chromatograms.asp)
- Technical Literature: [http://www.restek.com/ajci\\_lit\\_main.asp](http://www.restek.com/ajci_lit_main.asp)



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## What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions does Restek offer?

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## Reference

Restek is internationally known for the Siltek / and SilcoSteel deactivations;

Siltek treated parts are widely used and acknowledged in the petrochemical Industry for sulfur compound stability...

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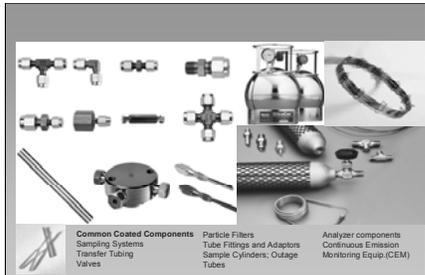
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All parts in the GC system that come in contact with sulfur compounds can be deactivated by the Siltek technology...



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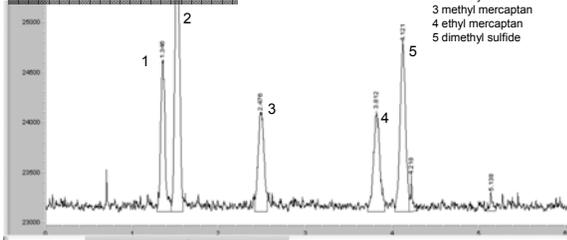
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### Sulfur Gases at 19 ppb

Column : Rxi™-1, 60m, 0.53mm ID, 7.00µm  
 Sample : 19 ppbv each in helium  
 Oven temp. : 30°C  
 Det. : SCID  
 Det. temp. : 800°C



- 1 hydrogen sulfide
- 2 carbonyl sulfide
- 3 methyl mercaptan
- 4 ethyl mercaptan
- 5 dimethyl sulfide

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### Most Important Issue for Trace Sulfur Analysis

## System Inertness

Use a complete Inert system

- Deactivated transfer tubing, Inert Valves / coupling devices, Inert columns, Inert detection liners, Pressure regulators

Sulfur compounds will disappear especially at low levels..

Restek Developed Siltek deactivated tubing..

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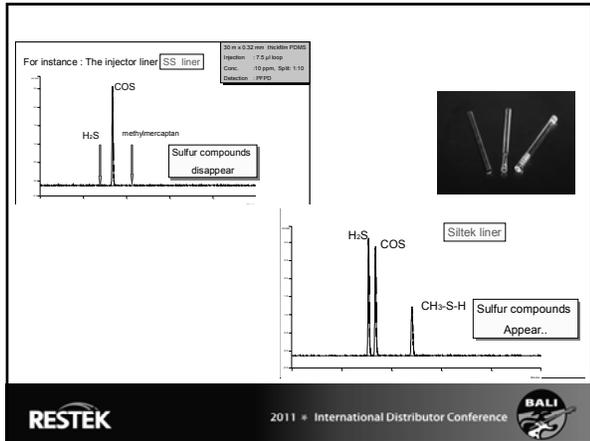
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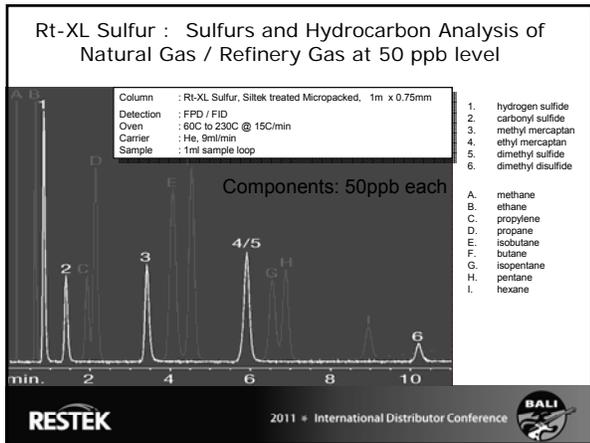
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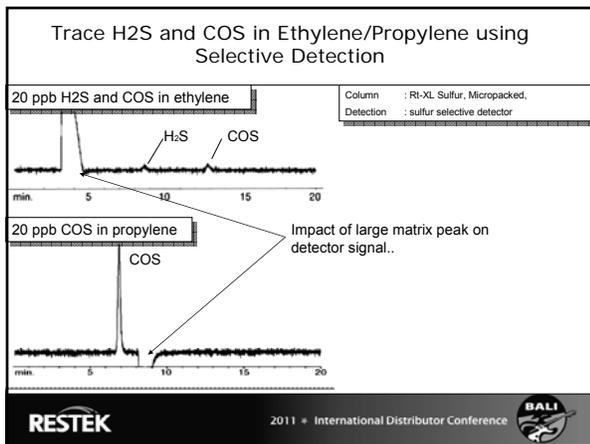
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## Product information Sulfur

- Restek Refined brochure : PCFL1195A, <http://www.restek.com/restek/images/external/PCFL1195A.pdf>
- Applications : [gc\\_pc00202.pdf](#); [gc\\_pc00202.pdf](#); [gc\\_pc00202.pdf](#); [gc\\_pc00199.pdf](#); [gc\\_pc00199.pdf](#)
- Powerpoint handout : [http://www.restek.com/restek/images/external/PASS6\\_SF\\_TraceLevelSulfurs\\_Restek.pdf](http://www.restek.com/restek/images/external/PASS6_SF_TraceLevelSulfurs_Restek.pdf)
- Applications see : <http://www.restek.com/restek/class/5004088.asp>
- Technical Literature : [http://www.restek.com/aol\\_lit\\_main.asp](http://www.restek.com/aol_lit_main.asp)
- Article: Stable Sulfur & Mercury Sampling in Refineries Using Sitek® and Sulfinet® Surface Treated Components: [http://www.restek.com/aol\\_petro\\_A016.asp](http://www.restek.com/aol_petro_A016.asp)



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## What does every refinery do?

- Simulated Distillation
- Detailed Hydrocarbon Analysis
- Benzene / Toluene/ ethanol in gasoline
- Refinery gases
- Sulfur analysis
- Oxygenates

What Solutions does Restek offer?

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## Oxygenates in Gasoline Analyses: ASTM D4815

- Oxygenates in Finished Gasoline (Octane boosters) D4815
- Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography
- TCEP column plus Rtx-1 column or Rtx-DHA column

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# Food Safety

Jonathan Keim

Sample Preparation Product  
Marketing Manager

Restek Corporation



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# Consumer Pressure

Back to Article | Click to Print

**TIME**  
WEDNESDAY, SEPTEMBER 14, 2007

Tuesday, Sep. 11, 2007  
**Tainted-Baby-Milk Scandal in China**  
By AUSTIN RAMSEY AND LIN YANG

**melamine**

USA TODAY  
News > Health & Behavior

**Report: China's animal feed tainted with melamine**

BEIJING (AP) — Animal feed producers in China commonly add the industrial chemical melamine to their products to make them appear higher in protein, state media reported Thursday, an indication that the scope of the country's latest food safety scandal could extend beyond milk and eggs.

The practice of mixing melamine into animal feed is an "open secret" in the industry, the Reuters Daily News Service said in an online report released on the website of the central government's state-run supply chain. The report said the practice was common in the country's largest feed mill, and that the company did not come forward with the recall because it was waiting for the recall process to move through the Chinese system. "I can look myself in the mirror and see the problem," said a senior executive at the company.

Discovery **Bisphenol A**

**Food Packaging Harbors Harmful Chemicals**  
Eating fresh food, as opposed to packaged ones, can reduce your level of exposure to bisphenol A, a chemical that has been linked to health problems.

By Emily Slaten | Tue Mar 29, 2011 07:05 AM ET

Photo: iStockphoto.com, Food.com

**phthalates**



**acrylamide**



**mycotoxins**

**Hormones In Food: Should You Worry?**

**Vet drugs**

**NOAA, FDA to make data public from continuing Gulf seafood tests**

By Caroline Scott-Thomas, 29-Mar-2011

Post a comment

**PAHs**

The National Oceanic and Atmospheric Administration (NOAA) has said it will continue to test seafood from the Gulf of Mexico into the summer.

**Consumer Alert: Beware of Pesticide Contamination of Non-Organic Foods**

"Dirty Dozen" produce carries more pesticide residues

By Danielle Dellorto  
CNN, June 1, 2010

**pesticides**

**Dioxins**

**California lists 4-Methylimidazole as a Proposition 65 carcinogen**

**4MEI**



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# Outline

- Customers – Who are they?
- Compounds and methods of interest
- The Restek Food Safety analysis arsenal
  - Sample preparation products (QuEChERS)
  - Reference standards (pesticides, melamine, etc.)
  - GC and LC columns and accessories
- Web tools, literature



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# Food Safety Customers

- Governmental organizations
  - FERA (UK), VWA (The Netherlands)
- Contract laboratories, research organizations
  - Eurofins, RIKILT (The Netherlands), Covance, Intertek, Silliker
- University research groups
  - Institute of Chemical Technology (Prague, CZ)
- Private food companies
  - Larger ones may do in-house testing



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# Food Safety Compounds

- Pesticides
  - Organochlorine/nitrogen/phosphorus, etc.
- Mycotoxins
- Veterinary drug residues
- Adulterants
  - Melamine, pesticides, other toxins
- PAHs, PBDEs, PCBs, dioxins, furans, etc.

# Food Safety Methods

- Codex Alimentarius (Food Law)
  - United Nations Food and Agriculture Organization
  - World Health Organization
  - Methods are somewhat outdated
- Analytical Methods for Pesticide Residues in Foodstuffs
  - The Netherlands
  - Well-developed, multiresidue methods
  - Still used, but becoming outdated

# Global Economy

## US

- FDA
- NIH
- EPA
- AOAC

## Food Safety Modernization Act

Passed December 2010  
Import regulations

## China

Growing  
following US & EU

## EFSA

European Food Safety Authority

- EU
- Regulation (MRLs)
- Scientific opinions
- WHO

## Individual Country

## Japan

Mature Food Safety system

## Latin America

driven by export laws

standardization of food  
safety regulations

## Some statistics

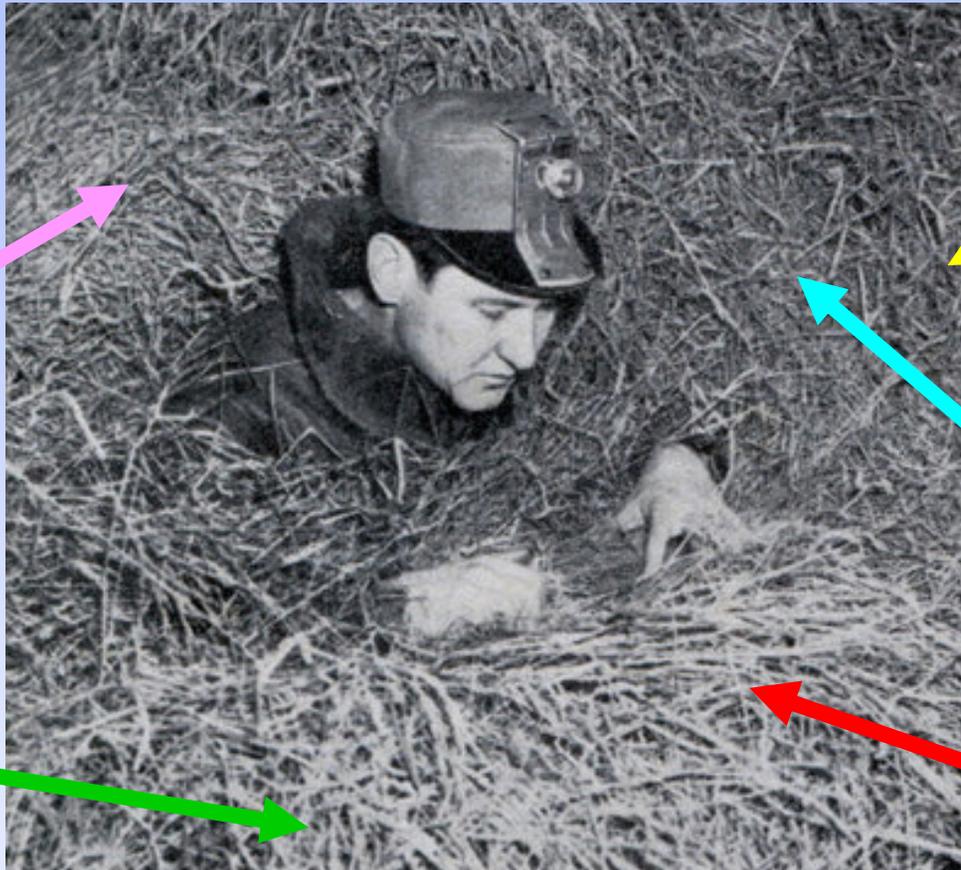
- 2008 = separations market accounted for more 20% of the laboratory analytical instrumentation demand in the food industry
- Growth driven by LC–MS and GC–MS systems
- U.S. food-safety testing market value  
2010 = \$3.4 billion  
2015 = \$4.7 billion in 2015
- Global food safety testing market  
\$11.4 billion by 2015



SDI's Market Analysis and Perspective report entitled, The Analytical Cornucopia: The Food, Beverage and Agriculture Market for Analytical Instruments.

# What challenges arise for the chemist?

## Trace analysis



many analytes

Diverse matrices

Sample matrix

sensitivity

compound stability

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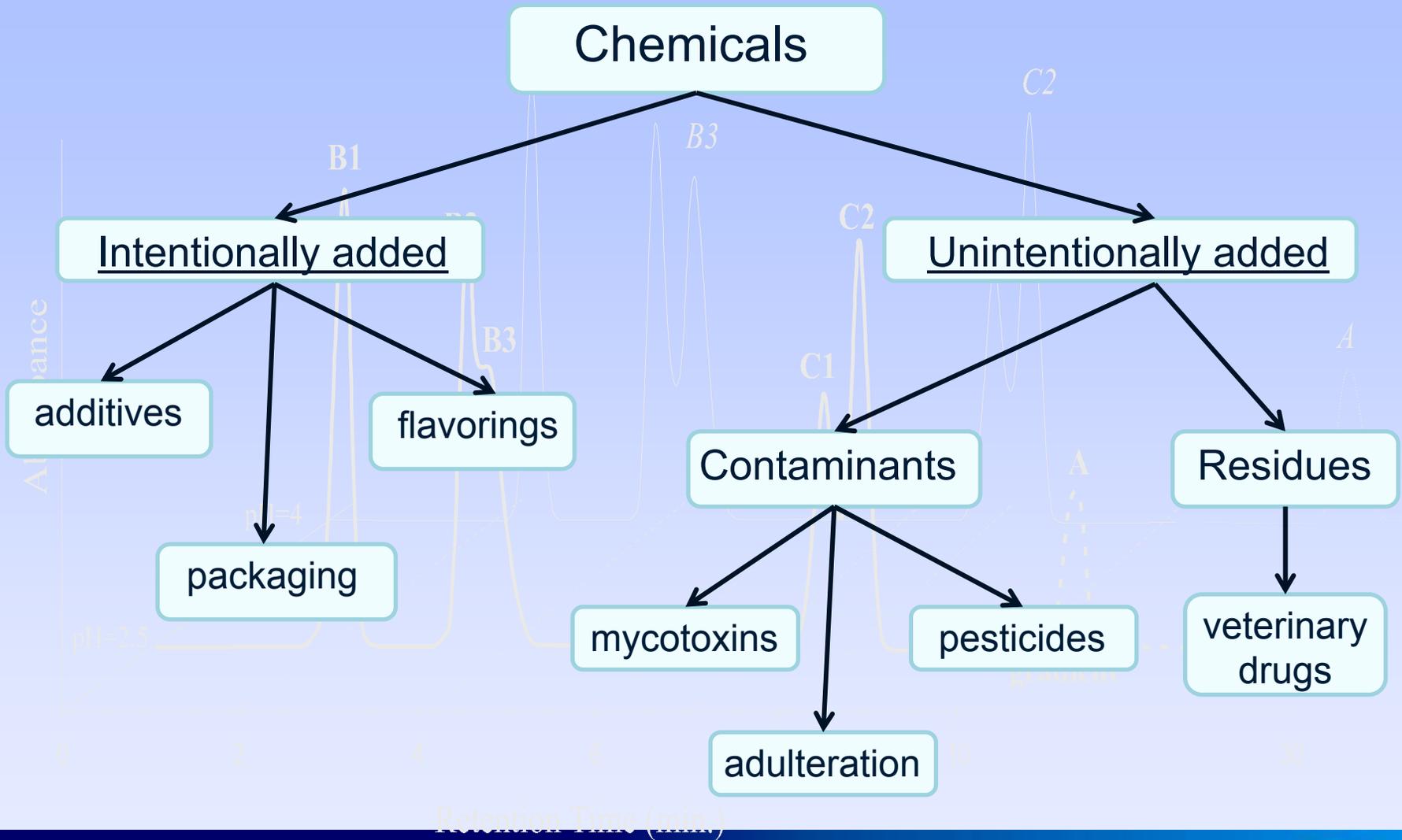
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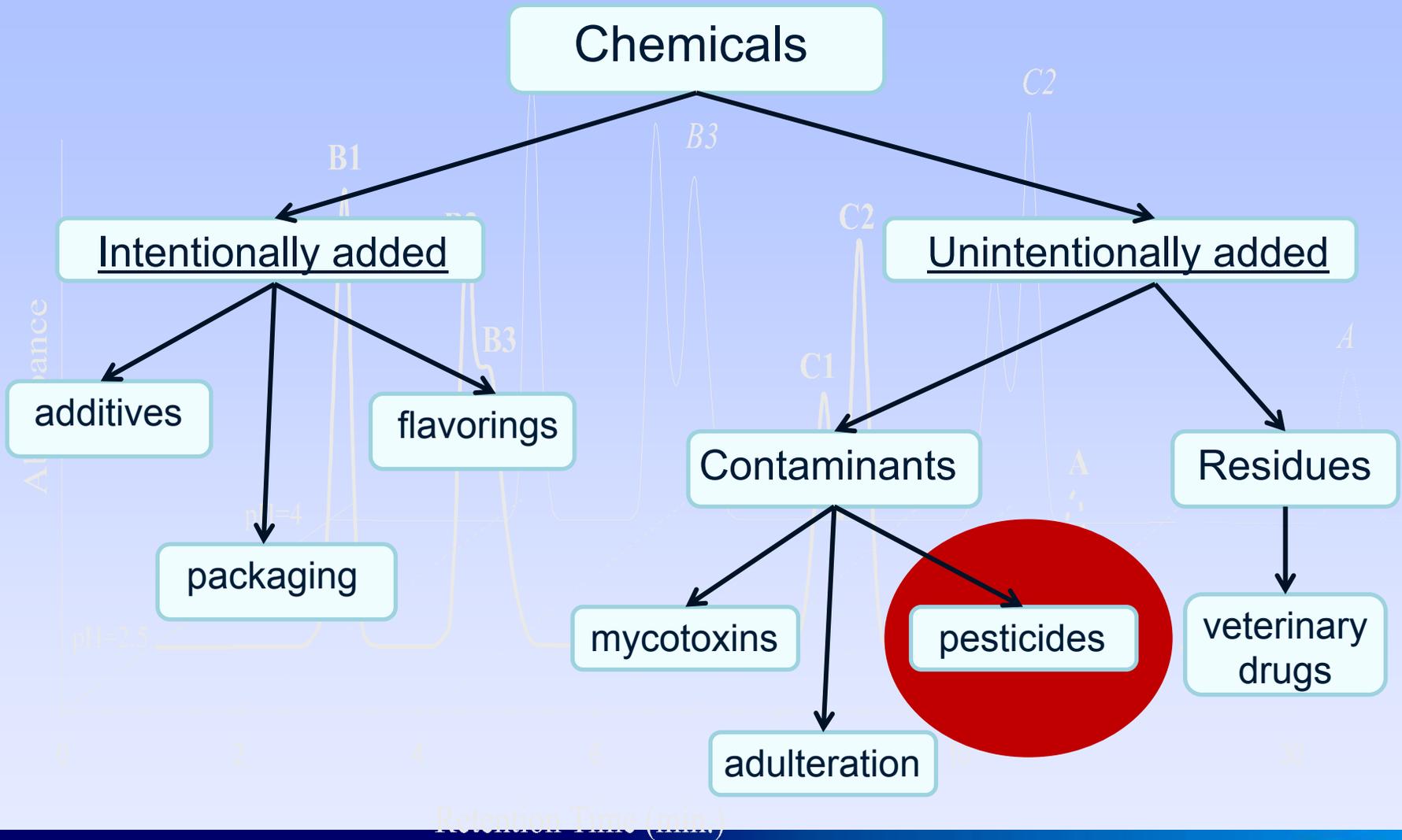
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# Function or Chemical Class



# Function or Chemical Class



# Pesticide Residues in Foods

- Techniques
  - Historically GC (ECD or MS)
  - Increasingly add LC-MS/MS
  - Comprehensive screening desirable
- Laboratory Needs
  - HIGH throughput
  - All-purpose phases
    - Ultra Aqueous C18
  - Application-specific columns
    - Target classes (e.g. Ultra Quat)



# Pesticide Residues in Foods

## Liquid Chromatography: LC/MS/MS

### LC Columns

### Aqueous C18

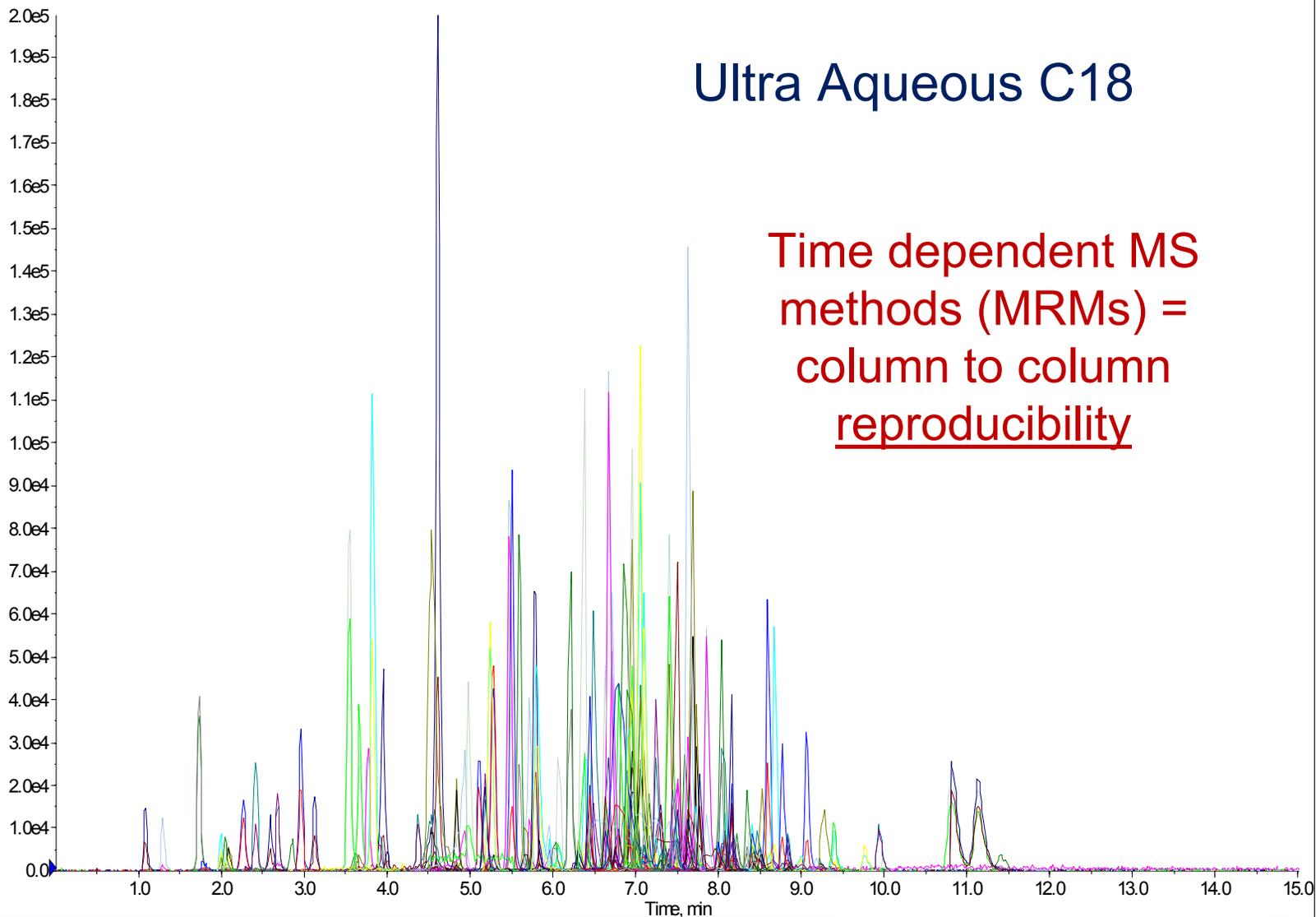
### Aqueous C18 Advantages

- Highly base-deactivated
- Compatible with highly aqueous (up to 100%) mobile phases

### General purpose

#### Great for

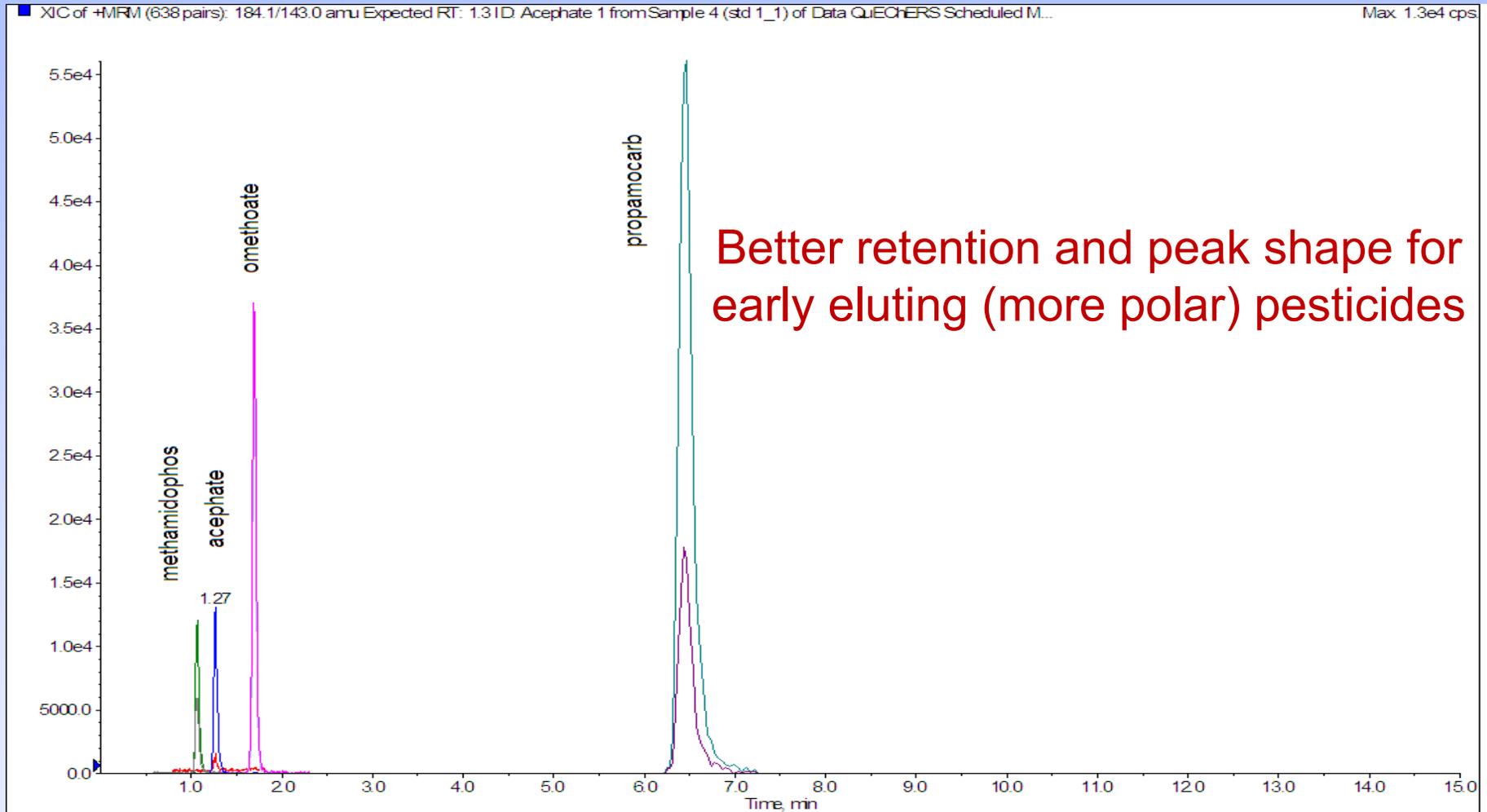
- Multiresidue analysis
- Variety of pesticide classes
- Scalable on different silica families



## Ultra Aqueous C18

Time dependent MS  
methods (MRMs) =  
column to column  
reproducibility

# LC/MS/MS Pesticide Analysis: Ultra Aqueous C18 3 $\mu$ m, 100x2.1mm



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# Pesticide Residues in Foods

## Gas Chromatography: GC-MS, GC-ECD and GC-NPD

### GC Columns

#### Rxi®-5Sil MS

#### Rxi technology

- Highest inertness
- Lowest bleed
- Exceptional reproducibility

#### General purpose

Great for

- Multiresidue analysis
- variety of pesticide classes

#### High sensitivity

- Ideal for applications requiring high sensitivity
- ppb detection limits

# Retention Indices for Pesticides on "5" Columns

Compound	Type	DB-5MS	Rxi-5Sil MS	Difference
Methamidophos	OPP	1222.0	1221.8	0.2
Omethoate	OPP	1585.1	1583.2	1.9
Diazinon	OPP	1788.7	1788.3	0.4
Chlorothalonil	OCP	1789.8	1791.2	-1.4
Malathion	OPP	1959.7	1958.8	0.8
Myclobutanil	ONP	2186.4	2186.6	-0.2
4,4-DDT	OCP	2351.5	2353.7	-2.3
Propargite	OSP	2391.0	2391.1	-0.1
Bifenthrin	Pyr	2465.1	2466.2	-1.0
trans-Permethrin	Pyr	2712.2	2714.1	-1.9



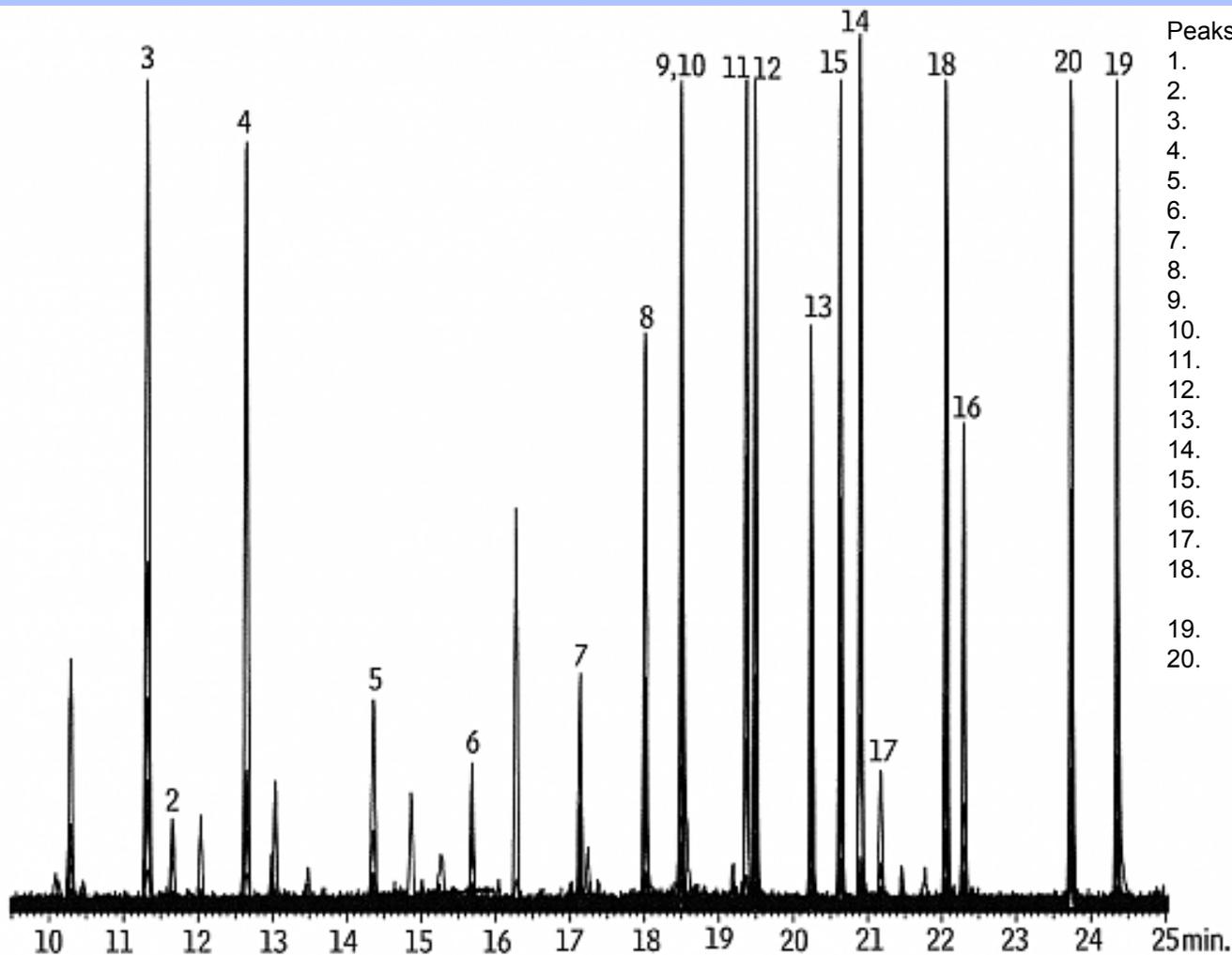

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# Chlorinated Pesticide Residues in Olive Oil Rxi®-5sil MS

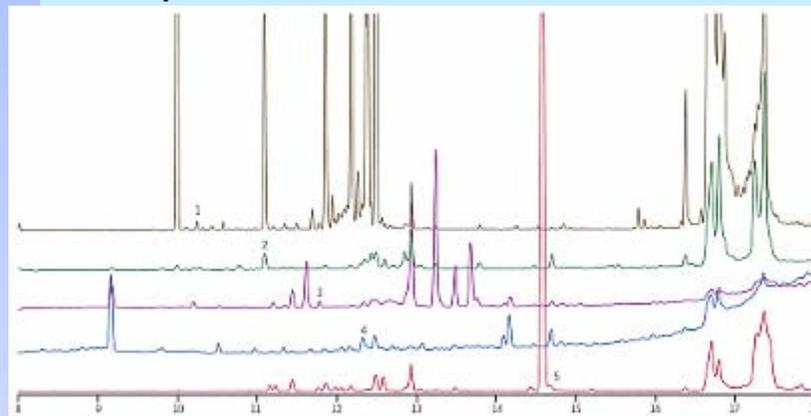
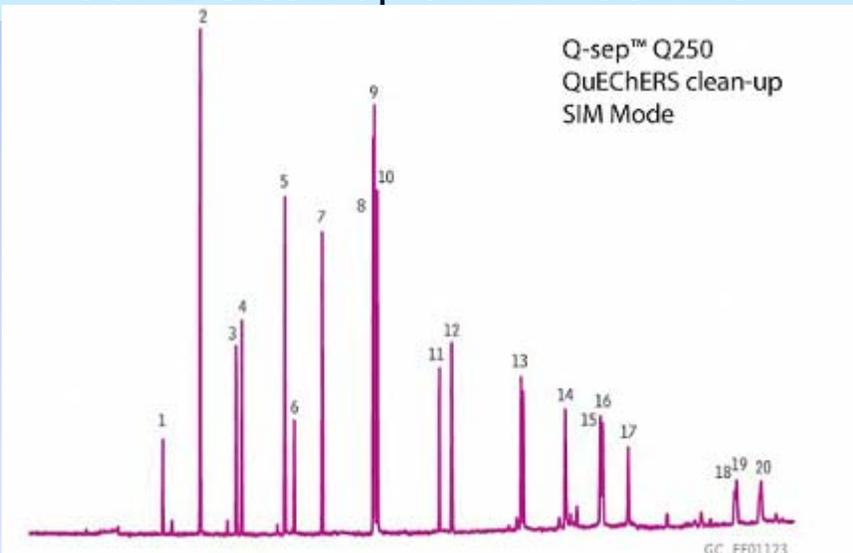


Peaks	Quant. ion	Qual. ion 1	Qual. ion 2
1.	α-BHC*	219	181
2.	γ-BHC	219	181
3.	β-BHC	219	181
4.	δ-BHC	219	181
5.	Heptachlor	272	237
6.	Aldrin	263	293
7.	Heptachlor epoxide		263
8.	δ-chlordane	272	237
9.	α-Chlordane	272	237
10.	Endosulfan I	195	207
11.	4,4'-DDE	246	318
12.	Dieldrin	79	263
13.	Endrin	263	281
14.	4,4'-DDD	235	165
15.	Endosulfan II	195	207
16.	4,4'-DDT	235	165
17.	Endrin aldehyde	67	250
18.	Endosulfan sulfate		272
	239		
19.	Methoxychlor	227	274
20.	Endrin ketone	67	317

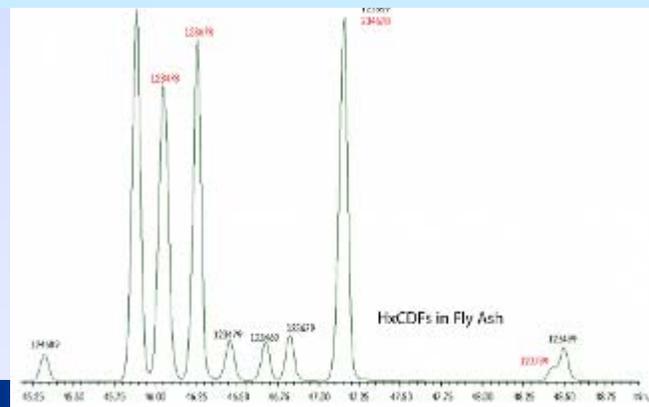
# Rxi®-5Sil MS: versatile for other analyses

## Analysis of Melamine and Related Compounds in Infant Formula

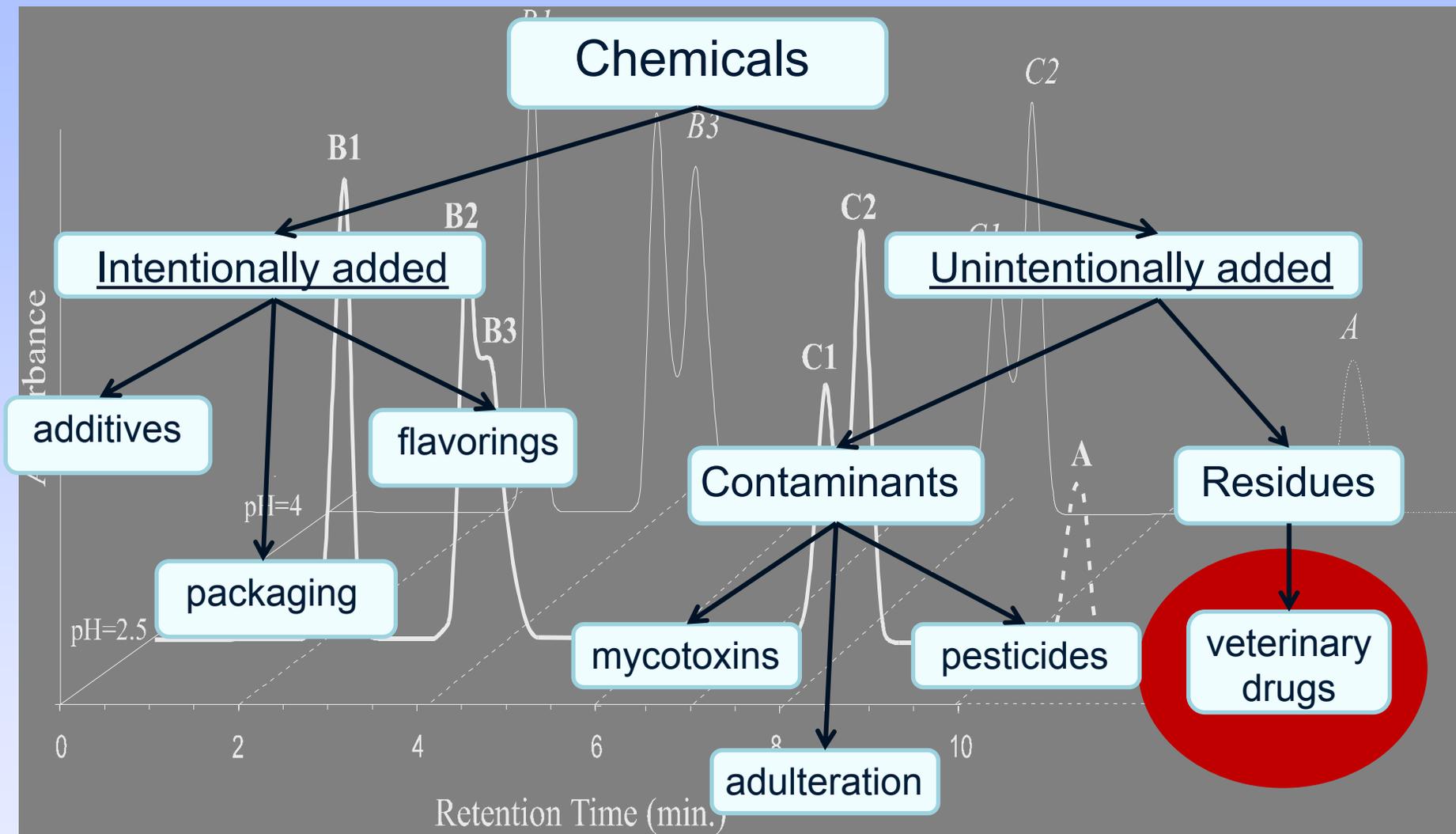
## Polycyclic Aromatic Hydrocarbons Infant Formula with and without QuEChERS dSPE Clean-up on Rxi®-5Sil MS



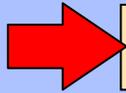
## Dioxins (HxCDFs) in Fly Ash on Rxi®-5Sil MS



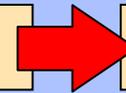
# Function or Chemical Class



drugs in feed



drug residues remain



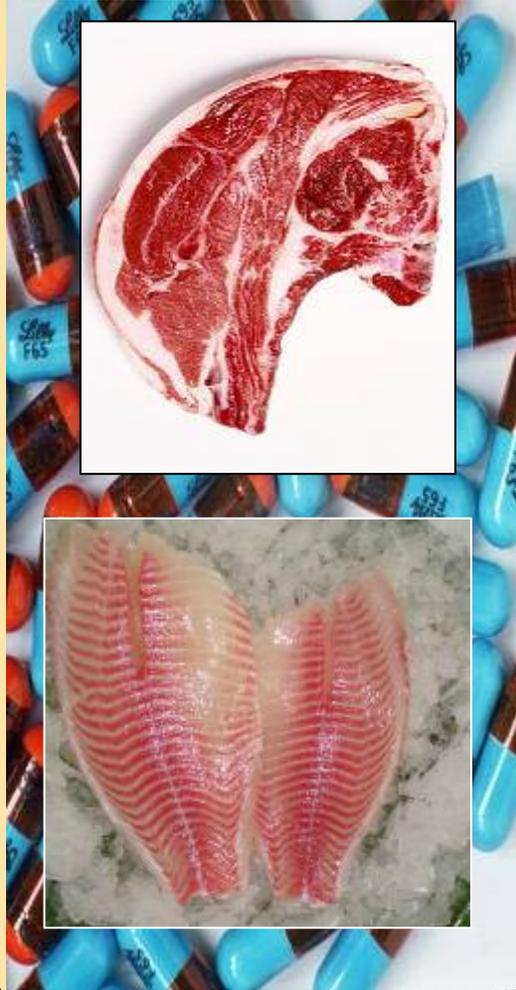
drug residues in food



- Prevent pathogen and disease
- Fear resistant strains



- Promote growth
- US, Canada, Australia



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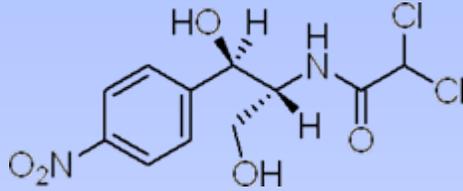


# Veterinary Drugs

## Tetracyclines



## Chloramphenicol

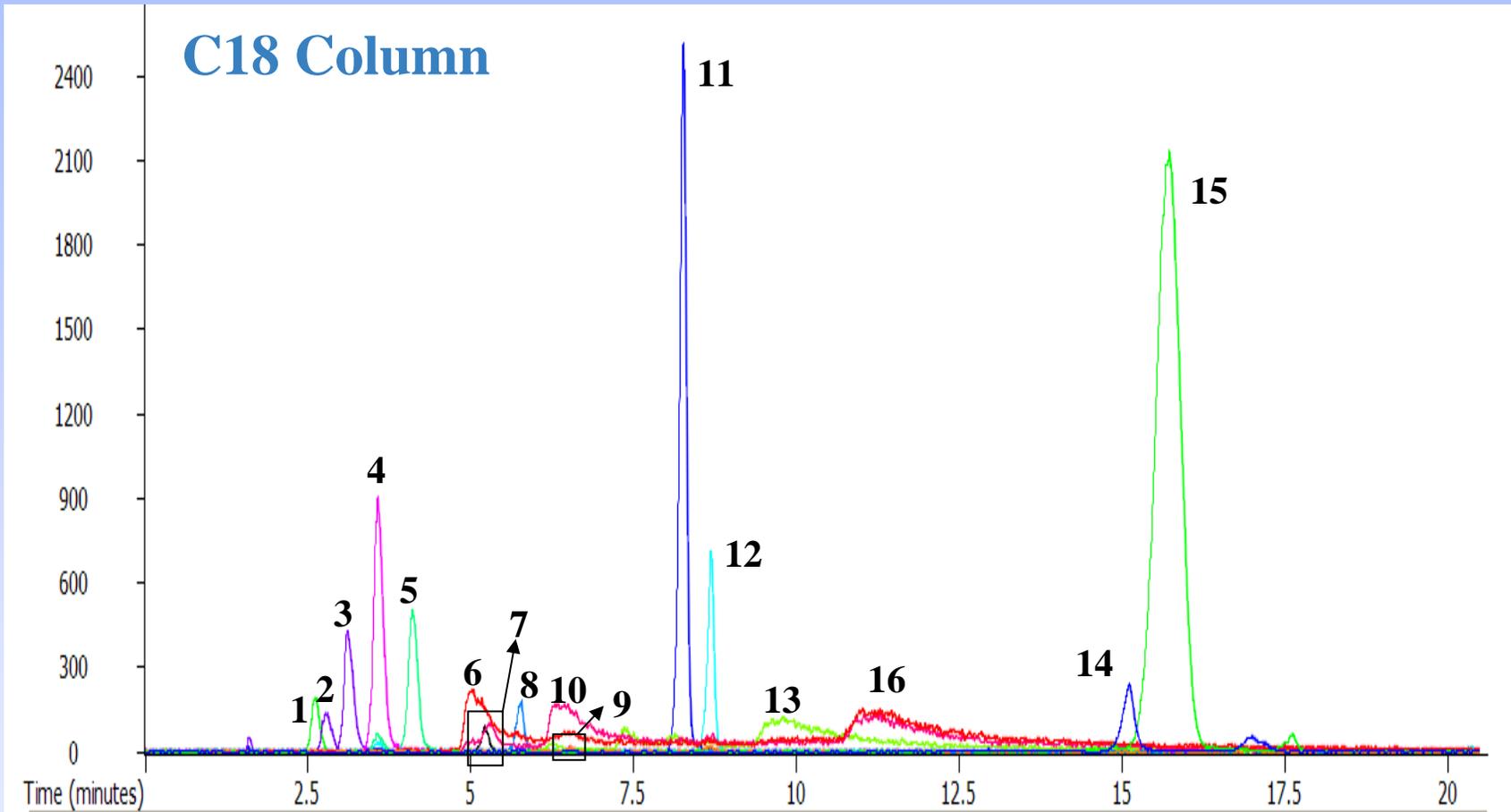


## erythromycin



Class	Compound
sulfonamides	sulfachloropyridazine
sulfonamides	sulfadiazine
sulfonamides	sulfadimethoxine
sulfonamides	sulfamerazine
sulfonamides	sulfamethazine
sulfonamides	sulfaquinoxaline
sulfonamides	sulfathiazole
sulfonamides	sulfamethoxypyridazine
sulfonamides	sulfamethoxazole
tetracyclines	chlortetracycline
tetracyclines	doxycycline
tetracyclines	oxytetracycline
tetracyclines	tetracycline
nitrobenzenes	chloramphenicol
macrolides	tylosin
macrolides	erythromycin

# Veterinary Drug Residues



Mobile phase A: 0.2% formic acid  
Mobile phase B: methanol/acetonitrile (80/20, v/v) with 0.2% formic acid  
Flow: 1 mL/min with pre-injection split delivering 0.2

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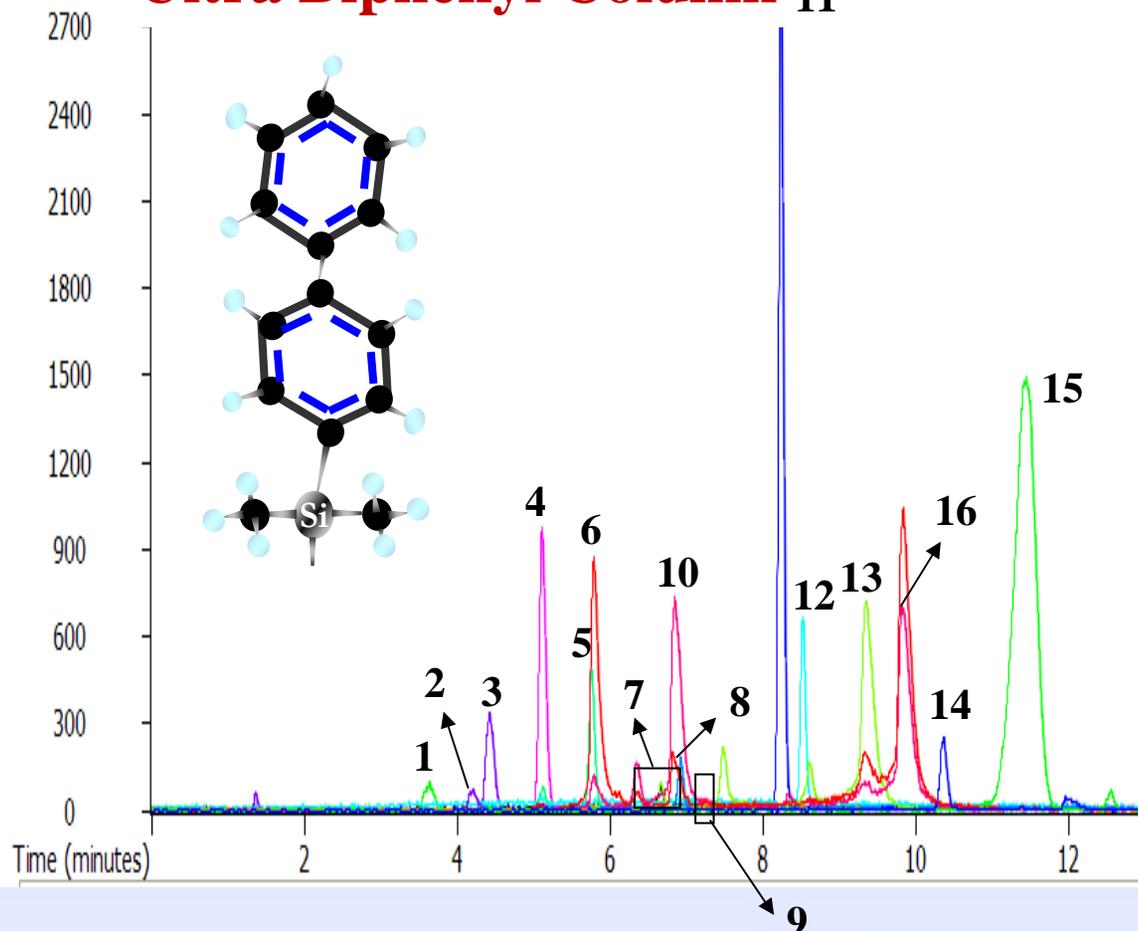
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# Veterinary Drug Residues

## Ultra Biphenyl Column 11

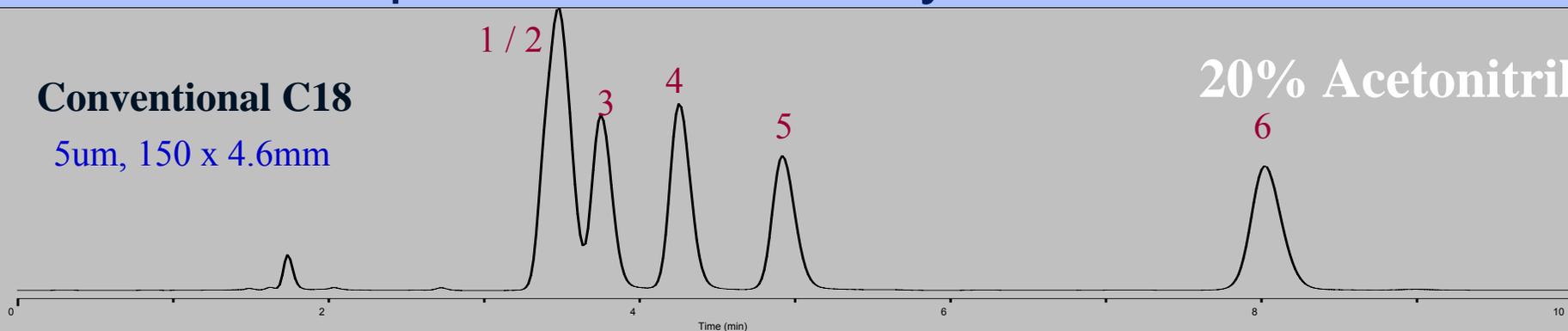


1	sulfadiazine
2	sulfathiazole
3	sulfamerazine
4	sulfamethazine
5	sulfamethoxypyridazine
6	oxytetracycline
7	sulfachloropyridazine
8	sulfamethoxazole
9	chloramphenicol
10	tetracycline
11	sulfadimethoxine
12	sulfaquinoxaline
13	chlortetracycline
14	erythromycin
15	tylosin
16	doxycycline

# Fluoroquinolone Stationary Phase Selection

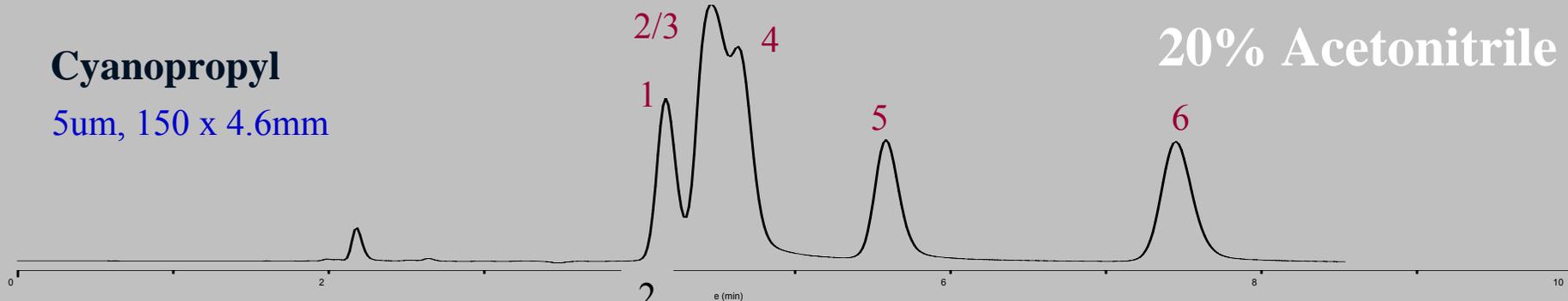
## Conventional C18

5 $\mu$ m, 150 x 4.6mm



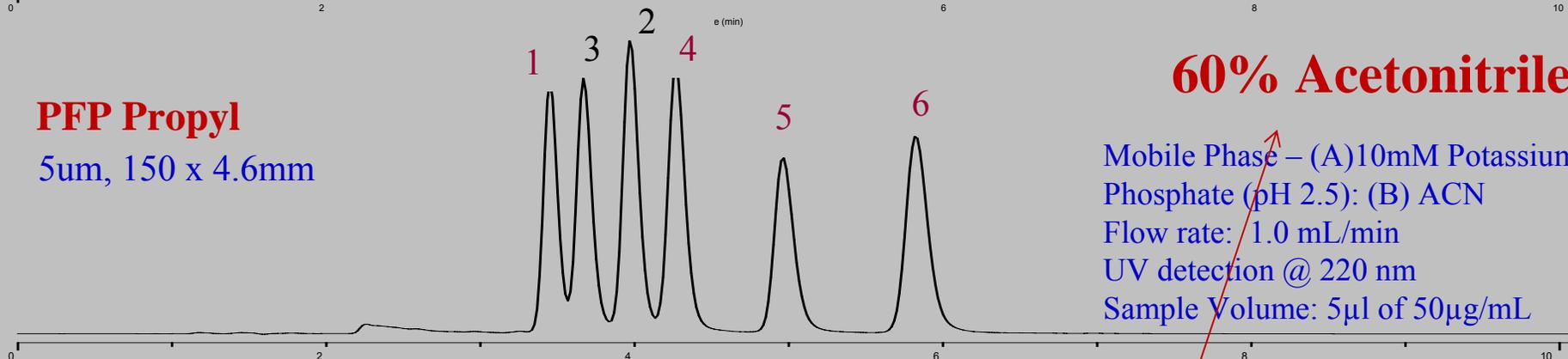
## Cyanopropyl

5 $\mu$ m, 150 x 4.6mm



## PFP Propyl

5 $\mu$ m, 150 x 4.6mm



Mobile Phase – (A) 10mM Potassium Phosphate (pH 2.5); (B) ACN  
Flow rate: 1.0 mL/min  
UV detection @ 220 nm  
Sample Volume: 5 $\mu$ l of 50 $\mu$ g/mL

Selectivity = better MS methods

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# Summary

**Rxi®-5Sil MS**

**Ultra Aqueous C18**

**Application Specific**

**Phases**

required resolution  
Isobaric compounds

required resolution and more  
amenable to LCMS interface



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## Think about...

- Focused or multi-method user = general or specific column
- MS or MS/MS user =
  - column bleed
  - specific problems  
(isobaric compounds and isobaric interferences)
  - LC interface
  - reproducibility
- Low levels = optimize chromatography strategies (S/N)



**QuECh-ing  
for  
Beginners**

**3 Easy Steps**

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# What is QuEChERS?

- Simplified shake extraction of fruits and vegetables for multi-pesticide residue analysis
- Acetonitrile solvent and partition/buffering salts
- Centrifugation
- Dispersive solid phase extraction cleanup
- Extract produced for GC and LC analysis!
- Quick – Easy – Cheap – Effective – Rugged – Safe

## Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and “Dispersive Solid-Phase Extraction” for the Determination of Pesticide Residues in Produce

Author(s): Michelangelo Anastassiades<sup>1</sup> | Steven J. Lehotay<sup>2</sup> | Darinka Štajnbahe<sup>3</sup> | Frank J. Schenck<sup>4</sup>

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Journal of AOAC INTERNATIONAL

Print ISSN: 1060-3271

Volume: 86 | Issue: 2

Cover date: March 2003

Page(s): 412-431

### Abstract

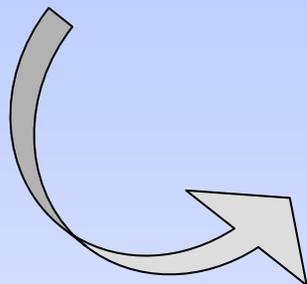
A simple, fast, and inexpensive method for the determination of pesticide residues in fruits and vegetables is introduced. The procedure involves initial single-phase extraction of 10 g sample with 10 mL acetonitrile, followed by liquid-liquid partitioning formed by addition of 4 g anhydrous  $MgSO_4$  plus 1 g NaCl. Removal of residual water and cleanup are performed simultaneously by using a rapid procedure called dispersive solid-phase extraction (dispersive-SPE), in which 150 mg anhydrous  $MgSO_4$  and 25 mg primary secondary amine (PSA) sorbent are simply mixed with 1 mL acetonitrile extract. The dispersive-SPE with PSA effectively removes many polar matrix components, such as organic acids, certain polar pigments, and sugars, to some extent from the food extracts. Gas chromatography/mass spectrometry (GC/MS) is then used for quantitative and confirmatory analysis of GC-amenable pesticides. Recoveries between 85 and 101% (mostly >95%) and repeatabilities typically <5% have been achieved for a wide range of fortified pesticides, including very polar and basic compounds such as methamidophos, acephate, omethoate, imazalil, and thiazendazole. Using this method, a single chemist can prepare a batch of 6 previously chopped samples in <30 min with approximately \$1 (U.S.) of materials per sample.



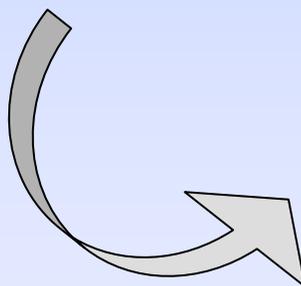
# 1. Blend



Add H<sub>2</sub>O to Leaves



**Blend!**

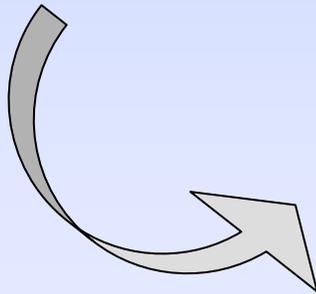


## 2. Extraction



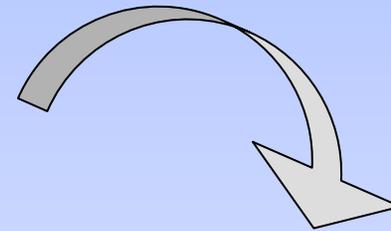
**10g sample + 10mL  
acetonitrile**

**Shake (1 min)**



**Add buffering  
salts to extraction  
sample**

**Shake (1 min)**



**Centrifuge (5 min)**

# 3. Cleanup



**Transfer supernatant to cleanup tube**



**Shake (30 sec)**

**Centrifuge (5 min)**

## Guide to Sorbents

Sorbent	Removes
Primary secondary amine (PSA)	Sugars and fatty acids
Octadecyl (C18)	Lipids
Graphitized carbon black (GCB)	Pigments and sterols

**Transfer cleaned extract to an autosampler vial for analysis**



# Analyzing QuEChERS Samples

- Rxi-5Sil MS
  - Gas chromatography
- Equivalent to popular DB-5MS
  - Selectivity
  - Rxi-5Sil MS is more inert and has lower bleed
- 30m x 0.25mm x 0.25 $\mu$ m suggested
  - Has capacity, is rugged towards “dirt”, inert
- Ultra Aqueous C18
  - HPLC
- Excellent peak shapes for early eluting, polar pesticides!
  - Methamidophos, acephate
- 100mm x 2.1mm, 3 $\mu$ m suggested
  - FDA and AB Sciex SRM and retention time data
    - >280 pesticides
  - Restek FFFF1185.pdf

# QuEChERS

- What is the problem?
  - Most competitors package the extraction salts in 50mL centrifuge tubes.
  - This results in an extra salt transfer step
  - And a wasted tube
- How can we:
  - Make it easier to follow the EN, AOAC, and original unbuffered methods...
  - ...while we reduce waste?



One tube, not two =  
"Greener" solution

Vapor and moisture  
barrier protection



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QuEChERS Extraction Salts

**Q150**

AOAC Method

6g MgSO<sub>4</sub>, 1.5g NaOAc

Pouch cat. - #26238

Pouch and tubes - cat. #26237

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"Packet" only  
packaging allows  
PTFE or FEP tube use

**Q-sep™**

QuEChERS Extraction Salts

**Q110**

EN Method

4g MgSO<sub>4</sub>, 1g NaCl,  
1g Trisodium citrate dihydrate,  
0.5g Disodium hydrogencitrate  
sesquihydrate

Pouch cat. - #26236

Pouch and tubes - cat. #26235

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Salts do not  
clump in  
packets  
(problem for  
Agilent packets)

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# Q-sep Extraction Salt Packets

- Salts are poured from the packet into the tube.
- No wasted tube
- Packets can be purchased by themselves to be used with reusable tubes
- This has also allowed us to dramatically improve our price



Look how happy and productive these lab workers are!



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# Product Offering

- EN and AOAC packets introduced in 2010
- Re-usable FEP tubes in January 2011
- Original unbuffered packets in May 2011

- Current Promotion - Buy one package of reusable FEP tubes, and *get one box of salt packets free.*



# Complete Line of QuEChERS Products

- Extraction Salts
- Empty Polypropylene and FEP Tubes
- 2mL and 15mL dSPE Tubes
- Tube Racks
- Bottle Top Solvent Dispenser
- GC & LC Columns
- Centrifuge
- Standards

**More Samples are available!**



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# Introducing NEW Q-sep™ 3000 Centrifuge for QuEChERS



- Meets AOAC and EU QuEChERS method requirements.
- Supports 50, 15, and 2 mL tubes.
- Small, bench-top footprint.
- Extremely price competitive.





## QuEChERS Standards & Products

[More Information About Restek Q-sep™ QuEChERS Products](#)

[Q-sep™ 3000 Centrifuge for QuEChERS](#)

[Q-sep™ QuEChERS Tubes for Pesticide Residue Analysis](#)

[QuEChERS Internal Standard Mix for GC/ECD Analysis](#)

[QuEChERS Internal Standard Mix for GC/MS Analysis](#)

[QuEChERS Internal Standard Mix for GC/NPD and LC/MS/MS Analysis](#)

[QuEChERS Internal Standard Mix for LC/MS/MS Analysis \(nicarbazin\)](#)

[QuEChERS Quality Control Standards for GC/MS Analysis](#)

[QuEChERS Single-Component Reference Standards](#)

### Featured Products

#### Q-sep™ 3000 Centrifuge

- First centrifuge specifically designed for QuEChERS methodology
- Compact, quiet, yet powerful unit spins at 3000g force required by the European method.

[Products](#)

#### QuEChERS Internal Standards and Quality Control Standards

- Single and multiple component standards as specified in EN Method 15662.
- Support for GC and HPLC with MS, MS/MS, and selective detectors.
- Ready to use for QuEChERS extractions—no dilutions necessary.

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Complete reference materials support for European Standard 15662 QuEChERS method!

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**Please note:** Restek will be closing at 3:30pm on May 20, while our employee-owners attend the ESOP shareholders meeting. Orders placed after 2:00pm will be processed Friday, May 21.

< [Food, Flavor, and Fragrance Reference Standards](#) < [QuEChERS Standards & Products](#)



## QuEChERS Internal Standard Mix for GC/MS Analysis

Pesticide analysis is fast and simple using QuEChERS methods. Use these cost-effective QuEChERS standards for even greater lab efficiency. Standards are compatible with all major methods, including mini-multiresidue, AOAC, and European procedures. Save time with convenient mixes or make your own blend using our full line of single component solutions.

- Ready to use for QuEChERS extractions--no dilutions necessary.
- Support for GC and HPLC with MS, MS/MS, and selective detectors.

PCB 18 50µg/mL

PCB 28 50

PCB 52 50

triphenyl phosphate 20

tris-(1,3-dichloroisopropyl)phosphate 50

triphenylmethane 10

[Chromatogram](#)
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Description	Conc. in Solvent/Volume	qty.	Cat.#	Price	Wish list	Cart
QuEChERS Internal Standard Mix for GC/MS Analysis	In acetonitrile, 5mL/ampul	ea.	33267	\$30.00	<input type="checkbox"/>	

Restek is currently only supplier.  
Snap-and-shoot standard additions.

# New QuEChERS Products in Development

COMING SOON

- More dSPE products
- Vials
- Shaker
- More standards



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# Other Applications for QuEChERS

- Antibiotics in honey
- Pesticides in olives and olive oil
- Acrylamide in food
- Flukicides and anthelmintics in meat and milk
- Veterinary drugs in meat
- Pharmaceuticals in whole blood
- Pesticides in marijuana



# Applications

- New Blog content regularly
- Restek.com/quechers
- Dietary Supplements
- Tylenol
- Cannabananoids
- Shellfish



# Restek ChromaBLOGraphy

- Developed by Kent Rauch
  - Restek webmaster extraordinaire
- Posts from Restek Innovations staff
- Often contains pertinent (but unreleased) technical info
- Heavily food safety oriented
  - QuEChERS, pesticides, injection techniques
- Restek exclusive!



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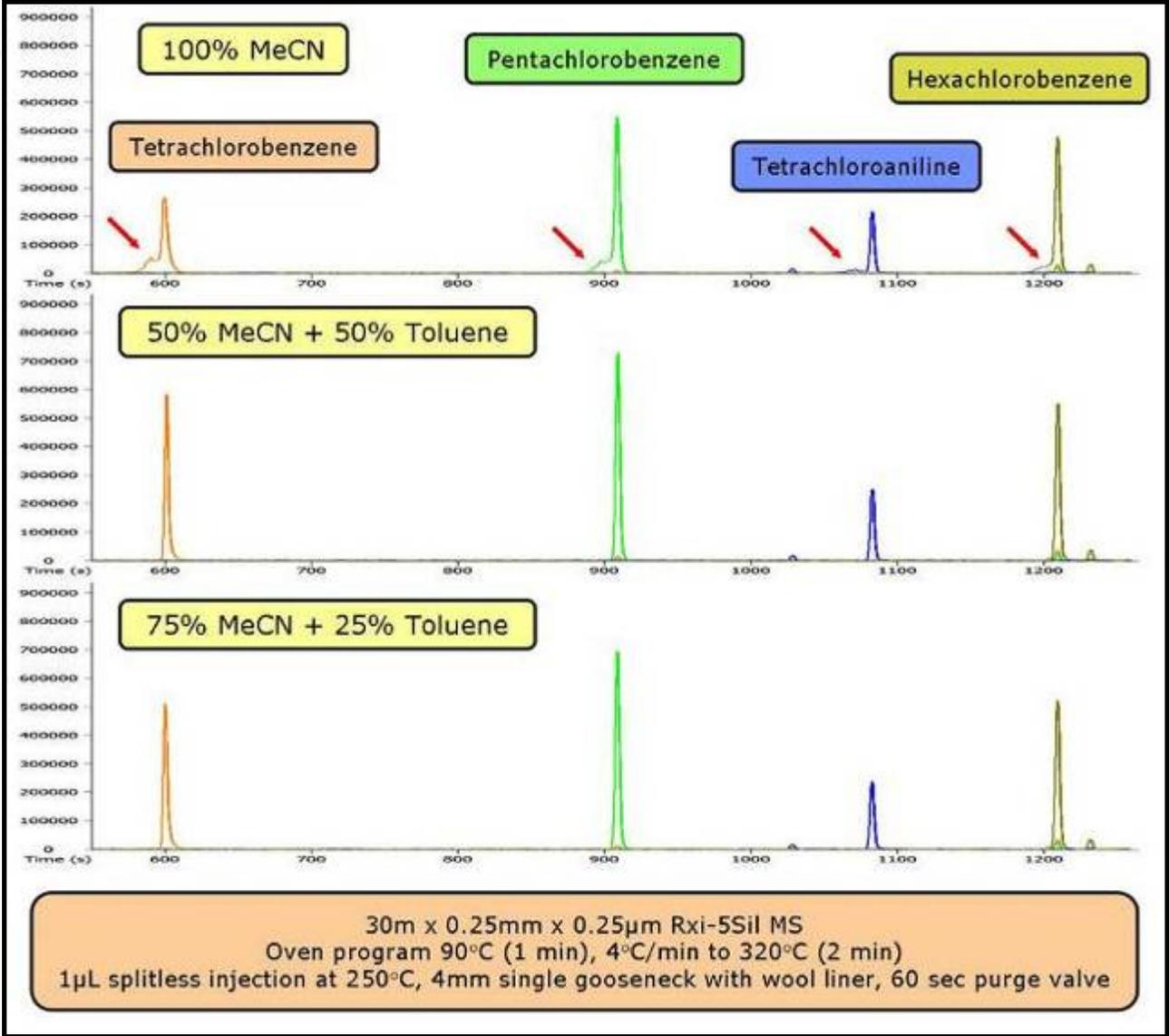


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not

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# Fast Screening of Recalled Tylenol® for Tribromoanisole and Related Adulterants

## Using QuEChERS and GC-TOFMS

- Rapid sample preparation with QuEChERS improves turnaround time for emergency response analysis situations.
- Prepackaged QuEChERS extraction salts and snap-and-shoot standards reduce human error and save time.
- Rugged, inert, thermally stable Rxi®-5Sil MS column extends applicability to acids, bases, and higher molecular weight adulterants.

### Introduction

The recent recall of Tylenol® pain reliever and other related products highlights the need for simple, quick sample preparation and a comprehensive analytical method for adulterants in consumer products. The rush to examine a multitude of samples in a short period of time is a common scenario for potential recalls, especially when a contaminant is found in a given product and rapid determinations need to be made to assess how widespread the problem may be.

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) sample preparation approach, originally developed to prepare fruit and vegetable samples for pesticide residue analysis, is being adopted for other applications and may be useful when rapid screening methods are required. QuEChERS employs a simple solvent shake and centrifugation step, with an optional dispersive solid phase extraction (dSPE) cleanup. In addition to being quick and easy, the use of acetonitrile in QuEChERS allows compounds containing a wide variety of chemical functionalities to be extracted, which is very important when trying to isolate an unknown adulterant. The resulting extract is appropriate for both GC/MS and LC/MS work.

The utility of the QuEChERS method is illustrated here using the aforementioned Tylenol® example, showing the applicability to this problem and, by extension, to others like it. This particular recall was due to the presence of 2,4,6-tribromoanisole (TBA) causing a musty smell in the product and, in some cases, nausea in the consumer [1]. TBA is a known breakdown product of 2,4,6-tribromophenol (TBP), which is a fumigant used on shipping pallets; TBA production occurs through a process actuated by a fungus, *Paecilomyces variotii* [2]. TBA is a common and undesirable odorant in the winemaking industry where

# QuEChERS and Cartridge SPE Cleanup for Pesticides in Dietary Supplements

Michelle Misselwitz, Julie Kowalski, Jack Cochran,  
Jason Thomas, Rick Lake

Restek Corporation, Bellefonte, Pennsylvania

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# Sample Preparation of Cannabinoids in Urine Using the QuEChERS Method



Jack Cochran, Amanda Rigdon, Kristi Sellers

Restek Corporation, Bellefonte, Pennsylvania

American Academy of Forensic Sciences Annual Scientific Meeting 2009



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## Comprehensive Pesticide Residue Analysis by LC/MS/MS

Using an Ultra Aqueous C18 Column

By Becky Wittig, Ph.D., Global HPLC Specialist, and Andre Schrabler, Ph.D., Applied BioSystems/MSD Analytical Technologies

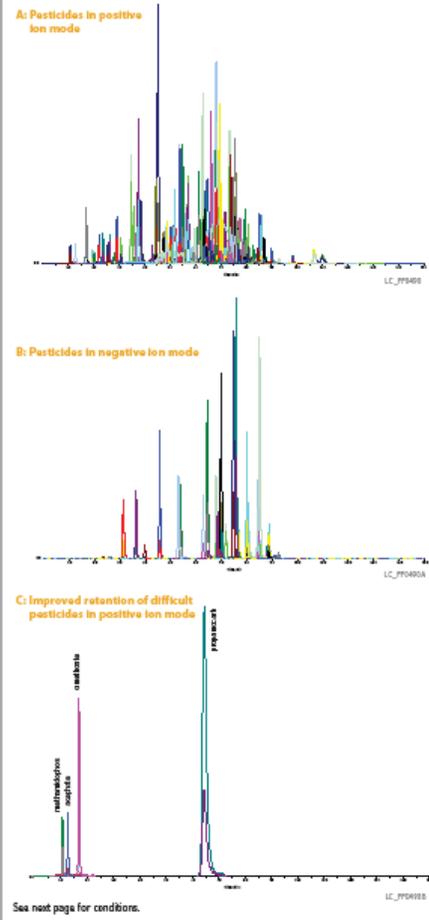
- Easily resolve and quantify more than 280 pesticide species.
- Use LC/MS/MS to reliably monitor difficult polar and/or thermally unstable species.
- Aqueous C18 phase offers optimal selectivity and retention.

Food safety is a topic of great interest globally. With recent contamination issues in a wide range of commodities, ensuring the quality of our food supply is becoming increasingly important. Pesticide residue content is one area of concern. While pesticides have typically been monitored by gas chromatography, polar and/or thermally unstable pesticides are difficult or impossible to monitor using this approach. Thus, traditional HPLC techniques are used for select pesticide classes, such as the carbamate and phenylurea pesticides.

With recent advances in LC/MS/MS instrumentation, this technique is quickly gaining acceptance for pesticide residue testing. LC/MS/MS can be used to simultaneously monitor hundreds of potential contaminants—including those difficult to detect by GC. Using both LC/MS/MS and GC approaches allows for a faster, more complete picture of pesticide residues. MS/MS technology also permits identification of the target pesticides through the selection of specific MRM transitions for each compound. For example, aldicarb, a carbamate pesticide, uses two MRM transitions of 208.2→89.1amu and 208.2→116.1amu.

While the MS/MS detector allows for specific, sensitive detection of the pesticide species, the LC separation is still important to ensure the highest quality data. Conventional C18 stationary phases are typically used for pesticide monitoring, but the selectivity and retention is poor for more polar species. In contrast, Ultra Aqueous C18 columns are ideal for multi-pesticide residue monitoring methods. In Figure 1, the analysis of more than 280 pesticides using the 3µm Ultra Aqueous C18 is shown. Optimized stationary phase selectivity

**Figure 1** More than 280 pesticide residues—including difficult polar species—show excellent peak shape and retention on a 3µm Ultra Aqueous C18 column.



### Conditions for Figure 1 (previous page):

**Sample:** multi-component pesticide standard  
**Inj.:** 10µL  
**Conc.:** 1µg/µL each pesticide  
**Sample diluent:** acetonitrile  
**Column:** Ultra Aqueous C18  
**Col. #:** 017831.L  
**Dimensions:** 100mm x 2.1mm  
**Particle size:** 3µm  
**Pore size:** 100Å  
**Outlet:** Ultra Aqueous C18  
**Dimensions:** 100mm x 2.1mm  
**Particle size:** 3µm  
**Pore size:** 100Å  
**Instrument:** Shimadzu Prominence® IFLC-01  
**Mobile phase:** A: 10 mM NH<sub>4</sub>CO<sub>3</sub> in water  
 B: 10 mM NH<sub>4</sub>CO<sub>3</sub> in methanol  
**Time (min.)** **SR**  
 0.0 20  
 8.0 90  
 12.0 100  
 34.0 100  
 34.0 20  
**Flow:** 500µL/min  
**Temp.:** 25°C  
**Det.:** Applied Biosystems 4000 QTRAP®  
 LC/MS/MS system  
 TurboIonSpray  
 A & C: ESI+  
 B: ESI-  
**Ion Spray Voltage:** 5kV (ESI+), -4.2kV (ESI-)  
**Gas 1:** 50psi  
**Gas 2:** 60psi  
**Source Temp.:** 600°C

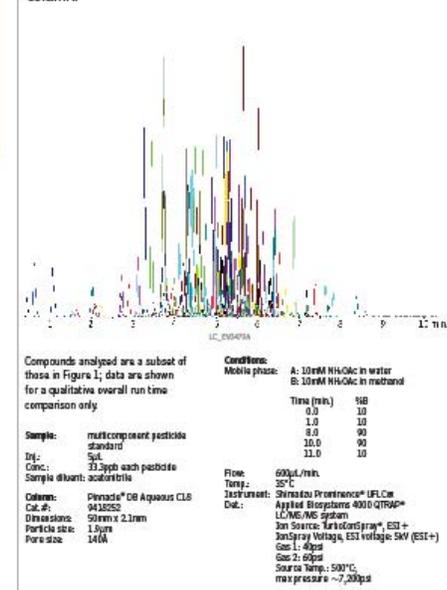
**Table I** Peak list for pesticides in negative ion mode.

Compound ID	Retention Time (min)	Transition 1	Transition 2
Azinthrin	9.09	540 → 372	540 → 372
Chlorfloxuron	9.24	539.9 → 356.8	539.9 → 356.8
Cyfluthrin	3.63	249.9 → 58	247.9 → 59
Diflufenican	7.48	319 → 156.1	310 → 286.9
Duron	6.28	220.9 → 185.8	220.9 → 140.8
Fenoxypipron	7.90	462.5 → 415.8	462.5 → 397.9
Fenprothiazin	6.93	246.9 → 179.9	246.9 → 125.9
Forchlorfenuron	6.44	246 → 126.9	246 → 91.2
Hexachlorocyclopentadiene	8.65	451 → 436.8	451 → 174.9
Zimprovacarb	8.82	409 → 250.9	411 → 253
Lufenuron	8.87	509 → 325.9	509 → 175
Metachlorophenol	8.5	505.1 → 301.9	505.1 → 140.9
Metolachlor	3.97	203 → 184.8	203 → 116.9
Methoxyfenozid	6.98	265.9 → 104.9	266.9 → 146.9
Milbexipyr	2.59	269.2 → 222.6	269.2 → 108.8
Novalon	8.42	491.1 → 470.7	492.1 → 472.7
Proflufenisid	7.57	464.3 → 277.9	464.3 → 126.8
Propiconazole	3.09	296.9 → 156	296.9 → 112.9
Triphenolmethane	7.16	342 → 106.1	342.9 → 99.9
Tebuconazole	7.66	350.9 → 149	350.9 → 105
Tebuconazole	8.85	378.6 → 318.8	378.6 → 195.9
Tepralozin	4.57	340 → 248	340 → 220.1
Terbufos	5.38	214.9 → 158.9	216.9 → 160.9
Trioxystrobin	6.28	328.1 → 253.8	328.1 → 202.8
Trifloxystrobin	7.01	292 → 47.9	292 → 234.9
Trifluralin	8	356.9 → 153.8	358.9 → 153.9

allows for an even distribution of the compounds throughout the retention time window (Tables I and II). As well, retention of more polar pesticides is greatly improved, as demonstrated in Figure 1C. The Ultra Aqueous C18 column, in a 100 x 2.1mm, 3µm configuration is the column of choice for LC/MS/MS pesticide monitoring methods.

Ultra-high pressure LC (UHPLC) can also be used with MS/MS detection for monitoring pesticide residues. UHPLC allows for higher sample throughput when used in conjunction with a highly efficient <2µm particle size column. The 1.9µm Pinnacle® DB Aqueous C18, in a 50 x 2.1mm configuration, is ideally suited for this application, as shown in Figure 2.

**Figure 2** Higher sample throughput can be achieved using UHPLC and MS/MS with a 1.9µm Pinnacle® DB Aqueous C18 column.



Using LC/MS/MS technology and Aqueous C18 columns, in combination with gas chromatography, results in the most comprehensive monitoring of pesticide residues. Labs interested in more complete multi-residue analysis of pesticides in food matrices, including difficult polar or thermally unstable compounds, should consider adding LC/MS/MS and Aqueous C18 columns to routine testing procedures. The Aqueous C18 phase is also available on 1.9µm Pinnacle® DB silica for UHPLC platforms.

### Acknowledgements

The authors wish to thank the US FDA for their collaboration and recognize the participation of multiple FDA labs in this work.



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### Q-sep™ QuEChERS Extraction Salts

Fast, Simple Sample Prep for Multi-Residue Pesticide Analysis

- 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.
- Ready-to-use tubes, no glassware required.
- Preweighed, ultra-pure sorbents.
- Support original unbuffered, AOAC (2007.01) and European (EN 15662) QuEChERS methods.

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**Q-sep™** innovative chromatography solutions

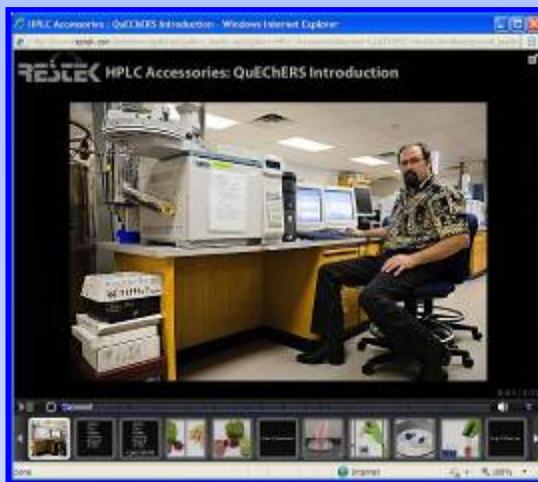
## QuEChERS Products

Fast, Simple Sample Prep for Multi-Residue Pesticide Analysis

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### Optimizing Dispersive Solid Phase Extraction and Splitless Injection-Gas Chromatography for QuEChERS Fruit and Vegetable Extracts

Jack Cochran, Julie Kowalski, Lydia Nelson

**Introduction**  
Introduction of the QuEChERS method for the analysis of pesticides in fruits and vegetables has revolutionized the field. This method is simple, fast, and efficient, and it has become the standard for the analysis of pesticides in these complex, highly botanical products. QuEChERS offers a simple, cost-effective approach that can reduce matrix interferences as well as variation among technicians. Here we demonstrate a procedure that incorporates a QuEChERS extraction, cSPE cleanup and GC-TOFMS, resulting in good recoveries for a wide range of pesticide chemistries in dandelion root powder.

**Results and Discussion**  
Results and Discussion

**Standards and Samples**  
Standards and Samples

**Sample Preparation**  
Sample Preparation

**Conclusion**  
Conclusion

**Table 1: Comparison of PAM 303 and QuEChERS + cSPE**

	PAM 303 Method	QuEChERS + cSPE	Benefits of QuEChERS + cSPE
Solvent used (ml)	1,200	12	20x less solvent, cleaner, greener, & cost-effective
# of Solvents	4	3	
Salt and sorbent used (g)	25	6.6	75% less salt/sorbent
Chromatography equipment	• Separatory funnel (1L capacity) • Filter apparatus • Filter column	• Cartridge • SPE manifold	Fewer, easy batch processing
Manual preparation	• Salt solution • Standards • Filter column	None—prepackaged salt and cSPE cartridge are ready to use	Highly reproducible, less manual prep means less human error

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## Applications Note

### Developing New Methods for Pesticides in Dietary Supplements

#### Advantages of the QuEChERS Approach

New requirements for dietary supplements to be manufactured under cGMP regulations have created a need for methods to detect pesticides in these complex, highly botanical products. QuEChERS offers a simple, cost-effective approach that can reduce matrix interferences as well as variation among technicians. Here we demonstrate a procedure that incorporates a QuEChERS extraction, cSPE cleanup and GC-TOFMS, resulting in good recoveries for a wide range of pesticide chemistries in dandelion root powder.

**Introduction**  
Recently the FDA announced that makers of dietary supplements (e.g. vitamins, herbal and botanical pills, etc.) will have to adhere to current Good Manufacturing Practice (cGMPs), marking a major shift in regulatory oversight and testing for the industry. Previously, compliance was voluntary, but in 2003, due to public and industry concern, the FDA proposed requiring dietary supplement manufacturers to adhere to cGMP standards. The final rule was issued in June 2007 and is in full effect June 2010 [1]. Basic cGMPs require implementing comprehensive procedures to ensure product quality and safety. Since many dietary supplements are largely derived from botanical sources, they must be tested for pesticide contaminants in order to meet cGMP regulations. As a result of this requirement, labs are working to develop and validate methods, an endeavor which is complicated by the wide range of pesticides and matrices to be tested.

Labs can begin method development with the FDA Pesticide Analytical Manual (PAM), which includes procedures for plant materials. While PAM Method 303 is an appropriate starting point, it has several disadvantages, including high solvent consumption, manual procedures that contribute to analytical variation, and the inability to extract polar pesticides. As an alternative, we developed a QuEChERS-based method for analyzing pesticides in dietary supplements that has several advantages over PAM 303 (Table 1). QuEChERS is an approach that was developed by the USDA Eastern Regional Research Center as a simple, rapid, effective, yet inexpensive way to extract pesticide residues from fruits and vegetables, followed by a novel dispersive solid phase extraction (dSPE) cleanup of the extract. Because of these benefits, the approach has become popular and has been expanded to include numerous other matrices. We chose QuEChERS as an alternative to PAM 303 because of its speed, simplicity, and low solvent use, as well as its ability to produce good extraction efficiencies for relatively polar pesticides [2].

Based on preliminary studies, we know that while the extraction part of QuEChERS would be successful, the dSPE cleanup step probably did not have the capacity to handle the matrix complexity of most dietary supplements. Thus, we compared dSPE to a cartridge solid phase extraction (cSPE) cleanup and established a procedure that uses a QuEChERS extraction, cSPE cleanup, and GC-TOFMS for accurate determinations of 46 pesticides in dandelion root powder. This approach saves time and can reduce analytical variation by minimizing manual preparation with prepackaged extraction salts and snap-and-shoot standards. As shown in Figure 1, it also uses much less solvent, salt, and sorbent, making it a greener, more cost-effective method than PAM 303.

**Table 1: Decrease costs and increase reproducibility with a GMP-Friendly QuEChERS approach to analyzing pesticides in dietary supplements.**

	PAM 303 Method	QuEChERS + cSPE	Benefits of QuEChERS + cSPE
Solvent used (ml)	1,200	12	20x less solvent, cleaner, greener, & cost-effective
# of Solvents	4	3	
Salt and sorbent used (g)	25	6.6	75% less salt/sorbent
Chromatography equipment	• Separatory funnel (1L capacity) • Filter apparatus • Filter column	• Cartridge • SPE manifold	Fewer, easy batch processing
Manual preparation	• Salt solution • Standards • Filter column	None—prepackaged salt and cSPE cartridge are ready to use	Highly reproducible, less manual prep means less human error

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Application notes, chromatograms, flyers, mini-catalogs, tear sheets, technical guides, audio slideshows, bibliographies, web links, instruction sheets, presentations



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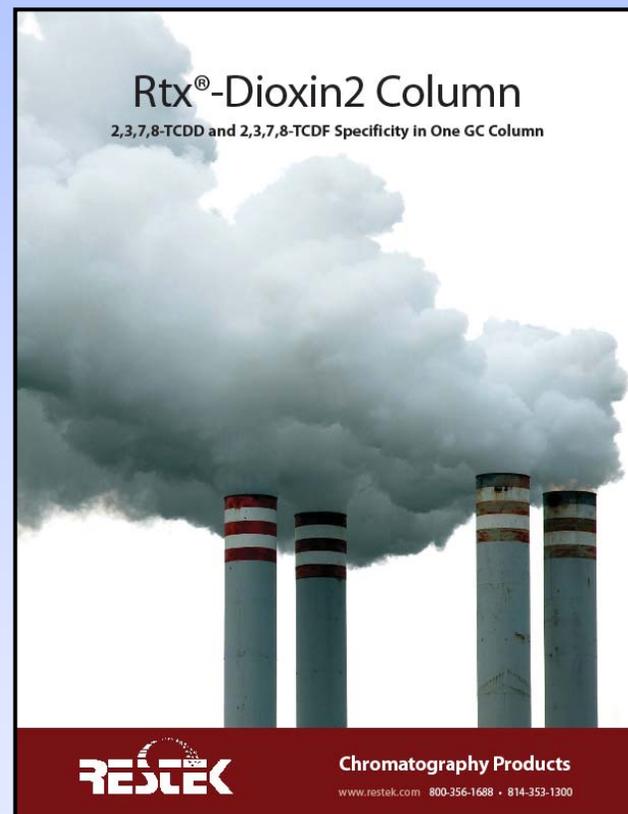
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# Chlorinated Dioxins and Furans in Food

## Restek GC Selectivity Solutions

- Determine 2378- TCDD and TCDF in one GC run
  - Rxi-5Sil MS (60m x 0.25mm x 0.25 $\mu$ m)
    - Primary column
    - Also does 12378 penta dioxin (very toxic)
  - Rtx-Dioxin2 (60m x 0.25mm x 0.25 $\mu$ m)
    - Secondary column for TCDF confirmation
- Customer already using DB-5MS
  - Selectivity equivalent to Rxi-5Sil MS
  - Rxi-5Sil MS is more inert, lower-bleed
- Rtx-Dioxin2 is a Restek exclusive!
  - Fully characterized for all 136 Cl<sub>4</sub> to Cl<sub>8</sub> chlorinated dioxins and furans



Rtx<sup>®</sup>-Dioxin2 Column  
2,3,7,8-TCDD and 2,3,7,8-TCDF Specificity in One GC Column

**RESTEK** Chromatography Products  
www.restek.com 800-356-1688 • 814-353-1300

# USPs for Restek Rxi-5Sil MS GC Column for Chlorinated Dioxins and Furans

- Replacement for DB-5
  - Specified in EPA Methods 1613 and 8290a
  - Primary column, but coelution on 2378 TCDF
- Rxi-5Sil MS separates 2378 TCDD and TCDF
- Rxi-5Sil MS separates 12378 PnCDD
- Rxi-5Sil MS is more inert and has lower bleed than DB-5

Rxi-5Sil MS has equivalent selectivity to DB-5MS and other "5" silphenylene columns.



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# USPs for Restek Rtx-Dioxin2 GC Column

- Replacement for DB-225, SP-2330/2331
  - Used for 2378 TCDF confirmation
  - High cyano content puts max temps at 220-270
- Rtx-Dioxin2 separates 2378 TCDF
- Rtx-Dioxin2 has high max temp of 340°C
- Rtx-Dioxin2 partially separates 23478 PnCDF
  - Sometimes an important congener
- Rtx-Dioxin2 is a Restek exclusive



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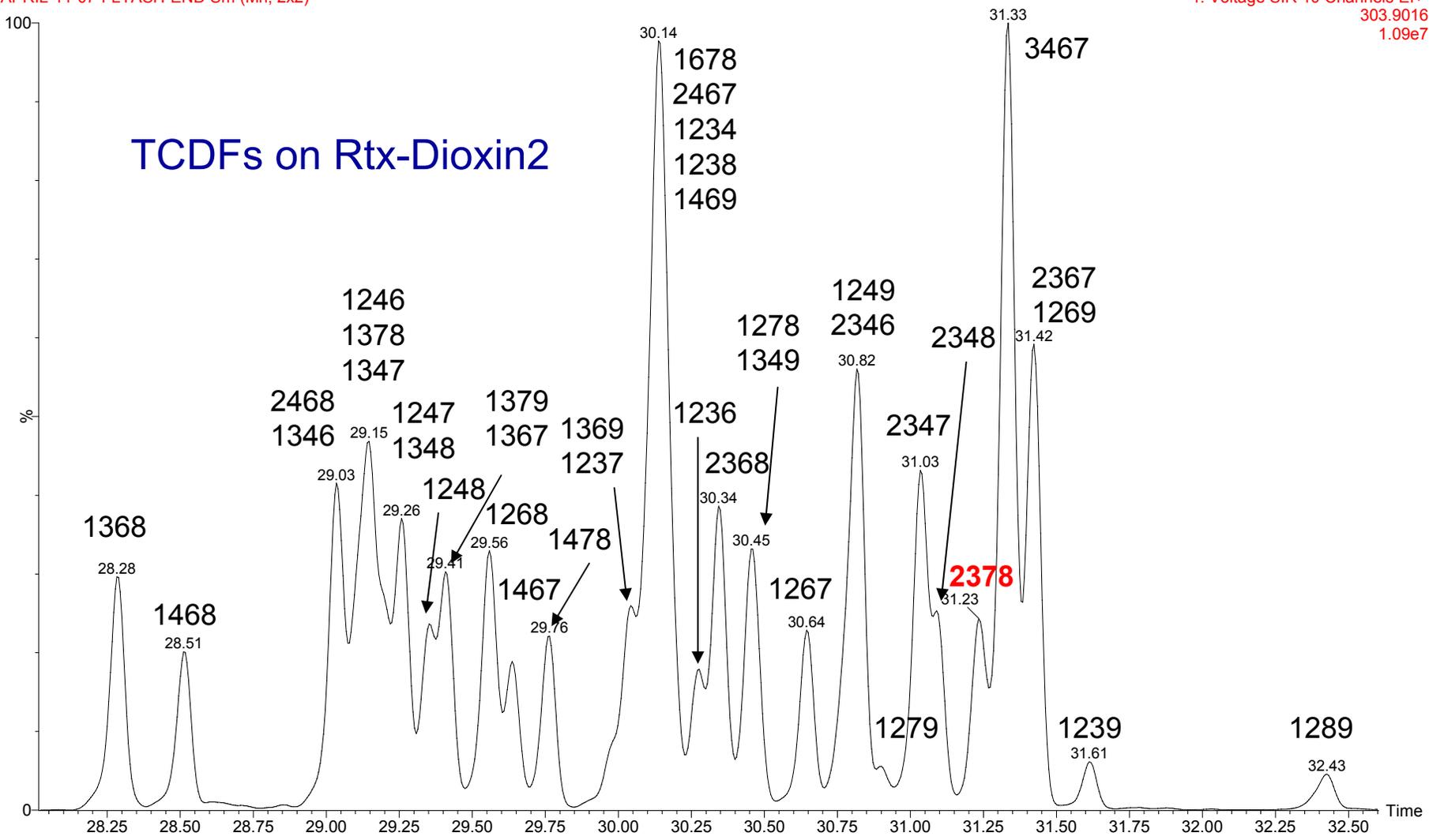


FLYASH-C149823-0006

APRIL-11-07-FLYASH-END Sm (Mn, 2x2)

1: Voltage SIR 10 Channels EI+  
303.9016  
1.09e7

# TCDFs on Rtx-Dioxin2

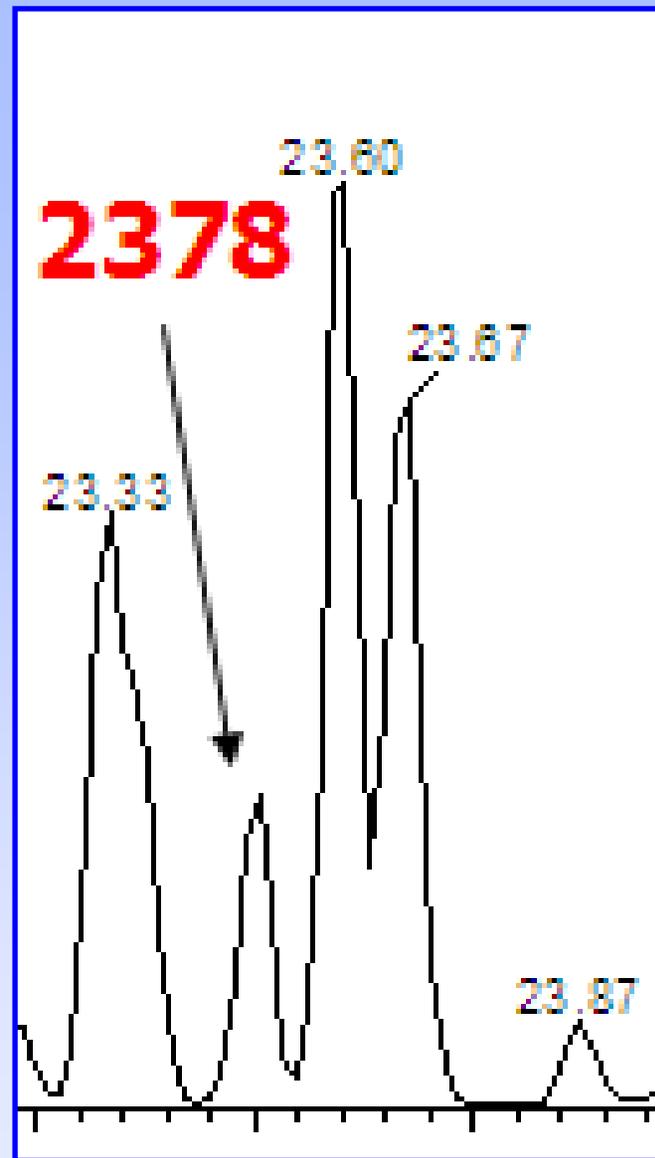
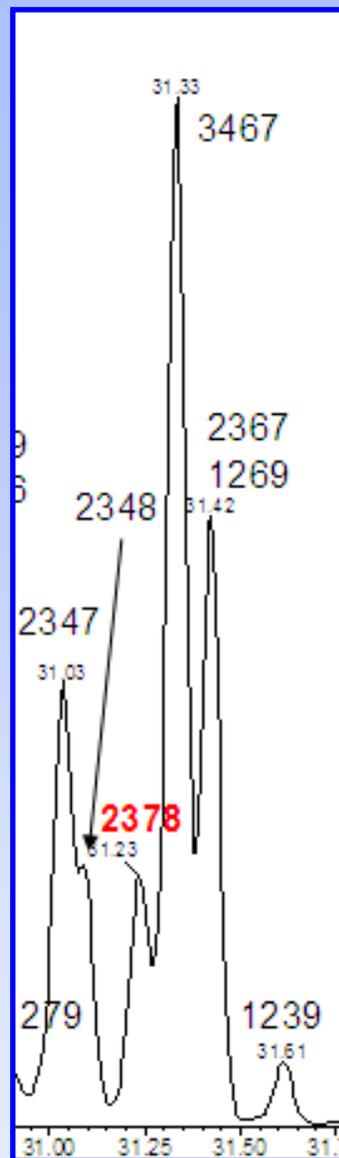


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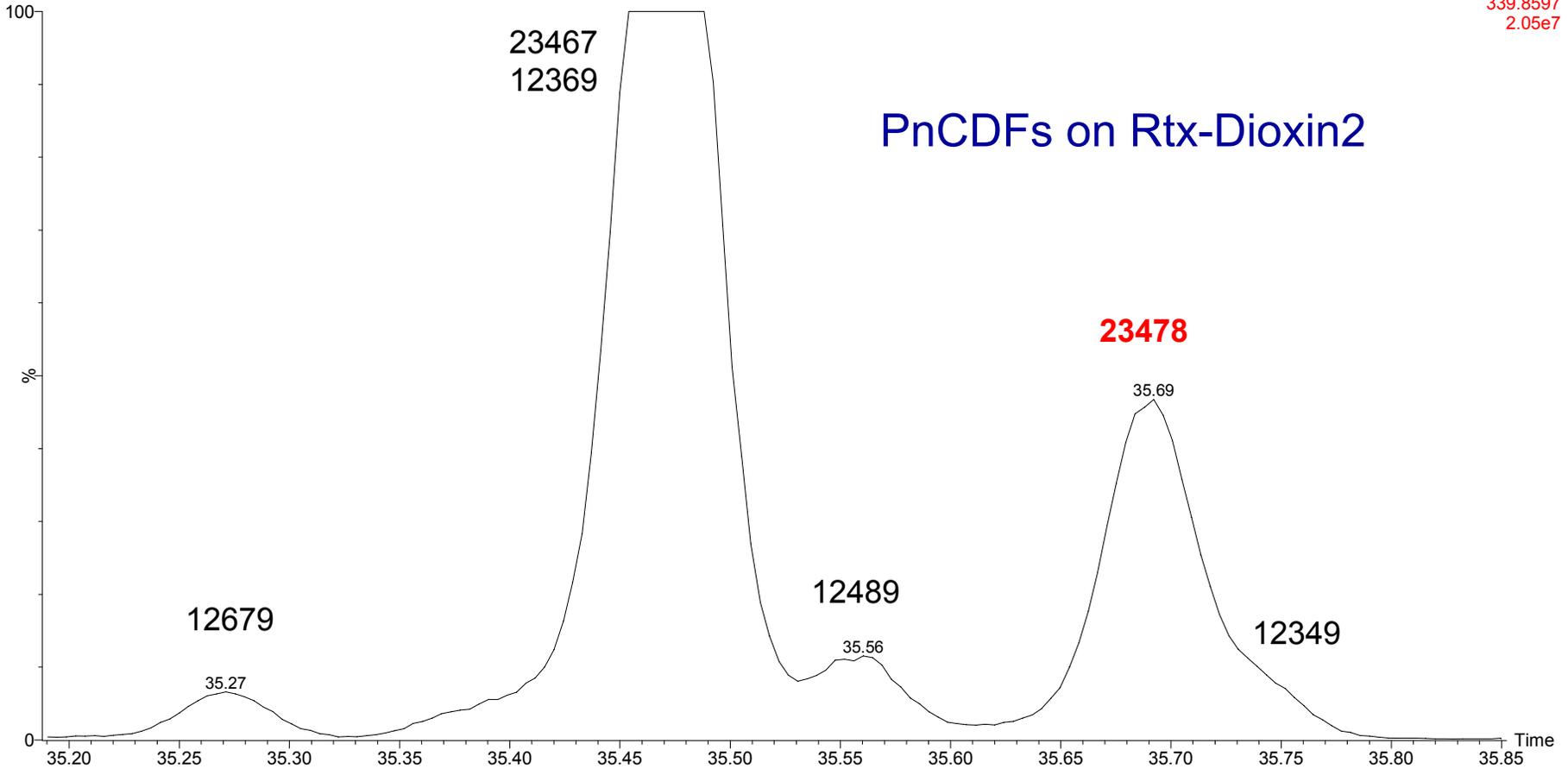
- 2378 TCDF in fly ash on Rtx-Dioxin2 under different GC conditions
- Flow and temperature program optimization can improve separations



FLYASH-C149823-0006

APRIL-11-07-FLYASH-END

2: Voltage SIR 8 Channels EI+  
339.8597  
2.05e7



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# Dioxin contamination scandal hits Belgium

1999

## Effects spread through European Union and beyond

*By Richard Tyler*  
8 June 1999

Farms in Belgium have been ordered to destroy livestock that were given animal feeds believed to be contaminated with dioxin, a serious carcinogen. The European Union has ordered a complete ban on Belgian agricultural exports of eggs, chickens, pork and beef. All chickens and eggs exported after January 15 have been ordered destroyed and this may be widened to include pigs, cattle and other foods containing possibly tainted ingredients.

# Polychlorinated Biphenyls in Food

- Do you need to do congener-specific PCB analyses in fish or other animal tissues?
- Do you need European indicator congener separations?
  - 31/28, 153/132, 163/138
- Rtx-PCB is a Restek exclusive!
  - Separates difficult congeners
  - Elution orders for all 209 PCBs
  - Excellent when combined with MS



## THE RESTEK ADVANTAGE

Turning Visions into Reality™  
2005 vol. 2

### Rtx®-PCB: Unique Selectivity for PCBs

110 of 158 target PCB Congeners Elute Individually, Using GC/ECED

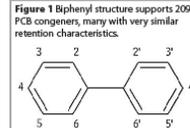
by Gary Siders, GC Columns Marketing Manager

- NEW low polarity, inert polymer phase provides distinct separations of PCB congeners.
- Unmatched selectivity and low bleed—a column of choice for trace analysis.
- Thermally stable to 340°C.

Rtx®-PCB columns show unique selectivity for polychlorinated biphenyl (PCB) congeners. In previous publications (Lit.# 59925 and Advantage 2005v1, Lit.# 59077, page 13), we discussed the excellent performance of this column and showed each of the European PCB congener indicator compounds—PCBs 28, 52, 101, 118, 136, 153, and 180—can be resolved from other, interfering PCB congeners and quantified, using GC/MS.

“Weathering” of Aroclor® mixes that have been in the environment for more than 50 years, and changes in Aroclor® patterns in tissue samples, due to bioaccumulation, have dictated that PCBs now be reported as congeners, rather than as Aroclor® mixes. Consequently, many laboratories are analyzing longer lists of PCB congeners, using the data in determining specific congener patterns, in compiling congener results to obtain an accurate total PCB concentration, and in other ways.

**Figure 1** Biphenyl structure supports 209 PCB congeners, many with very similar retention characteristics.



The structure of the biphenyl molecule is shown in Figure 1. Identification and quantification of PCB congeners is chromatographically challenging because there are 209 possible combinations in which chlorine atoms can be added to the biphenyl structure, ranging from addition of a single chlorine (monochlorobiphenyls) to addition of every available carbon atom (decalchlorobiphenyl). In 1996, George Frame published work he performed in order to determine which PCB congeners are present in Aroclor® mixes. Using this work as a guide, the list of 209 possible PCB congeners can be reduced to a target list of 158 congeners. This final target list includes PCB congeners found in Aroclor® mixes above 0.01%wt/wt, and a few compounds not detected in Aroclor® mixes, but detected in tissue due to

### in this issue

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**Restek Goes West!**



Restek celebrates continued growth in 2005 with the opening of Restek West, our new R&D facility in Shingler Springs, California. Roy Leutemio, Director of Innovative Research Chromatography, is managing the facility, focusing on R&D for our chromatography column product lines. Roy has an extensive range of experience in chromatography, acquired over more than a quarter of a century of research.

We welcome Roy and his staff into the Restek family!

**Correction**

In Advantage 2005v1, Fast GC/MS Analysis of Semivolatile Organic Compounds, Figure 1 (page 14) the splitless hold time and pressure pulse time are reversed. The splitless hold time should be 0.15 min, and the pressure pulse time should be 0.20 min. We apologize for any inconvenience caused by this error.



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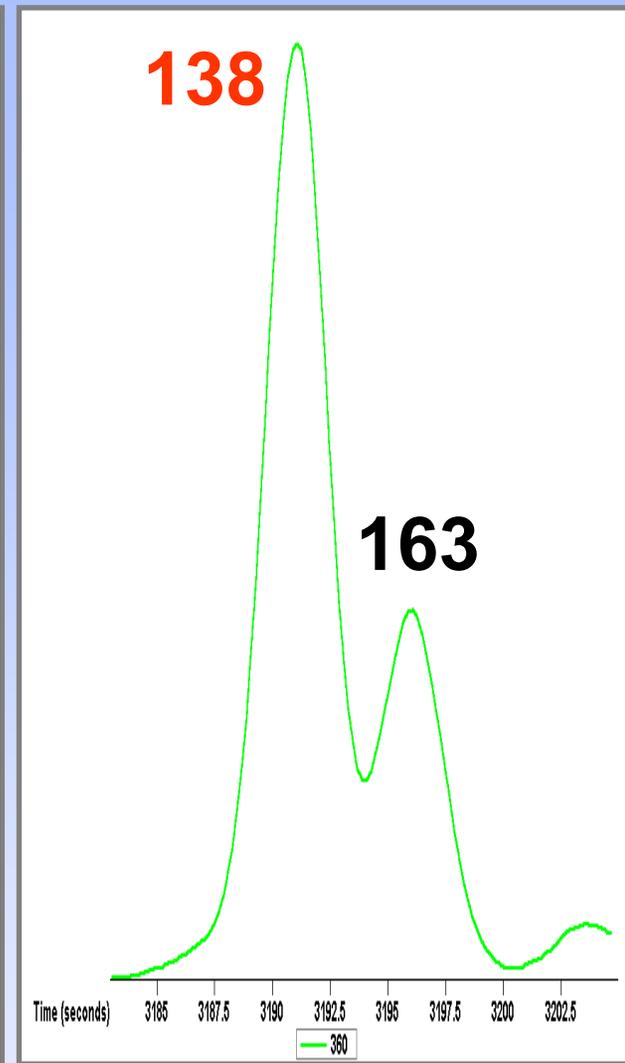
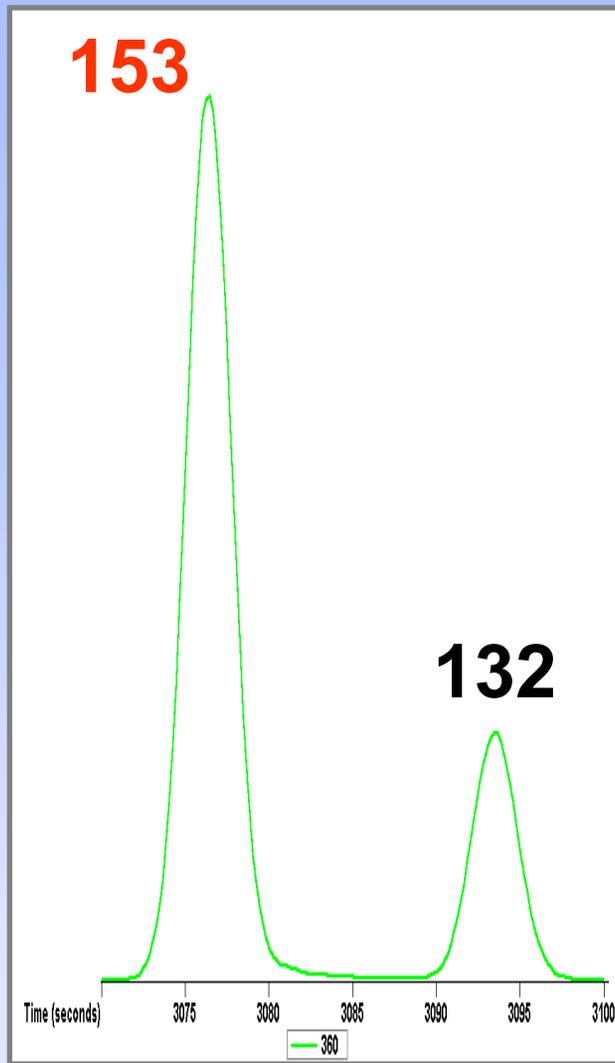
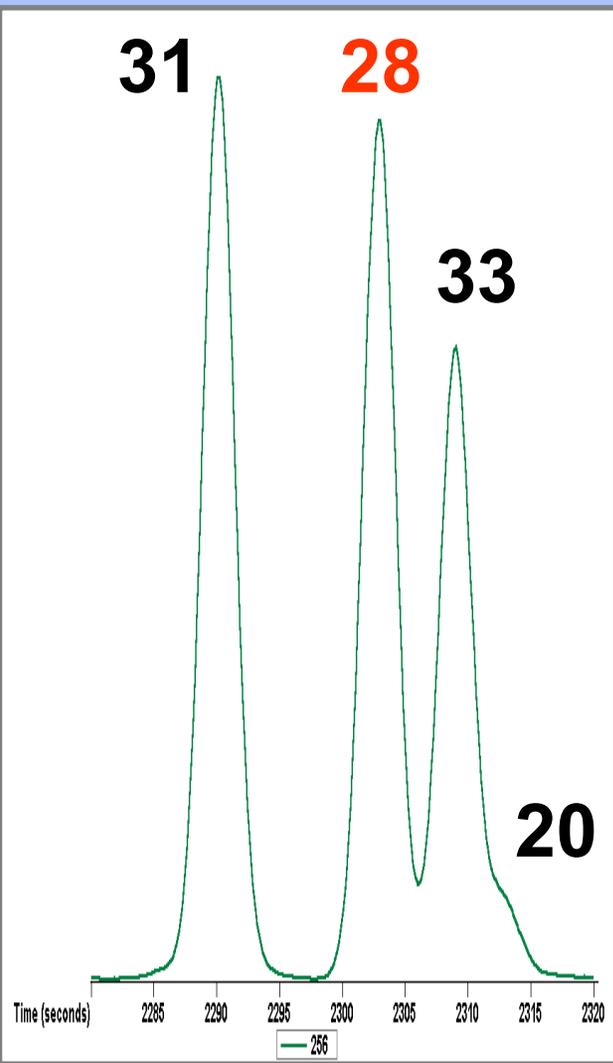
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European Indicator PCB Congeners Separated on Rtx-PCB



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# USPs for Restek Rtx-PCB GC Column

- Replacement for 5 and 5Sil columns, etc.
  - Do not separate European congeners
  - Especially 31/28, 153/132, and 163/138
- Rtx-PCB separates EU indicator PCBs
  - When used with MS
- Rtx-PCB is a Restek exclusive
- Watch for SGE HT8 and J&W DB-XLB
  - Restek has Rxi-XLB



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## Polycyclic Aromatic Hydrocarbons in Food<sup>1</sup>

### Scientific Opinion of the Panel on Contaminants in the Food Chain

(Question N° EFSA-Q-2007-136)

Adopted on 9 June 2008

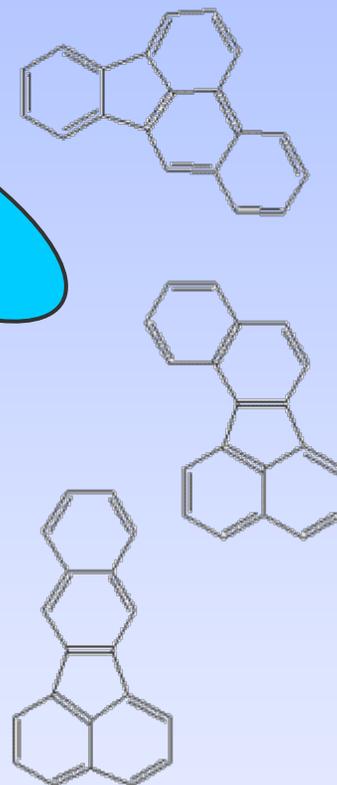
benzo(a)anthracene; benzo(b)fluoranthene;  
benzo(j)fluoranthene; benzo(k)fluoranthene;  
benzo(ghi)perylene; benzo(a)pyrene; chrysene;  
dibenz(a,h)anthracene; dibenzo(a,e)pyrene;  
dibenzo(a,h)pyrene; dibenzo(a,f)pyrene;  
dibenzo(a,l)pyrene; 5-methylchrysene

# GC Selectivity Questions

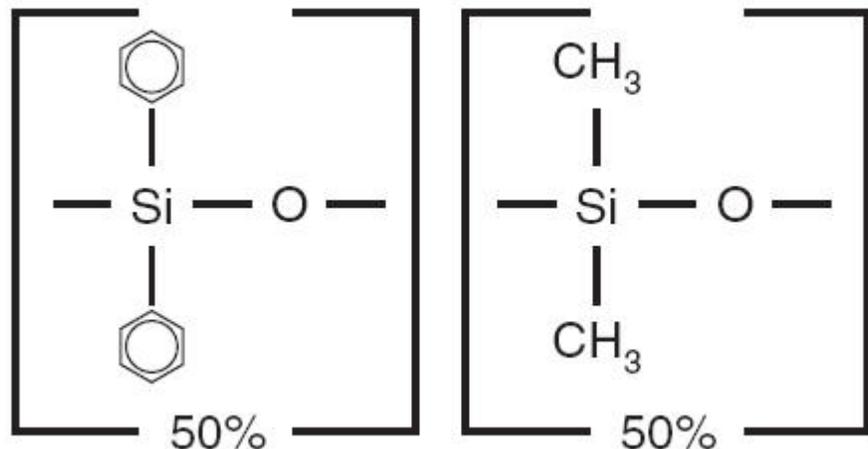
## Polycyclic Aromatic Hydrocarbons

- Need to separate benzo b, j, and k fluoranthenes?
  - Rxi-17Sil MS (50% diphenyl type)
- Need to separate chrysene and triphenylene?
  - Rxi-XLB (close elution though)
- Rt-PAH? (liquid crystal)
  - Separates chrysene, triphenylene
  - Benzo b and j fluoranthenes coelute
  - Narrow temperature range (80-285°C)

New!

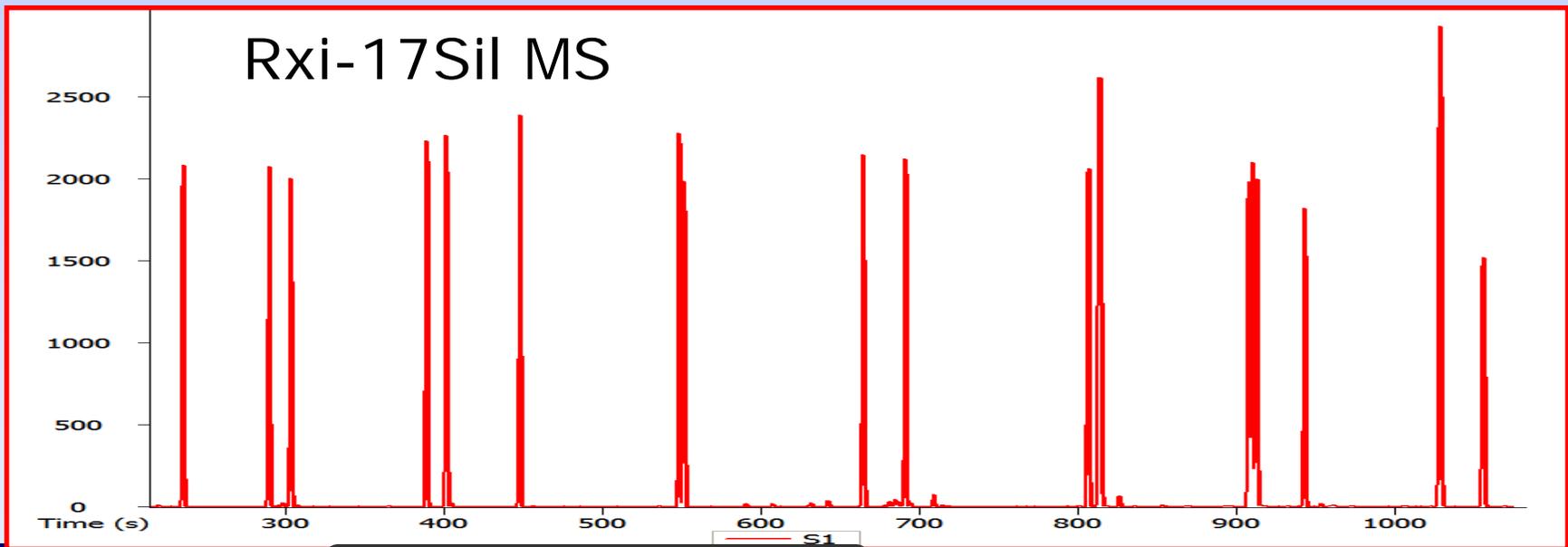
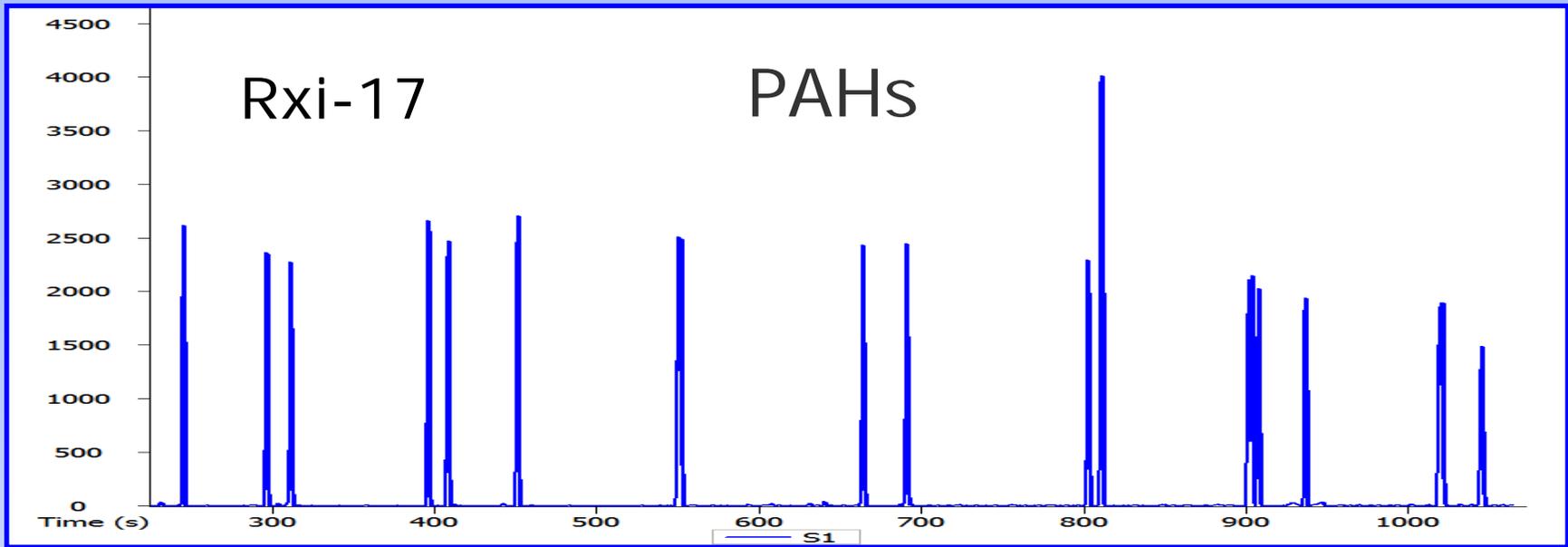


# Rxi-17



Structure  
not  
shown

**Rxi-17Sil MS**: an arylene-based polymer with 50% diphenyl selectivity



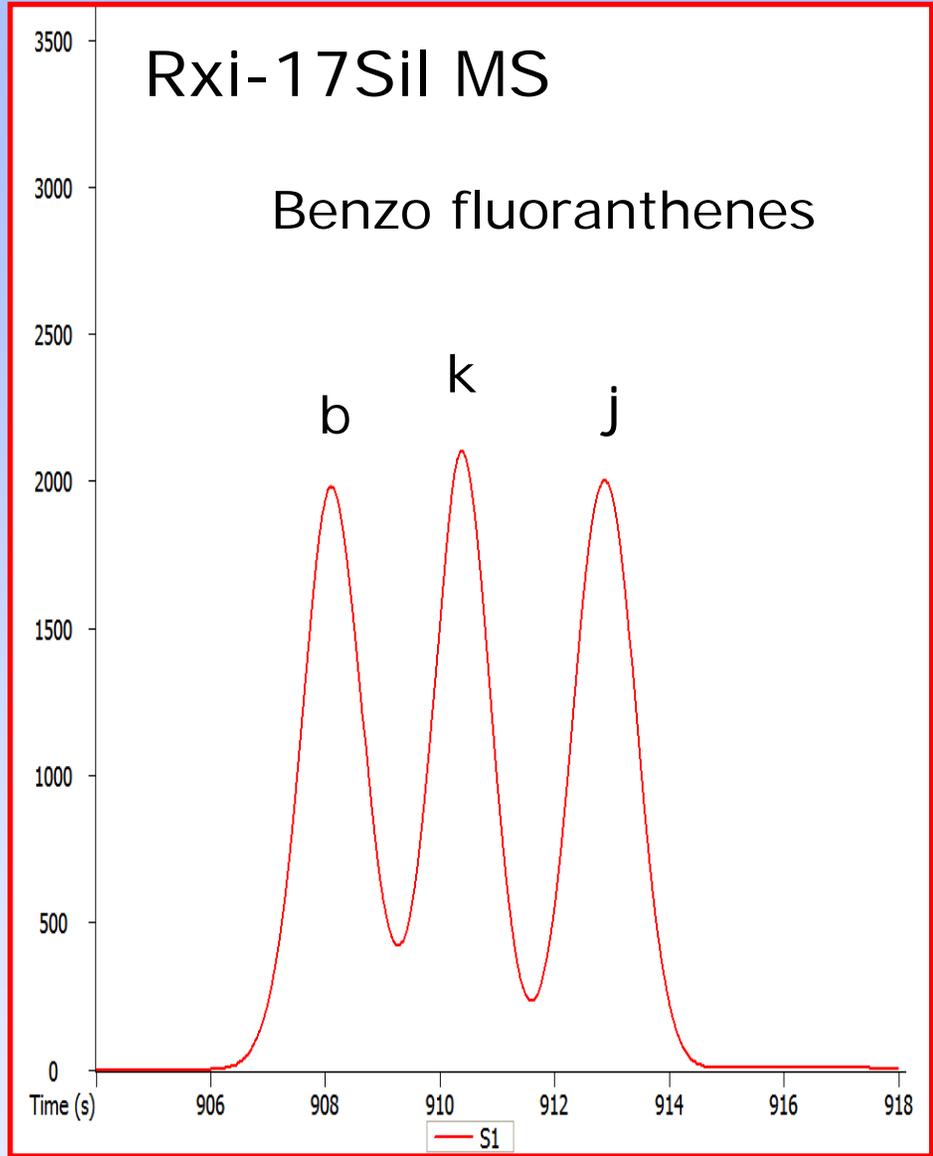
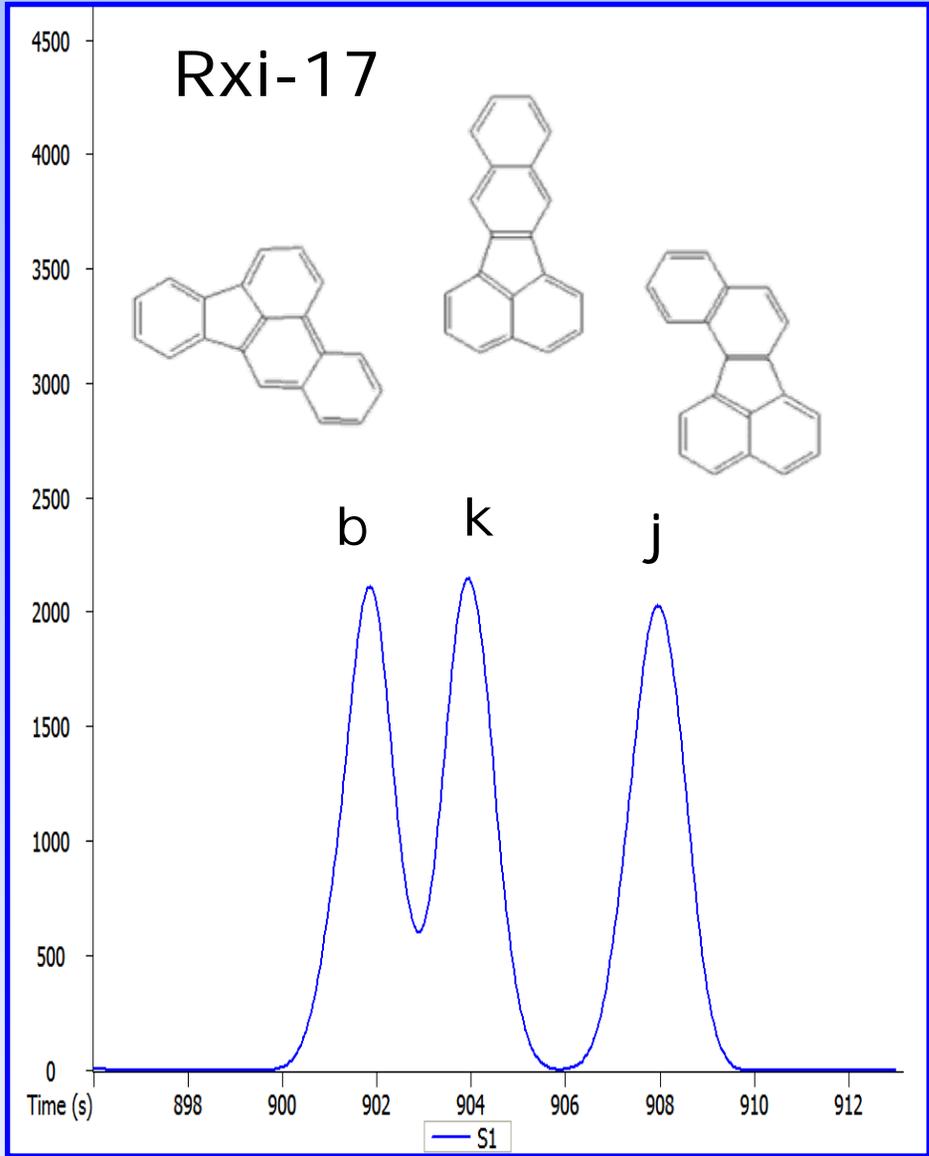
15m x 0.25mm x 0.25µm



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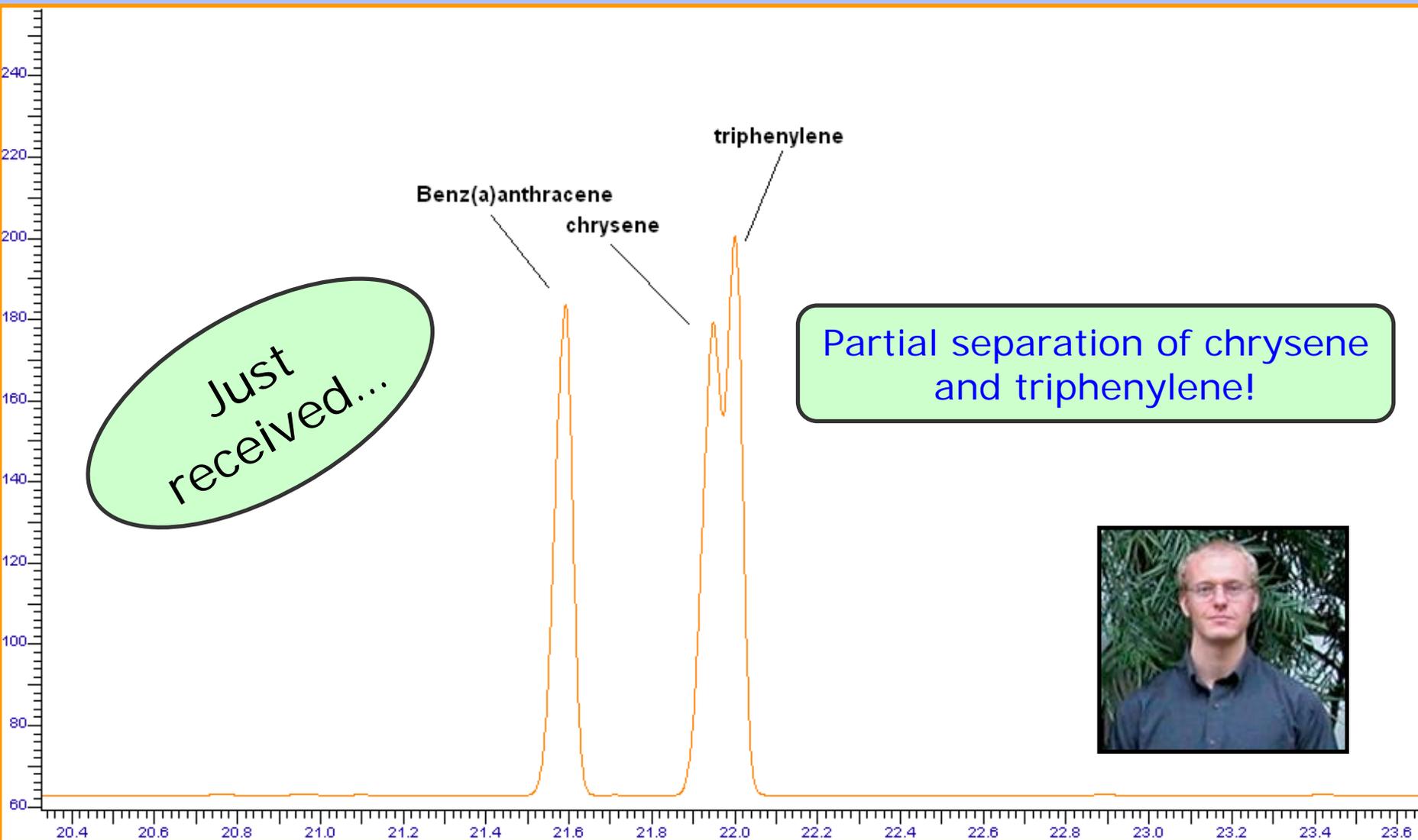
15m x 0.25mm x 0.25µm



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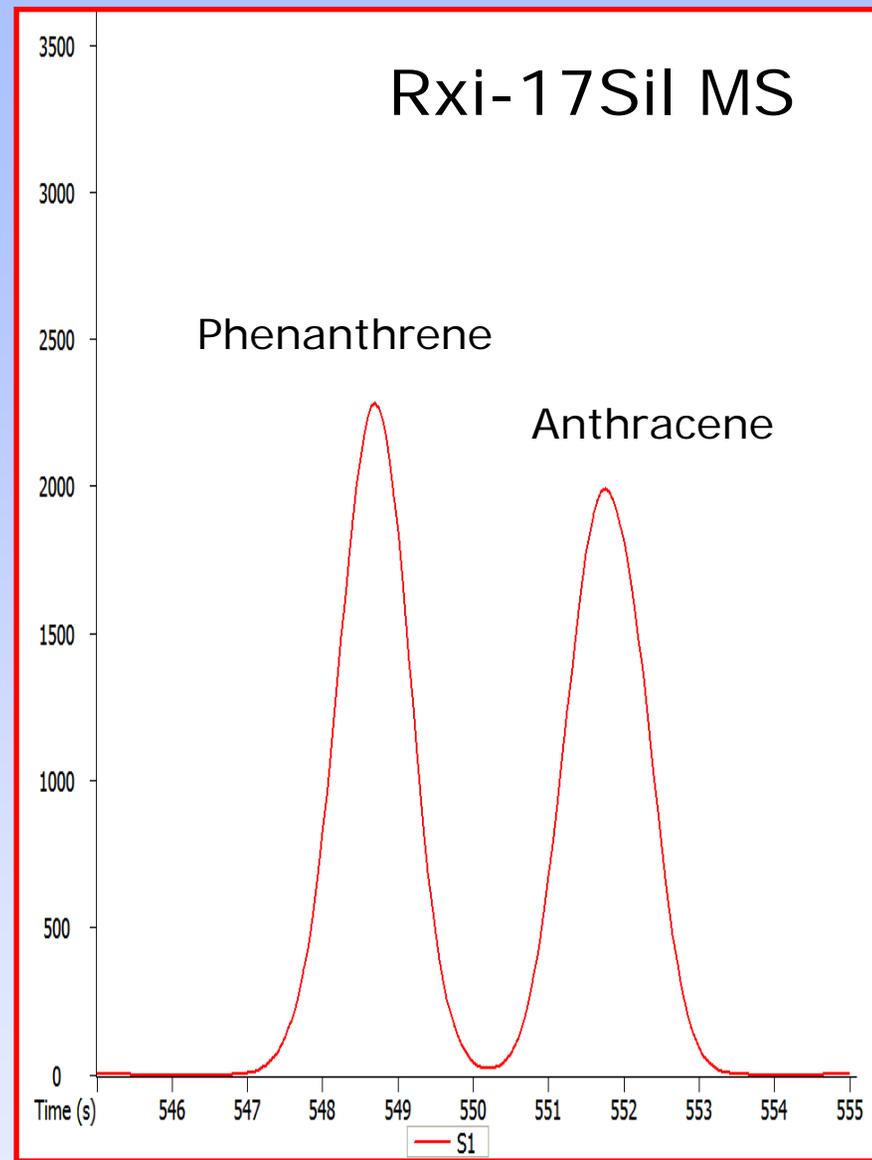
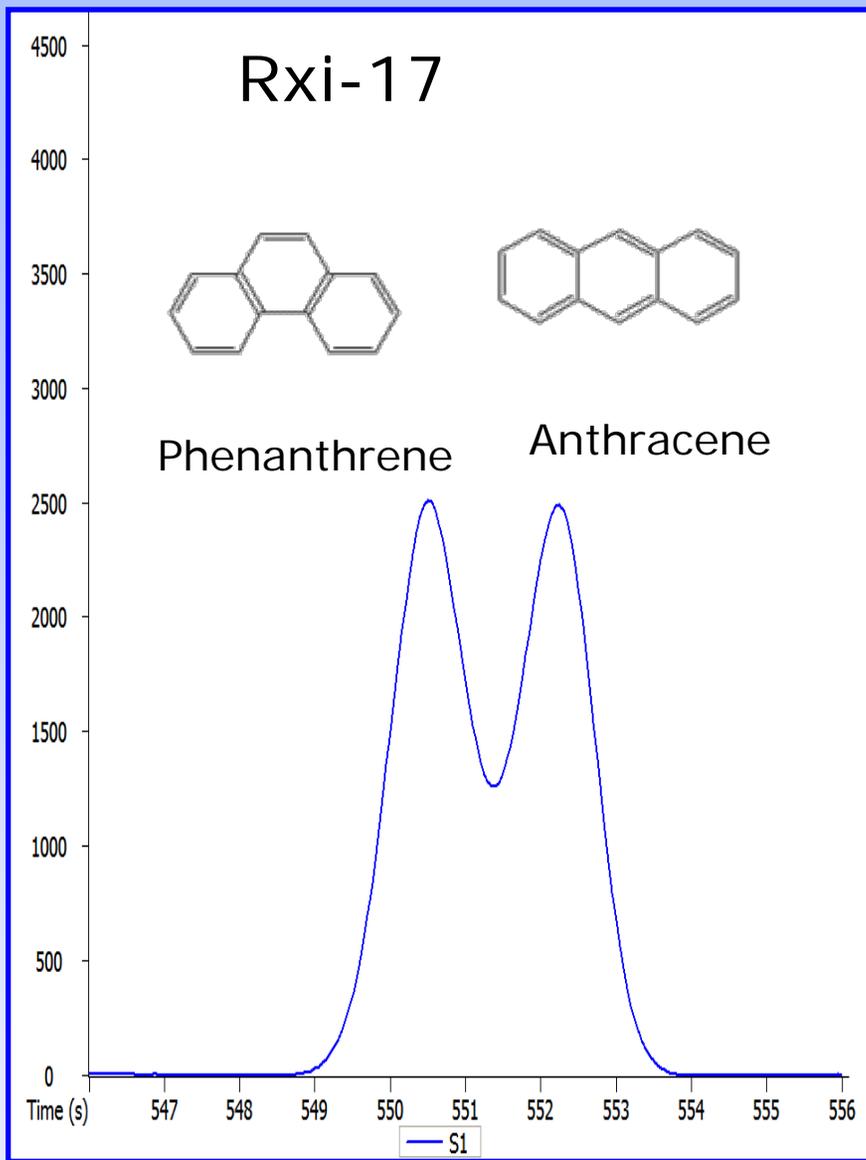
30m x 0.25mm x 0.25µm Rxi-17Sil MS



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# USPs for Restek Rxi-17Sil MS GC Column

- Rxi-17Sil MS is a low-bleed analog of Rxi-17
  - Maximum operating temperature of 360°C
  - Similar, but not exact, selectivity to Rxi-17
- Rxi-17Sil MS separates benzo b,k, and j fluoranthenes
- Rxi-17Sil MS partially separates chrysene and triphenylene
- Watch for Agilent DB-EUPAH and Varian Select PAH



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# HPLC of PAHs

Peak List:	Retention Time
1. naphthalene	6.19
2. acenaphthylene	7.16
3. 1-methylnaphthalene	7.78
4. 2-methylnaphthalene	8.17
5. acenaphthene	8.46
6. fluorene	8.82
7. phenanthrene	9.64
8. anthracene	10.39
9. fluoranthene	11.22
10. pyrene	11.75
11. benzo(a)anthracene	13.55
12. chrysene	13.91
13. benzo(j)fluoranthene	14.92
14. benzo(b)fluoranthene	15.27
15. benzo(k)fluoranthene	15.82
16. benzo(a)pyrene	16.35
17. dibenzo(a,h)anthracene	17.10
18. benzo(ghi)perylene	17.67
19. indeno(1,2,3-cd)pyrene	17.99

- Pinnacle II PAH
  - Restek exclusive!
  - Shape selective

**Sample:**  
Inj.: 10 $\mu$ L  
Conc.: 20 $\mu$ g/mL each component  
Sample diluent: acetonitrile

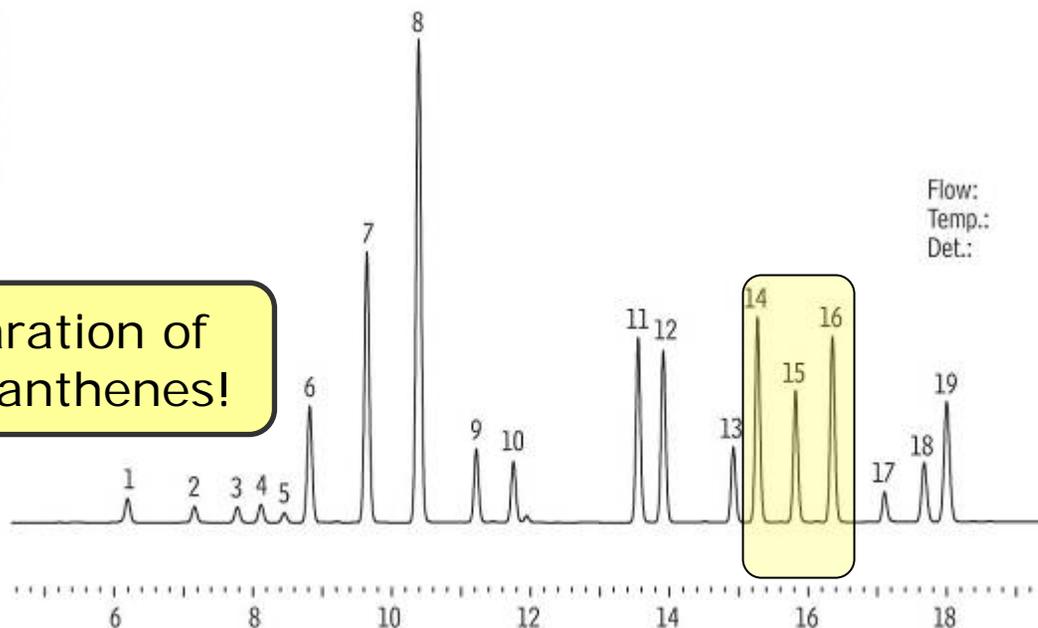
**Column:** Pinnacle® II PAH  
Cat.#: 9219463  
Dimensions: 150mm x 3.2mm  
Particle size: 4 $\mu$ m  
Pore size: 110Å

**Conditions:**  
Instrument: Shimadzu Prominence HPLC  
Mobile phase: A: purified water  
B: acetonitrile

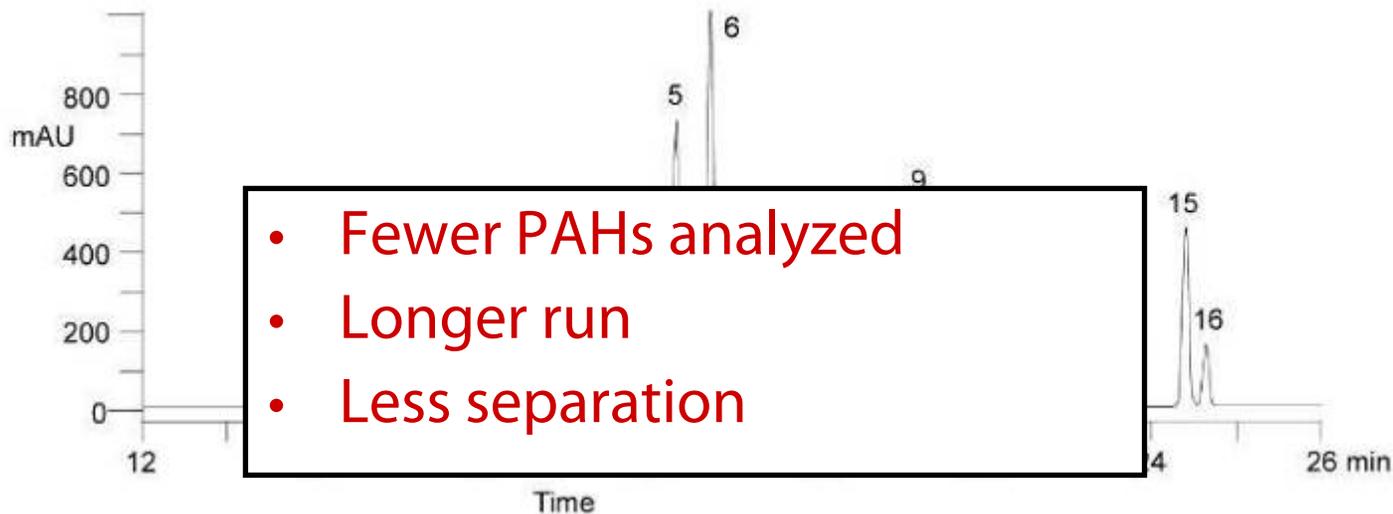
Time (min.)	%B
0	40
7	60
16	100
18.9	100
19	40

Flow: 1.2mL/min.  
Temp.: 30°C  
Det.: UV @ 254nm

Good separation of benzo fluoranthenes!



# Competitor HPLC of PAHs



## ANALYTES:

- 1 naphthalene
- 2 acenaphthylene
- 3 acenaphthene
- 4 fluorene
- 5 phenanthrene
- 6 anthracene
- 7 fluoranthene
- 8 pyrene
- 9 benzo (a) anthracene
- 10 chrysene
- 11 benzo (b) fluoranthene
- 12 benzo (k) fluoranthene

- 13 benzo (a) pyrene
- 14 dibenzo (a,h) anthracene
- 15 benzo (g,h,i) perylene
- 16 indeno (1,2,3-cd) pyrene

Phenomenex Synergi Max-RP  
4 $\mu$ m, 250x4.6mm, 80Å

# Restek UHPLC of PAHs – Very Fast Run

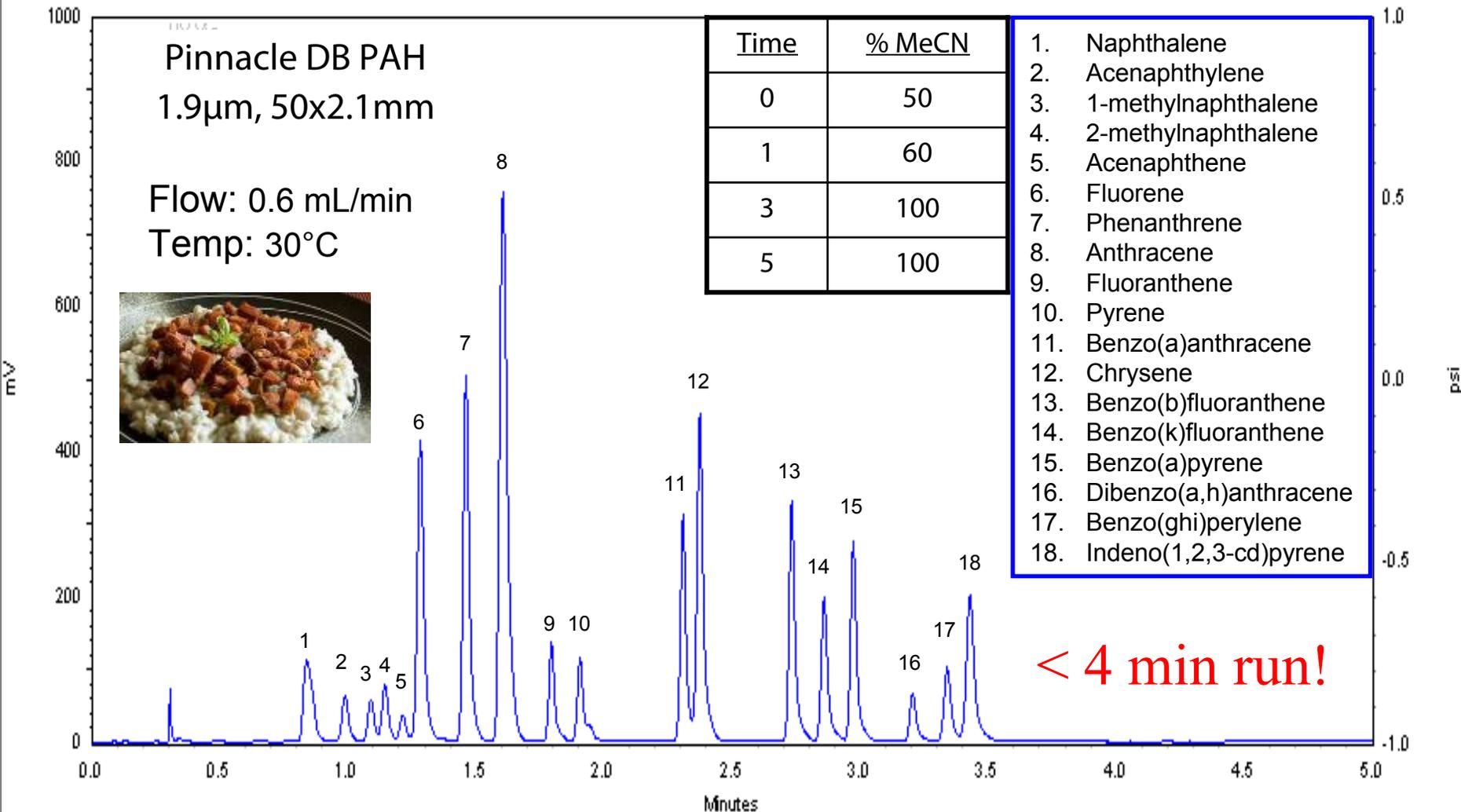
Pinnacle DB PAH  
1.9µm, 50x2.1mm

Flow: 0.6 mL/min  
Temp: 30°C



Time	% MeCN
0	50
1	60
3	100
5	100

1. Naphthalene
2. Acenaphthylene
3. 1-methylnaphthalene
4. 2-methylnaphthalene
5. Acenaphthene
6. Fluorene
7. Phenanthrene
8. Anthracene
9. Fluoranthene
10. Pyrene
11. Benzo(a)anthracene
12. Chrysene
13. Benzo(b)fluoranthene
14. Benzo(k)fluoranthene
15. Benzo(a)pyrene
16. Dibenzo(a,h)anthracene
17. Benzo(ghi)perylene
18. Indeno(1,2,3-cd)pyrene



# Summary of Food Safety

- Years of personnel experience at Restek
  - Best food safety technical support
- Wide range of QuEChERS products
  - Rapidly growing food safety sample preparation method
- GC and HPLC columns for food safety
  - Inert, selective, tested, adopted
- Total solution offerings
  - Sample preparation, standards, GC/LC columns/consumables
- Innovative new products
  - QuEChERS, Rxi-17Sil MS, 5mm GC inlet liner
- Technical information support
  - Web, literature, presentations, ChromaBLOGraphy, etc.



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# Appendix

- Extra slides for distributors to review
- Additional topics and/or applications



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# Other Topics for Food Safety

- PBDEs in food
  - Rtx-1614 column (Restek exclusive!)
    - Fast runs and minimal BDE 209 breakdown
    - [www.restek.com/aoi\\_env\\_A035.asp](http://www.restek.com/aoi_env_A035.asp)
- Melamine in infant formula
  - Rxi-5Sil MS, standards, derivatization reagents
    - [www.restek.com/restek/images/external/FFAN1137.pdf](http://www.restek.com/restek/images/external/FFAN1137.pdf)
- Veterinary drug residues
  - Biphenyl HPLC columns (Restek exclusive!)
    - Julie Kowalski (Florida Pesticide Residue Workshop)



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# Other Topics for Food Safety

- Dietary supplements
  - Restek publishes first QuEChERS method
    - [www.restek.com/restek/images/external/PHAN1242.pdf](http://www.restek.com/restek/images/external/PHAN1242.pdf)
- Food authentication and origin
  - Not really food safety, but hot food topic
    - Lots of GCxGC work being done
- GCxGC for food safety and food applications
  - Restek has two GCxGC systems
    - LECO Pegasus 4D GCxGC-TOFMS, GCxGC-ECD/FID
  - Full line of 0.10, 0.15, 0.18mm columns for GCxGC



Jaap de Zeeuw  
International  
GC Specialist

#### Fast GC Using 0.10mm and 0.18mm ID Capillary Columns and Comprehensive GC

- Significantly reduces analysis time without sacrificing resolution.
- Highest column efficiencies, great for GC/MS.
- Excellent for comprehensive GC (GCxGC) as second dimension column.

Narrow bore (0.18mm ID) columns are attractive alternatives to conventional-diameter capillary columns because they provide faster analysis times and higher resolving power. As column ID decreases, column efficiency (plates/meter) greatly increases. For instance, a 0.18mm ID column (5,150 plates/meter) is 52% more efficient than a 0.25mm ID column (3,900 plates/meter). Therefore, resolution can be achieved with a shorter column, which decreases the analysis time. When switching from a 0.25mm ID column to a 0.10mm ID column (8,500 plates/meter) the column efficiency is improved dramatically (118%).

Typically, 0.18mm ID columns are used for fast GC analysis, and methods are easily converted. The 0.10mm ID columns require more research to switch methods to the smaller ID due to higher back pressures and lower column capacity.

The outer diameter of the 0.10mm and 0.18mm ID tubing is the same as 0.25mm ID tubing, which makes connections less complicated.

#### Rxi®-1ms Columns (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	-90 to 330/350°C	13101	
0.18mm	0.18	-90 to 330/350°C		13302

#### Rxi®-5ms Columns (fused silica)

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

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	-90 to 330/350°C	13401	
0.18mm	0.18	-90 to 330/350°C		13402
0.20	0.20	-90 to 330/350°C		13403
0.26	0.26	-90 to 330/350°C		13411

#### Rxi®-5Sil MS Columns (fused silica)

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

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	-90 to 330/350°C	43601	
0.18mm	0.18	-90 to 330/350°C		43602
0.26	0.26	-90 to 330/350°C		43604

#### Rxi®-17 Columns (fused silica)

(Crossbond® 50% diphenyl/50% dimethyl polysiloxane)

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	40 to 280/320°C	13510	

#### Rtx®-Wax Columns (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	20 to 250°C	41001	41002
0.20	0.20	20 to 240/250°C	41003	41004

#### Rt™-LC50 Columns (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	100°C to 270°C	16736	
0.18mm	0.18	100°C to 270°C		16735

#### Rtx®-CLPesticides (fused silica)

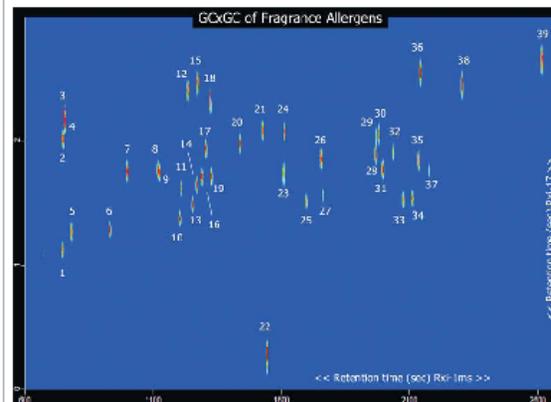
ID	df (µm)	temp. limits	10-Meter	20-Meter
0.18mm	0.18	-60 to 310/330°C	42101	
0.18mm	0.18	-60 to 310/330°C		42102

#### Rtx®-CLPesticides2 (fused silica)

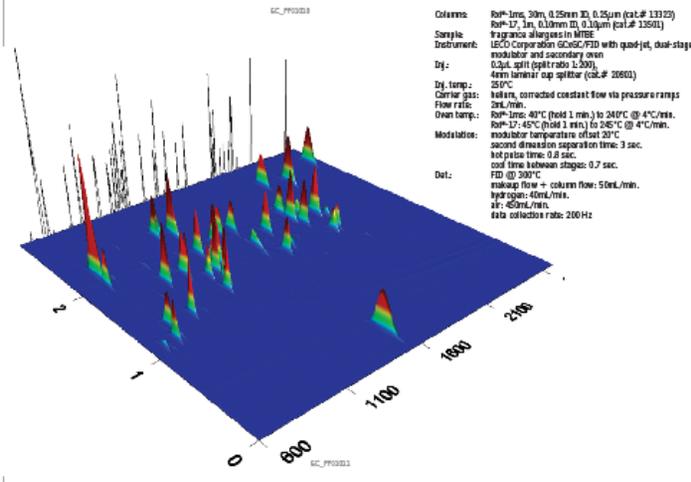
ID	df (µm)	temp. limits	10-Meter	20-Meter
0.10mm	0.10	-60 to 310/330°C	42301	42302
0.18mm	0.18	-60 to 310/330°C	42301	42302

RESTEK www.restek.com

#### Fragrance Allergens Rxi®-1ms & Rxi®-17 (GC x GC)



1. limonene
2. 1-fluoronylhexane
3. benzyl alcohol
4. phenyl acetate/diethyl
5. eucalyptol
6. linalool
7. camphor
8. methyl-2-octylate
9. eugenol
10. citral/indol
11. citral 1
12. trans-dimethylsilylolefin
13. geranyl
14. citral 2
15. anise alcohol
16. hydroxyethyl cinnol
17. styrene
18. cinnamyl alcohol
19. methyl-2-nonylate
20. eugenol
21. methyl eugenol
22. coumarin
23. hydroxyethyl cinnol  
contaminant
24. isoeugenol
25. α-terpinenyl lincane 1
26. linal
27. α-terpinenyl lincane 2
28. anisyl cinnamal
29. linal 1
30. linal 2
31. dimethylsilylolefin 1
32. dimethylsilylolefin 2
33. farnesol 1
34. farnesol 2
35. heptyl cinnamal 1
36. benzyl benzoate
37. heptyl cinnamal 2
38. benzyl salicylate
39. benzyl cinnamate



Columns: Rxi®-1ms, 30m, 0.15mm ID, 0.25µm (cat.# 13323)  
Rxi®-17, 1m, 0.10mm ID, 0.10µm (cat.# 13501)

Sample: Fragrance allergens in MIBK

Instrument: LECO Corporation GCxGC-PTD with quad-jet, dual-stage modulator and secondary oven

Dil: 0.2µl split (split ratio 1:200)

Dil: 4mm laminar cap splitter (cat.# 20003)

Dry temp.: 25°C

Carrier gas: helium, constant flow via pressure ramps

Flow rate: 2ml/min

Oven temp.: Rxi®-1ms: 40°C (hold 1 min) to 240°C @ 4°C/min.  
Rxi®-17: 40°C (hold 1 min) to 240°C @ 4°C/min.  
modulator temperature offset 20°C

Modulation: second dimension separation time: 3 sec.  
hold pulse time: 0.8 sec.  
cool time between stages: 0.7 sec.

Det.: FID @ 300°C  
makeup flow + column flow: 50ml/min.  
hydrogen: 40ml/min.  
air: 450ml/min.  
data collection rate: 200 Hz

www.restek.com RESTEK

# Columns for Comprehensive GC (GCxGC)

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# GCxGC Selectivity Kit A

The selectivity kit contains four columns of different selectivity for method development. Includes one each of the following:

- Rxi®-17, 1.1m (±3cm), 0.10mm ID, 0.10µm, 50% diphenyl dimethylpolysiloxane
- Rtx®-CLPesticides, 1.1m (±3cm), 0.10mm ID, 0.10µm, trifluoropropyl containing polymer
- Stabilwax®, 1.1m (±3cm), 0.10mm ID, 0.10µm, polar polyethylene glycol
- Rt®-LC350, 1.1m (±3cm), 0.15mm ID, 0.10µm, liquid crystalline phase selective for aromatic compounds

Description	Length	qty.	Cat.#	Price	Wish list	Cart
GCxGC Selectivity Kit A		kit	15105		<input type="checkbox"/>	
<b>0.15mm ID</b>						
<b>0.10µm df</b>						
Rt-LC350	1.1m (±3cm)	ea.	15101		<input type="checkbox"/>	
<b>0.10mm ID</b>						
<b>0.10µm df</b>						
Stabilwax	1.1m (±3cm)	ea.	15102		<input type="checkbox"/>	
Rtx-CLPesticides	1.1m (±3cm)	ea.	15103		<input type="checkbox"/>	
Rxi-17	1.1m (±3cm)	ea.	15104		<input type="checkbox"/>	



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# Environmental Program



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# Outline

- Semivolatile organic compounds
- Volatile organic compounds
- Pesticide
- Air sampling

# Semivolatile Overview

- Method Summary:
  - Semi-volatile compounds are extracted from sample matrix using solvent (methylene chloride). The extract is concentrated and an aliquot is typically analyzed by GC/MS.
- Compound lists include US EPA Methods 8270, 625, & 525
- Products offered:
  - GC Columns (Rxi-5Sil MS)
  - Reference Standards
  - Inlet Supplies (liners, inlet seals, septa)
  - Sample prep products (extraction disks, ASE Parts)
  - Autosampler Vials & Syringes

# What Compounds are Considered Semi-Volatiles?

- Solvent extractable (over 100 compounds)
- Wide variety of compound classes
- pH adjustment for base-neutral & acidic compounds

pH = 2

pH = 12

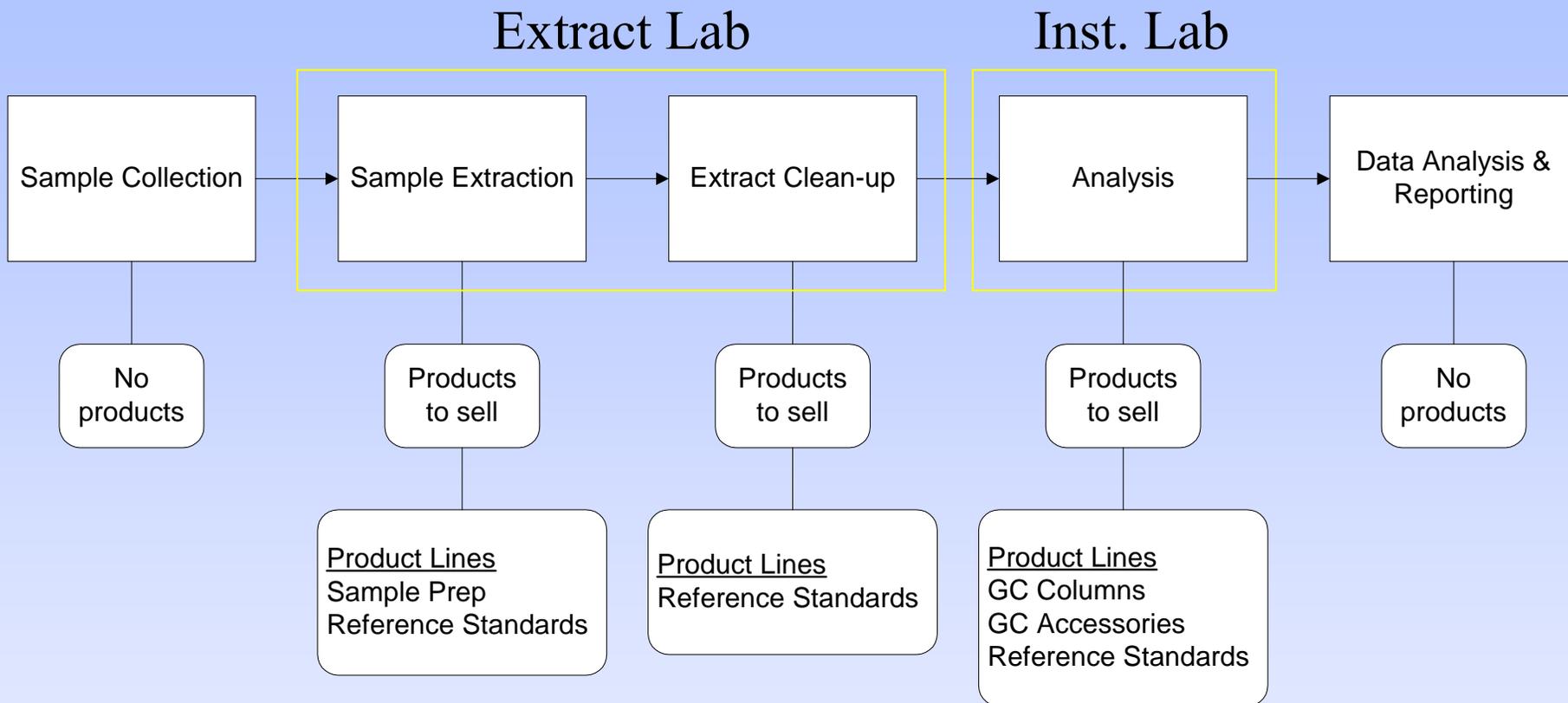


Phenols

PAHs, phthalates, pesticides,  
chlorinated hydrocarbons

Nitrosamines,  
anilines

# Sample Process for Semi-Volatiles



# Sales Strategy

- Lead with:
  - Rxi-5Sil MS columns (best in-class for inertness, bleed, resolution of benzo(b&k)fluoranthene, and reproducibility)
  - Calibration mixes
    - Mega Mixes (75 compounds in one ampoule)
    - Surrogate & matrix spikes
    - Custom standards
  - Sky Inlet Liners
    - Single gooseneck w/ wool
  - Flip Inlet Seals (more reliable seal to eliminate injection port leaks and double the use)
  - Leak detector
- Follow with:
  - Other instrument supplies
  - Vials & Syringes

# Semivolatiles Analytical Solution

- GC Column
  - 30m x 0.25mm x 0.25µm Rxi-5Sil MS
    - Excellent choice for capacity and ruggedness
    - Flow matches well with MS pumping capacity
    - 5m or 10m Integra Guard columns available
- Injection port set-up
  - New Sky Inlet Liners
    - 4mm ID single gooseneck liner w/ wool
  - New optimized deactivation results in more inert liner

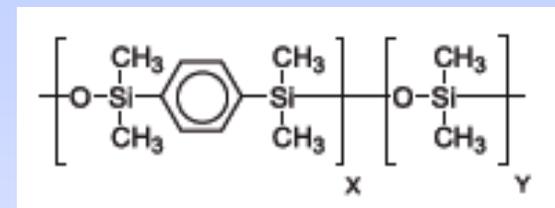


# Rxi-5SiI MS

## GC Column Technology



- Highest inertness
- Lowest bleed
- Exceptional reproducibility

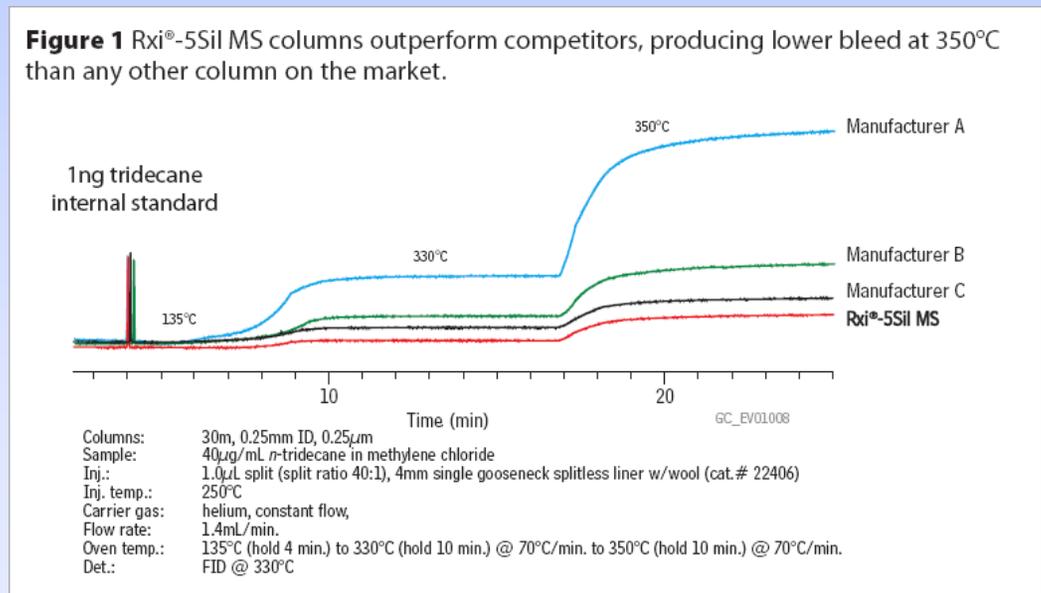


Rxi-5SiI MS

- Combination of features are what is important for environmental analysis of semivolatile organic compounds

# Rxi-5Sil MS Column

- Low-bleed
  - Very important for MS, especially ion trap
  - But most vendors have low-bleed columns
    - Rxi is even better
      - DB-5ms, ZB-5ms, VF-5ms



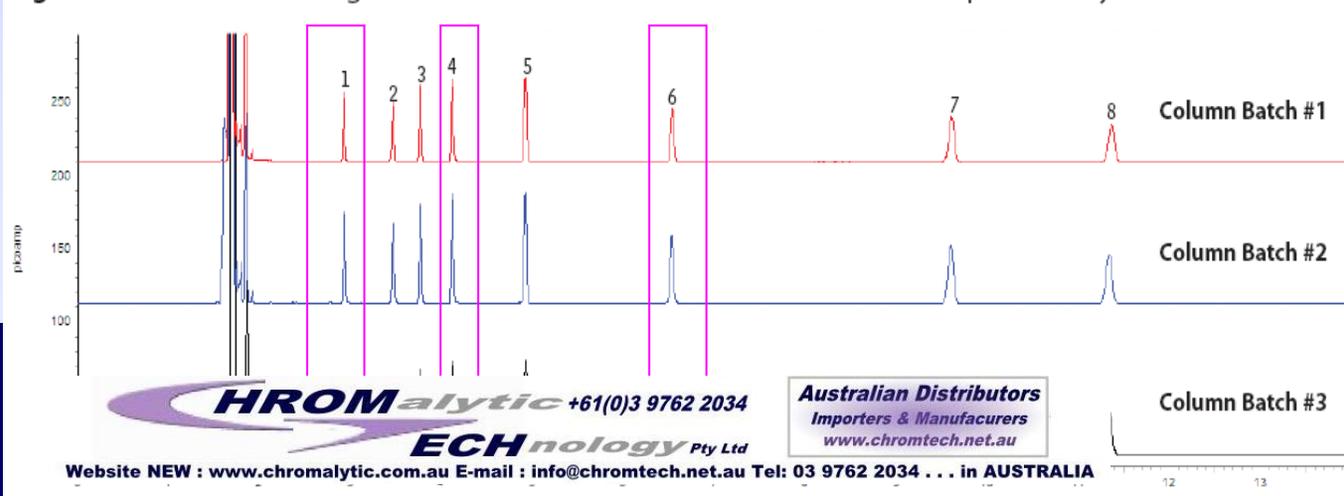
# Rxi-5Siil MS Column

## Reproducibility

- Customers want the same chromatography every time they install new column
- Critical parameters of column:
  - Bleed: **Signal to noise**
  - Film thickness: **Absolute retention time**
  - Plates/meter: **Peak sharpness**
  - Selectivity: **Peak relative retention times**
  - Inertness: **Peak response of active compounds**

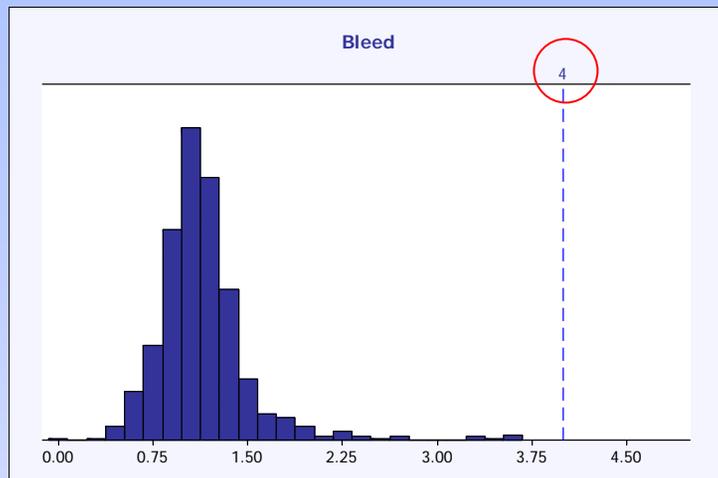
1. 1,6-hexanediol
2. 4-chlorophenol
3. methyl nonanoate
4. 1-decylamine
5. tridecane
6. 1-undecanol
7. acenaphthylene
8. pentadecane

Figure 6 Rxi® columns are engineered to assure column-to-column and lot-to-lot reproducibility.



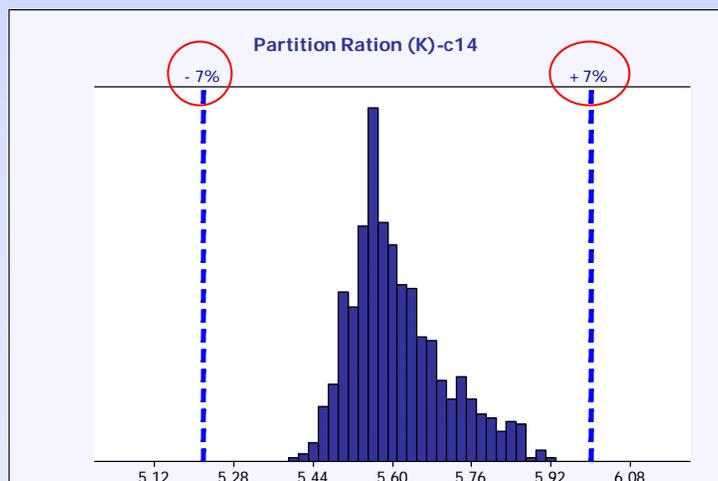
# Rxi-5Sil MS Column Reproducibility

(Rxi-5Sil MS, 30m x 0.25mm, 0.25um)



Bleed  
Mean: 1.1  
Std dev: 0.38  
Ind. Std: **4pA**

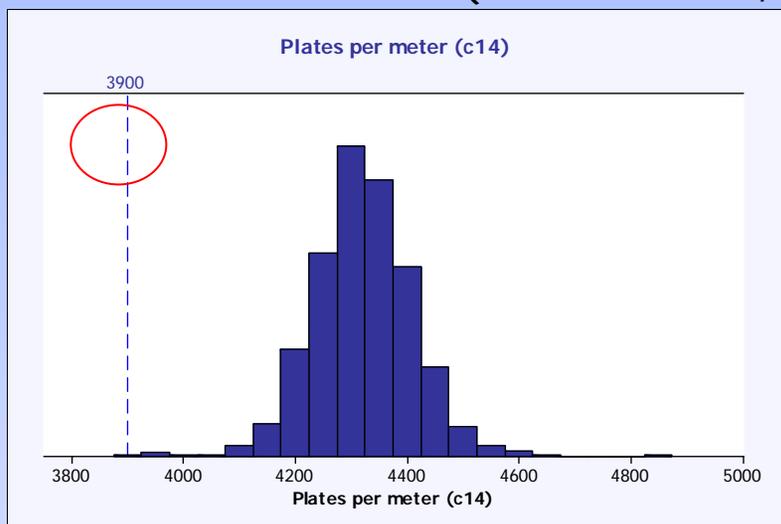
All columns manufactured  
in a years time



Partition Coefficient (k)  
Mean: 5.6  
Std dev: 0.09  
Ind. Std: **+/- 7%**

# Rxi-5Sil MS Column Reproducibility

(Rxi-5Sil MS, 30m x 0.25mm, 0.25um)



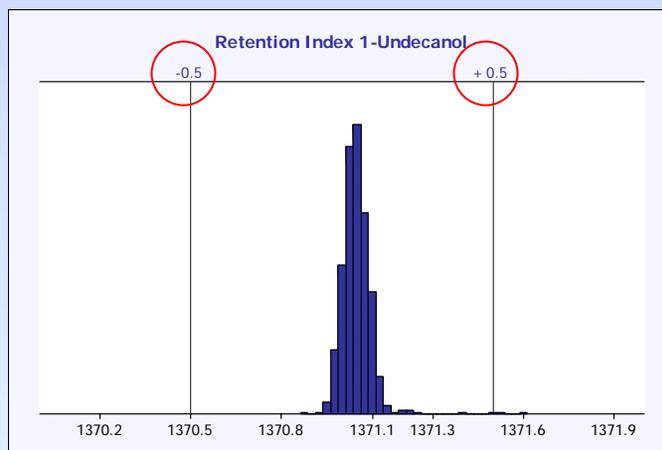
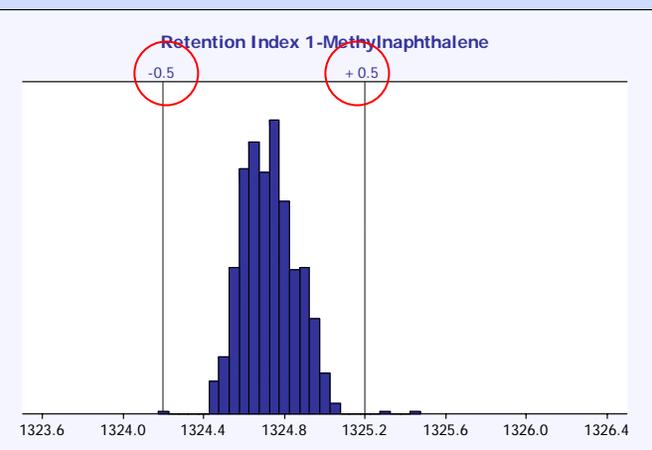
Plates/meter

Mean: 4321

Std dev: 91

Ind. Std: 3900

All columns manufactured  
in a years time



Retention Indice (RI)

Methylnaphthalene

Mean: 1324.7

Std dev: 0.13

1-Undecanol

Mean: 1371.0

Std dev: 0.05

Ind. Std: +/-5%

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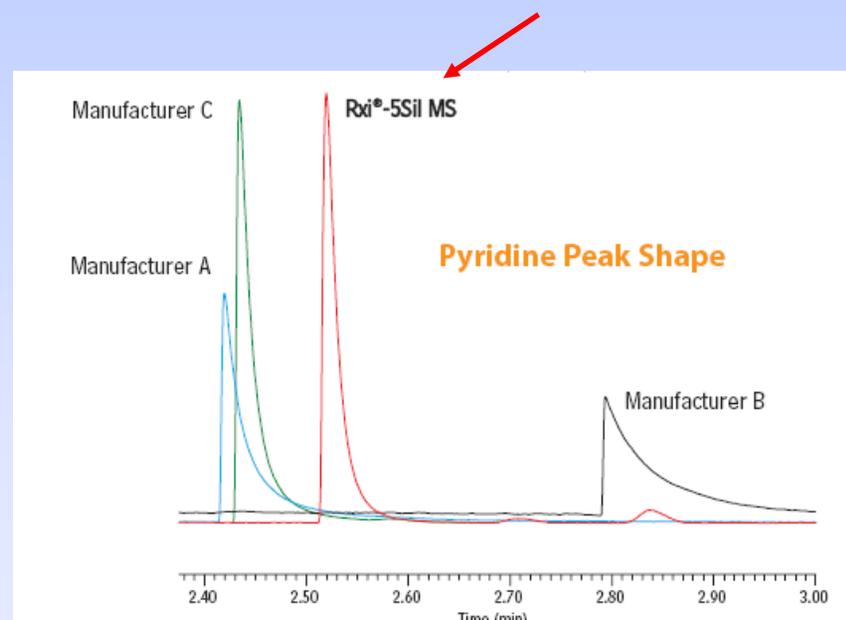
# Rxi-5Sil MS Column

- Inertness!
  - An IMPORTANT selling feature for the Rxi-5Sil MS and semivolatile analyses
- Semivolatiles includes a wide range of acids, bases, neutrals, polar compounds
  - Pyridine; 2,4-dinitrophenol; etc.

**Table I** Rxi®-5Sil MS columns produce high response factors for both basic and acidic compounds.

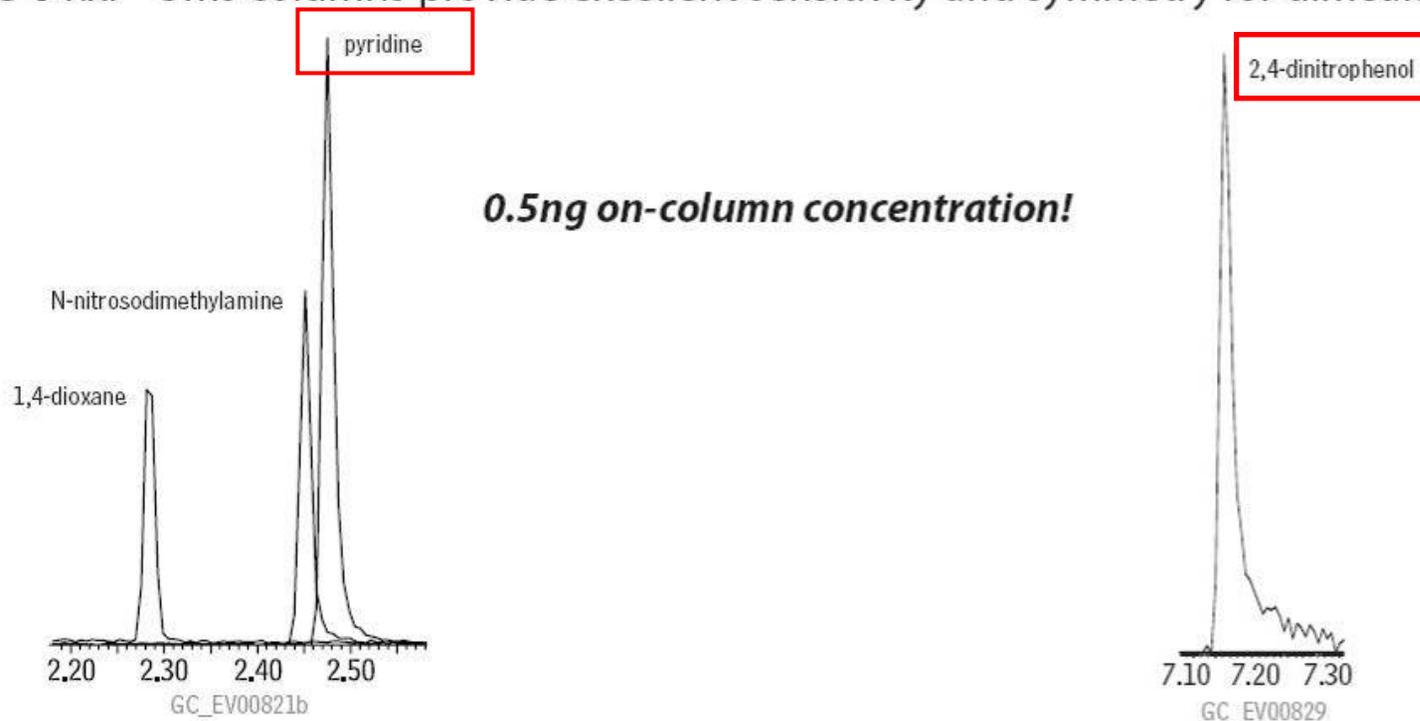
	2,4-dinitrophenol (average RF)	pyridine (average RF)
Rxi®-5Sil MS	0.24	0.74
Manufacturer A	0.20	0.63
Manufacturer B	0.22	0.64
Manufacturer C	0.24	0.65

Response factors are based on phenanthrene. (n=7)



# High Inertness of Rxi for Semivolatile Base and Acid at Low Level

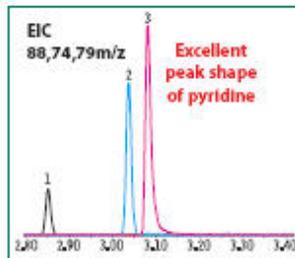
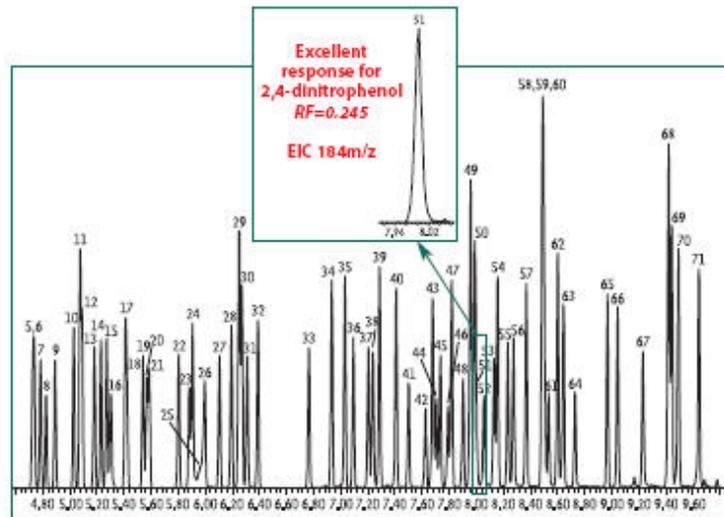
**Figure 1** Rxi™-5ms columns provide excellent sensitivity and symmetry for difficult compounds.



# Semivolatile Analysis Rxi-5Si1 MS 30m x 0.25mm, 0.25um

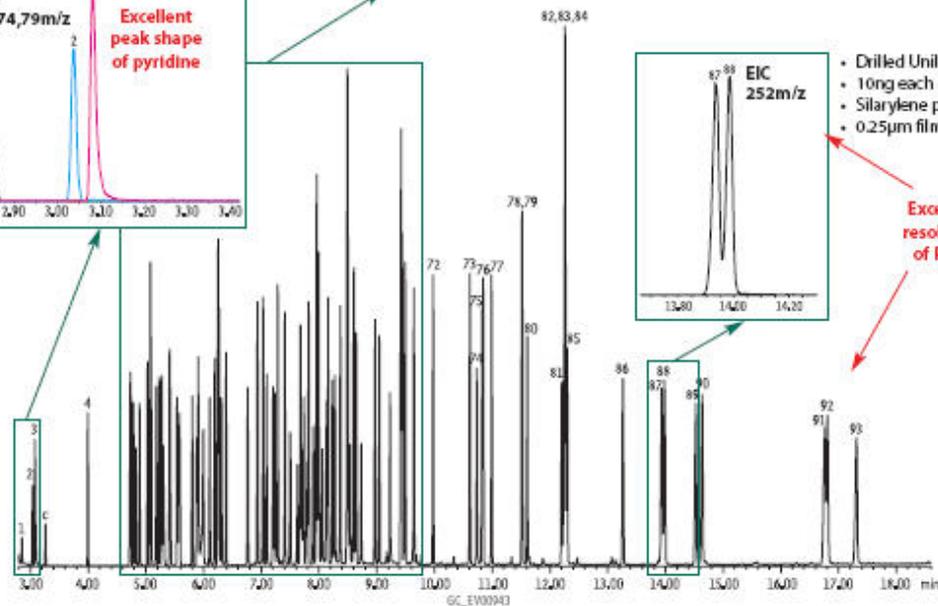
## Semivolatile Organics US EPA Method 8270 Rxi®-5Si1 MS

Column: Rxi®-5Si1 MS, 30m, 0.25mm ID,  
0.25um (cat.# 13623)  
Sample: US EPA Method 8270 Mix, 1µL of  
10µg/mL (IS 40µg/mL)  
8270 MegaMix® (cat.# 31850)  
Benzoic Acid (cat.# 31879)  
8270 Benzodioxin Mix (cat.# 31852)  
Acid Surrogate Mix (4/89 SOW) (cat.# 31025)  
Revised B7N Surrogate Mix (cat.# 31857)  
1,4-Dioxane (cat.# 31853)  
SV Intamat Standard Mix (cat.# 31206)  
1µL (10ng on-column concentration),  
4mm Drilled Unliner® (hole near bottom)  
inlet liner (cat.# 20756),  
pulsed splitless: pulse 25psi @ 0.2 min.,  
60mL/min. @ 0.15 min.  
Inj. temp.: 250°C  
Carrier gas: helium, constant flow  
Flow rate: 1.2mL/min.  
Oven temp.: 40°C (hold 1.0 min.) to 280°C @ 25°C/min.  
to 320°C @ 5°C/min. (hold 1 min.)  
Det.: MS  
Transfer line temp.: 280°C  
Scan range: 35-550amu  
Ionization: EI  
Mode: scan



- Drilled Unliner® liner,
- 10ng each compound
- Silarylene phase
- 0.25µm film thickness

Excellent resolution of PAHs



10ng on-column

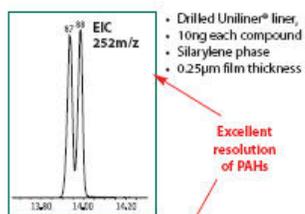
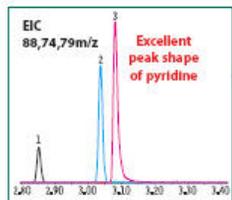
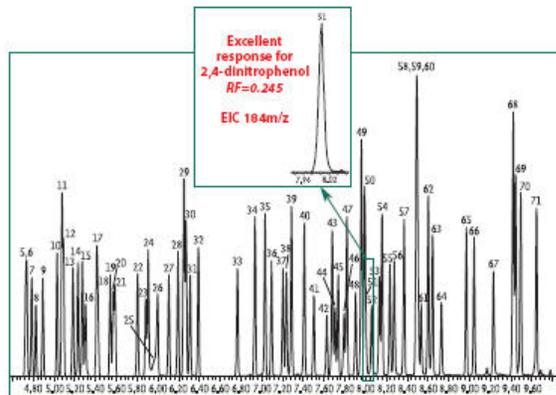
# Rxi-5Sil MS

30m x 0.25mm, 0.25um

10ng on-column

## Semivolatile Organics US EPA Method 8270 Rxi®-5Sil MS

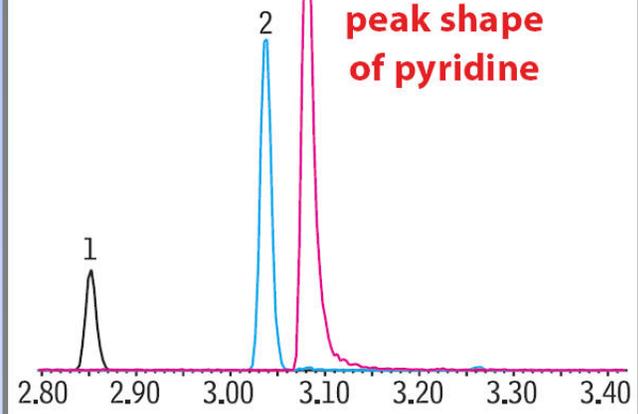
Column: Rxi®-5Sil MS, 30m, 0.25mm ID,  
0.25um (cat.# 13823)  
Sample: US EPA Method 8270 Mix, 1µL of  
1.0µg/mL (15 40µg/mL)  
8270 MegaMix® (cat.# 31850)  
Benzoic Acid (cat.# 31870)  
8270 Benzidines Mix (cat.# 31852)  
Acid Surrogate Mix (4/89 SOW) (cat.# 31025)  
Revised B/N Surrogate Mix (cat.# 31887)  
1,4-Dioxane (cat.# 31893)  
SV Internal Standard Mix (cat.# 31206)  
Inj: 1.0µL (10ng on-column concentration),  
4mm Drilled Uniliner® (hole near bottom)  
inlet liner (cat.# 20750),  
pulsed splitless: pulse 25psi @ 0.2 min.,  
60mL/min. @ 0.15 min.  
Inj. temp.: 250°C  
Carrier gas: helium, constant flow  
Flow rate: 1.0mL/min.  
Oven temp.: 40°C (hold 1.0 min.) to 280°C @ 25°C/min.  
to 320°C @ 5°C/min. (hold 1 min.)  
Det.: MS  
Transfer line temp.: 280°C  
Scan range: 35-550amu  
Ionization: EI  
Mode: scan



- Drilled Uniliner® liner,
- 10ng each compound
- Silarylene phase
- 0.25µm film thickness

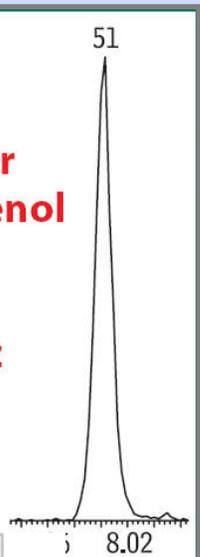
EIC

88,74,79m/z



Excellent response for 2,4-dinitrophenol  
RF=0.245

EIC 184m/z



High inertness on Rxi-5Sil MS

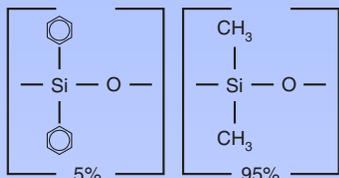


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# Rxi-5ms and Rxi-5Sil MS columns are not the same

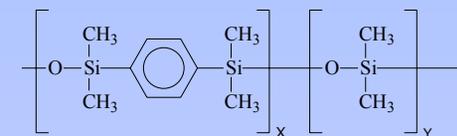


## Rxi-5ms

(5% Diphenyl-PDMS)

## Rxi-5Sil MS

(Silarylene-stabilized equivalent of  
5% diphenyl-PDMS)



**Restek**

Rtx-5, Rtx-5ms

**Rxi-5Sil MS**

**Agilent**

HP-5, HP-5ms, DB-5,  
Ultra-2

DB-5ms

**Varian**

CP-Sil 8 CB

VF-5ms, CP-Sil 8 CB LB/MS

**Alltech**

AT-5

AT-5ms

**Supelco**

Equity-5

MDN-12, SLB-5ms

**SGE**

BP-5

BPX-5

**Phenomenex**

ZB-5

ZB-5ms

**Machery-Nagel**

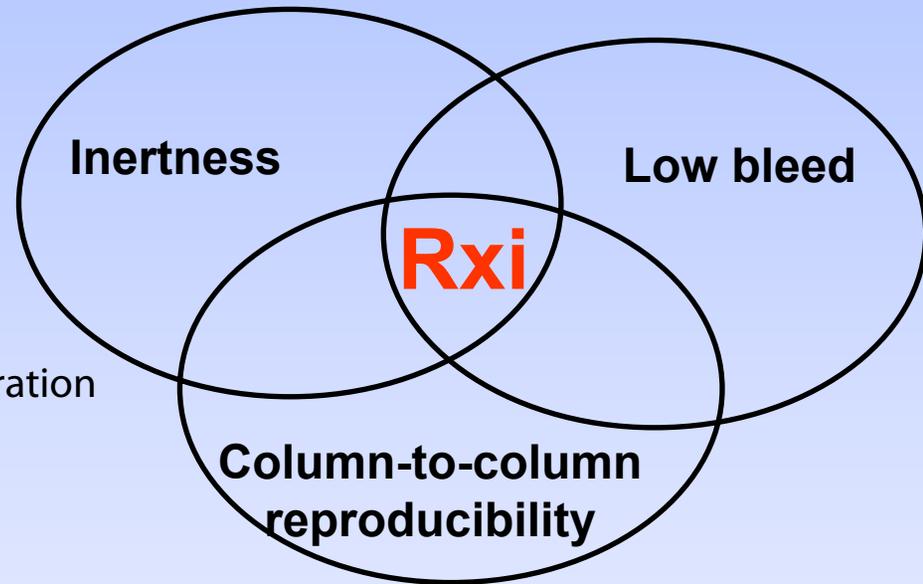
Optima-5

Optima-5ms

# Value to Customer



- The combination of column inertness, low bleed and column-to-column reproducibility is what makes the Rxi-5Sil MS valuable to analytical chemists
- Ultra inert column
  - Reduced tailing of active compounds
    - Higher response factors
    - More accurate data from better integration
- Low bleed
  - Higher signal to noise
  - Lower detection limits
- Column-to-column reproducibility
  - Reduced down time from column exchange



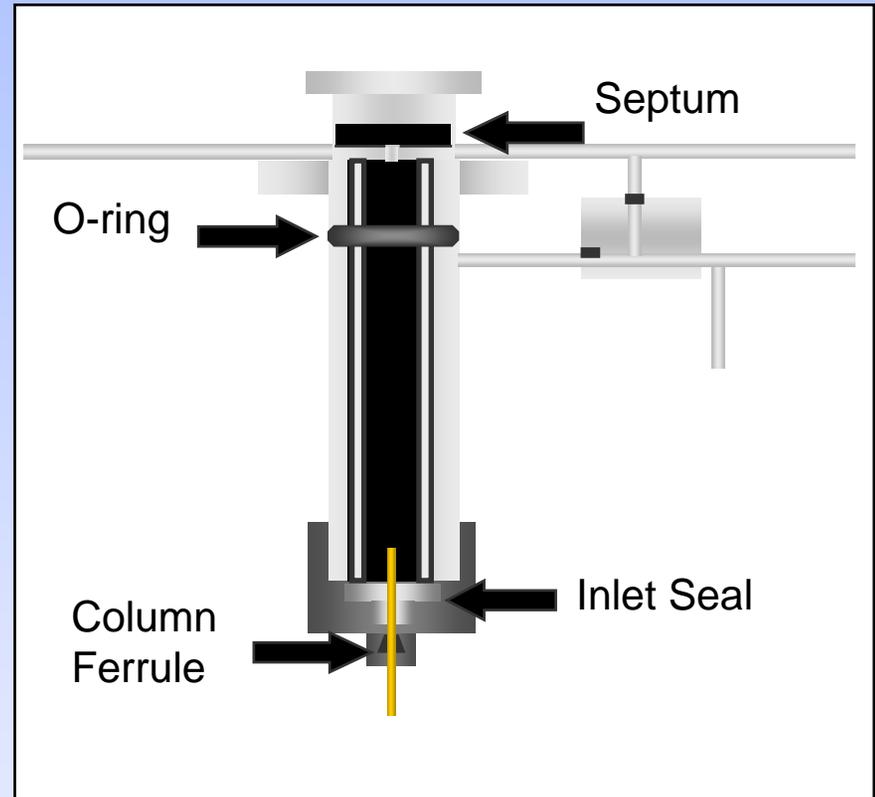
# Semivolatile Standards

- Analytical reference materials
  - Up to 76 compound MegaMix®
    - Additional compounds available
  - Matrix spike solutions
  - Internal standards
  - Surrogate standards



# Injection Ports

- Regular maintenance required
  - Septum
  - O-ring
  - Liner
  - Inlet seal
  - Column ferrule
- Why?
  - Build up of nonvolatile material in liner and beginning of column causes tailing of late eluting compounds (PAHs), active sites for acids (PCP, 2,4-dinitrophenol)



# Injection Ports

- Septum
  - 3 types
    - IceBlue (250°C)
    - Thermolite (340°C)
    - BTO (400°C)
  - Recommend Thermolite
- O-rings
  - Max. Temp: 350°C



IceBlue® Septa

Thermolite® Septa

BTO® Septa

**Septa for Agilent GCs** Preconditioned and ready to use!

Septum Diameter	25-pk.	50-pk.	100-pk.
Thermolite Septa (usable to 340°C inlet temp.)			
5mm (1/8")	27120	27121	27122
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143

**High Temperature O-Rings for Agilent GCs** NEW!

- Usable to 350 °C inlet temperature.
- Fits split (6.3 mm OD) and splitless (6.5 mm OD) liners.



Description	Max. temp.	10-pk. cat.#	25-pk. cat.#
High Temperature O-Rings for Agilent GCs	350°C	23403	23404

# Injection Ports

## • Sky Inlet Liners

- Many geometry of liners used
- Recommend single gooseneck w/ wool
  - Inert for active compound
  - Wool traps nonvolatile materials



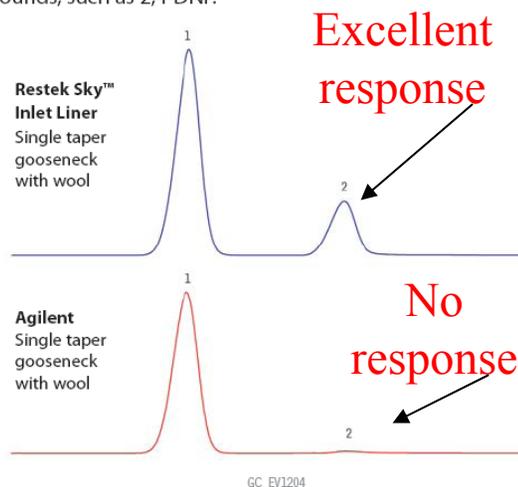
Wool

Gooseneck at bottom of liner



## 2,4-dinitrophenol response

**Figure 2** The state-of-the-art deactivation used for Sky™ liners with wool results in higher responses for active acid compounds, such as 2,4-DNP.



Increase the response of active analytes with inert Sky™ inlet liners!

	2,4-DNP Response Factor
Restek	0.28
Agilent	0.02

Peaks  
1. Acenaphthene (IS)  
2. 2,4-DNP

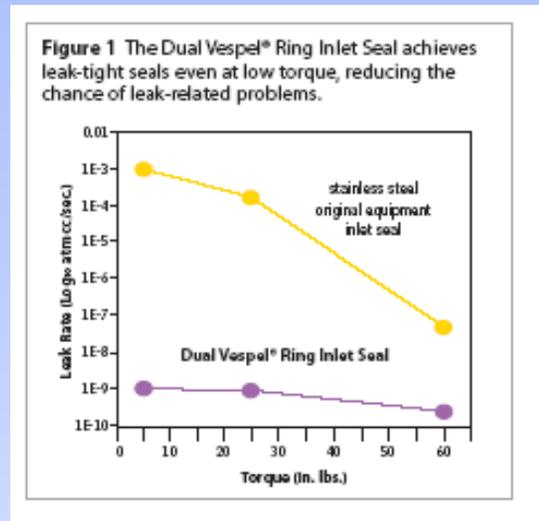
10ng injected

Column Rxi®-5Sil MS, 15 m, 0.25 mm ID, 0.25 μm (cat.# 13620);  
Sample 10 μg/mL each 2,4-dinitrophenol and acenaphthene standard in methylene chloride; Injection Inj. Vol.: 1 μL splitless (hold 1 min.);  
Liner: Comparison of Sky™ Single Taper Gooseneck Inlet Liner with Wool (cat.# 23303.5) and Agilent Single Taper Gooseneck Inlet Liner with Wool (cat.# 5062-3587); Inj. Temp.: 250 °C.

# Injection Port

- Inlet seals

- Recommend Gold Flip Seal
- Double use
- Gold plating is inert for active compounds
- Note: Do need adaptor, 1 time purchase



Easy to seal

Feature	Benefit
Reversible design.	Allows twice as many uses as other seals, at the same cost.
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.

# Sample Extraction for Semi-Volatiles

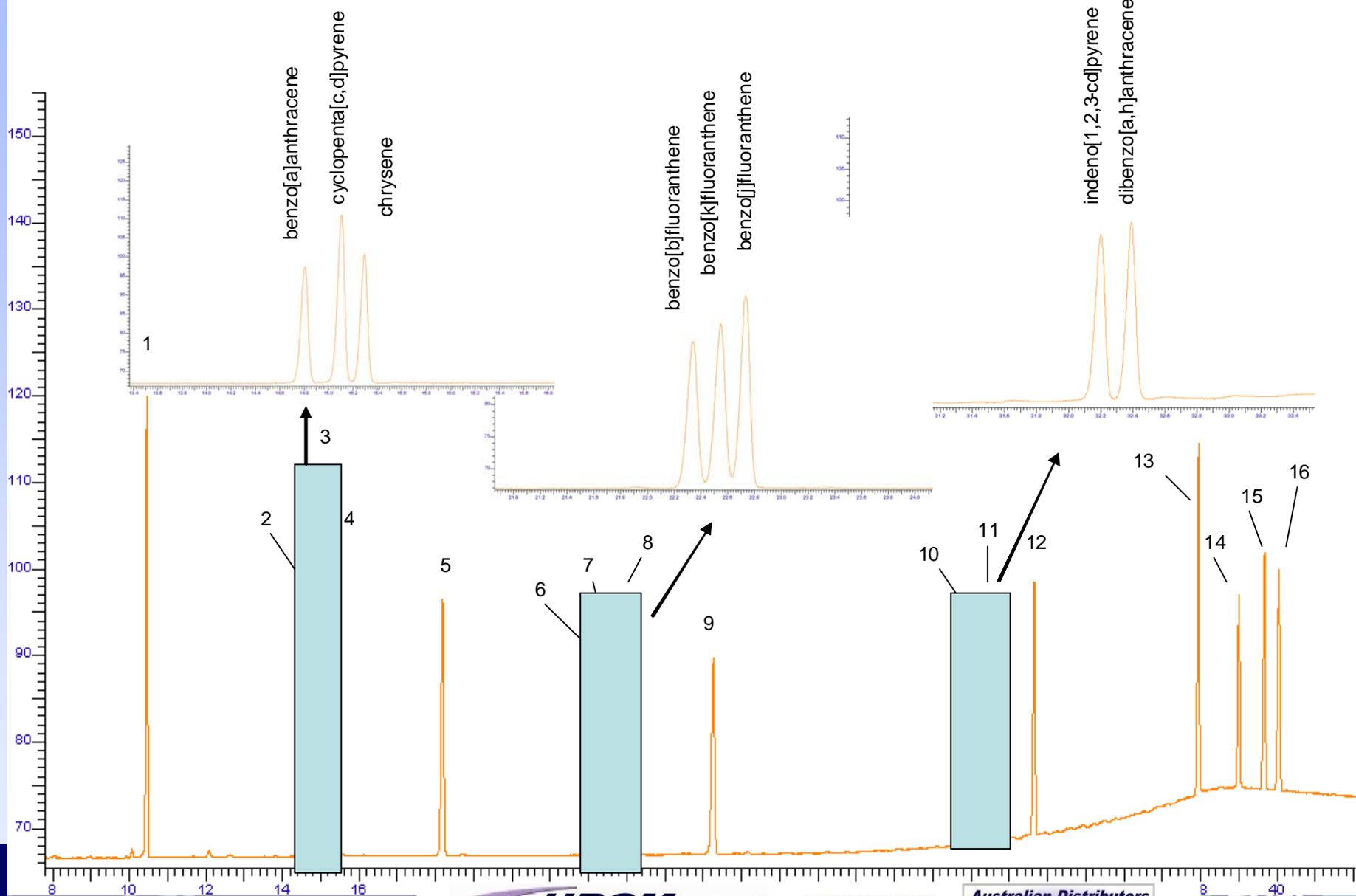
- Extraction Disks
  - 47mm C18, C8, and DVB
  - Drinking water methods
- ASE Replacement Parts
  - Models 200 & 300
  - Cells, PEEK Washers, Filters, tools
  - Used with Dionex automated solvent extractor
    - Soil extraction
- Surrogate & matrix spikes
  - Added to samples prior to extraction
  - Higher concentration & larger package size for lower spike per sample cost
  - QA documentation available
  - On-line data pack



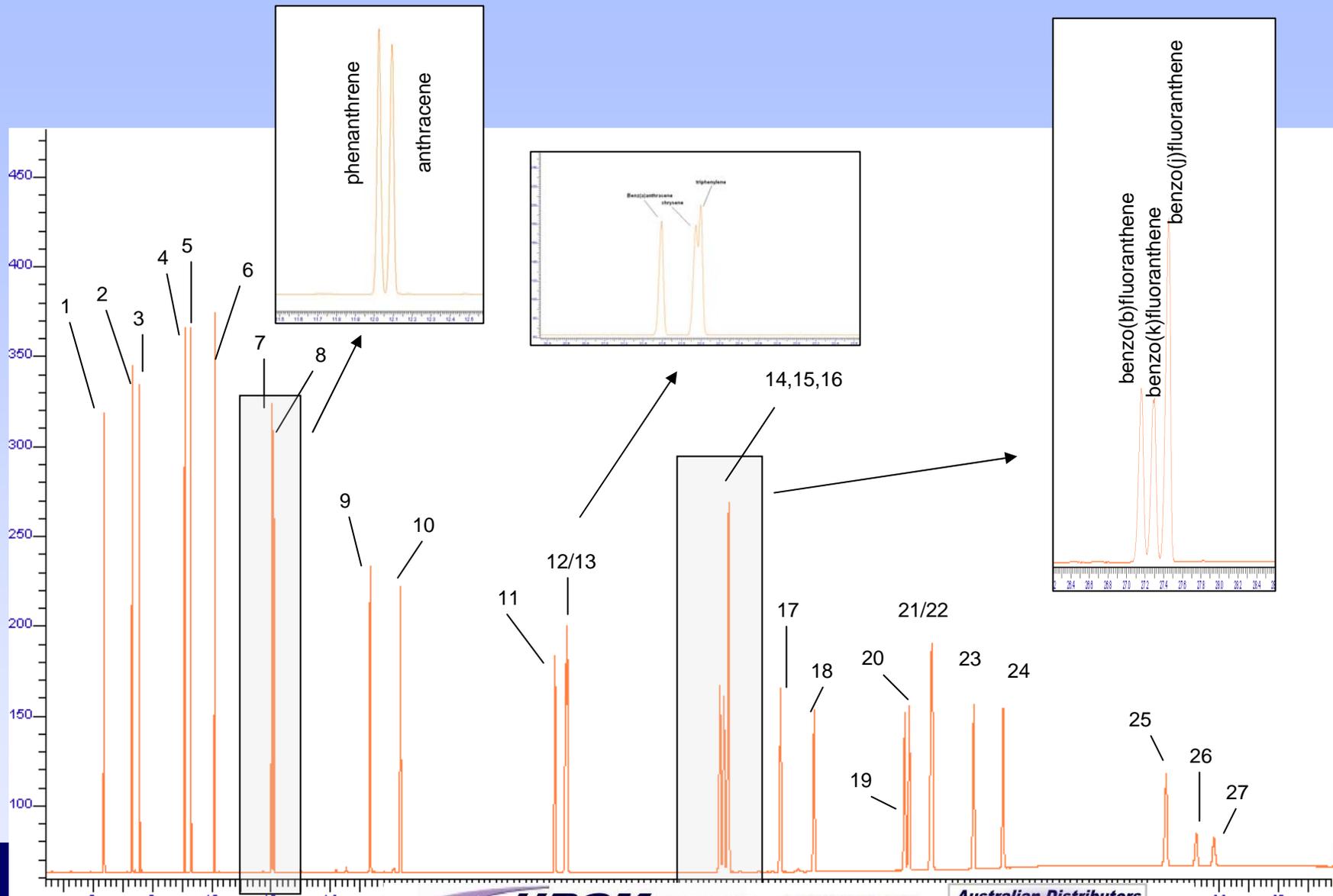
# Polycyclic Aromatic Hydrocarbons (PAHs)

- Rxi-17Sil MS
  - High max temp @ 360°C
  - Column dimension: 30m x 0.25mm, 0.25um
- Competitor columns
  - DB-EUPAH (340°C)
  - Varian Select PAH (350°C)
  - Column dimensions:
    - Columns “down coated”
      - 30m x 0.25mm, 0.15um
      - 20m x 0.15mm, 0.1um
    - We are investigating thinner coating affects

# EU 15 + 1 PAHs using an Rxi-17sil ms (20m x 0.18mm x 0.18 $\mu$ m)



# PAHs using an Rxi-17Sil MS (30m x 0.25mm x 0.25 $\mu$ m)



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# General Chromatography Supplies

- Ferrules



- Ferrule removal kit



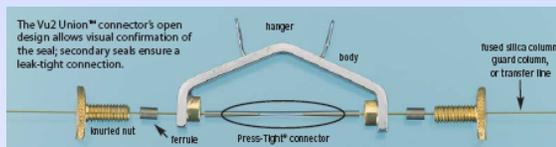
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

# Volatile Analysis

- How laboratory is set-up
- Analysis of volatile compounds
  - Rtx-VMS
  - Rxi-624Sil MS
- Instrument maintenance



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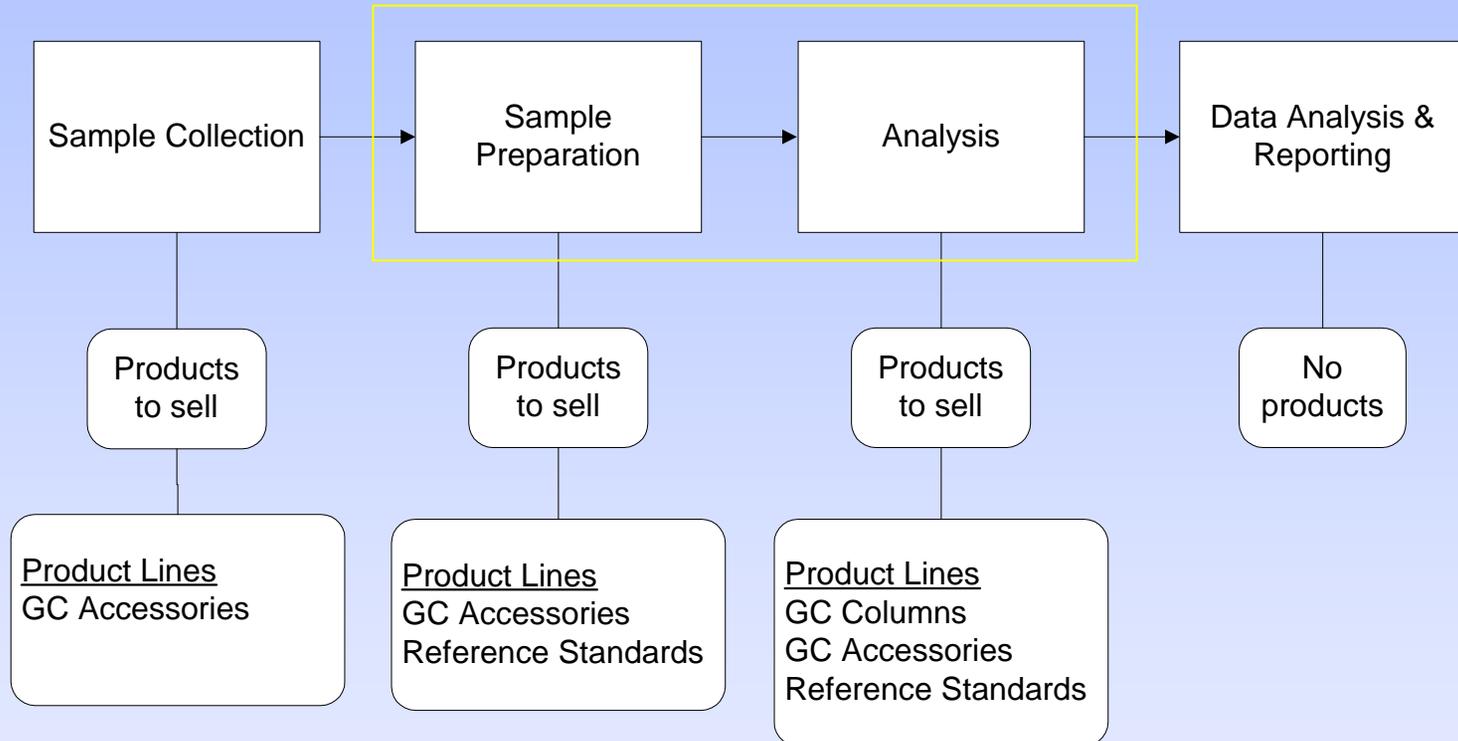


# Volatiles Overview

- Method Summary:
  - Volatile compounds are extracted from sample matrix using a Purge & Trap or headspace concentrator. Analysis is typically done by GC/MS, but GC with selective detectors are also used.
- Compound lists to reference: US EPA Methods 8260, 624, & 502.2
  - [http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/8\\_series.htm](http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/8_series.htm)
- Products offered:
  - GC Columns (Rtx-VMS, Rxi-624Sil MS)
  - Reference Standards
  - Purge & Trap replacement parts (sparger tubes, transfer lines)
  - Pre-cleaned VOA Vials for sample collection

# Volatiles Overview

## Inst. Lab



# Competitive Positioning for Selling into the Volatiles Lab

- Lead with:
  - Rtx-VMS columns (best overall separation, fast analysis time)
  - Mega Mixes (fewest ampoules and lowest overall cost for standards)
  - NEW Column phase: Rxi-624Sil MS
- Follow with:
  - Purge & Trap Supplies
  - VOA Vials, etc.



# Analysis for Volatiles

## GC Columns

- Rtx-VMS
  - First choice for complex volatiles analysis
  - Offers most complete separation
  - Unique phase exclusive to Restek
  - Specifically designed to separate over 100 compounds
  - Optimized analysis time under 12 minutes
  - Available in 0.18, 0.25, 0.32, 0.45, & 0.53mm ID
    - GC/MS Recommend 30m x 0.25mm x 1.4df



# Analysis for Volatiles

## GC Columns – Rtx-VMS

- Available in 0.18, 0.25, 0.32, 0.45, & 0.53mm ID
  - GC/MS *30m x 0.25mm x 1.4df*
    - Selectivity not column length key factor
    - 60m columns have 2x bleed (2x cost to customer)
    - 0.25mm ID better with water/methanol vs. 0.18mm
  - GC *75m x 0.45mm x 2.55df*  
*60m x 0.32mm x 1.80df*
    - Good capacity & excellent sensitivity

# Analysis for Volatiles

## Other GC Columns for Volatiles

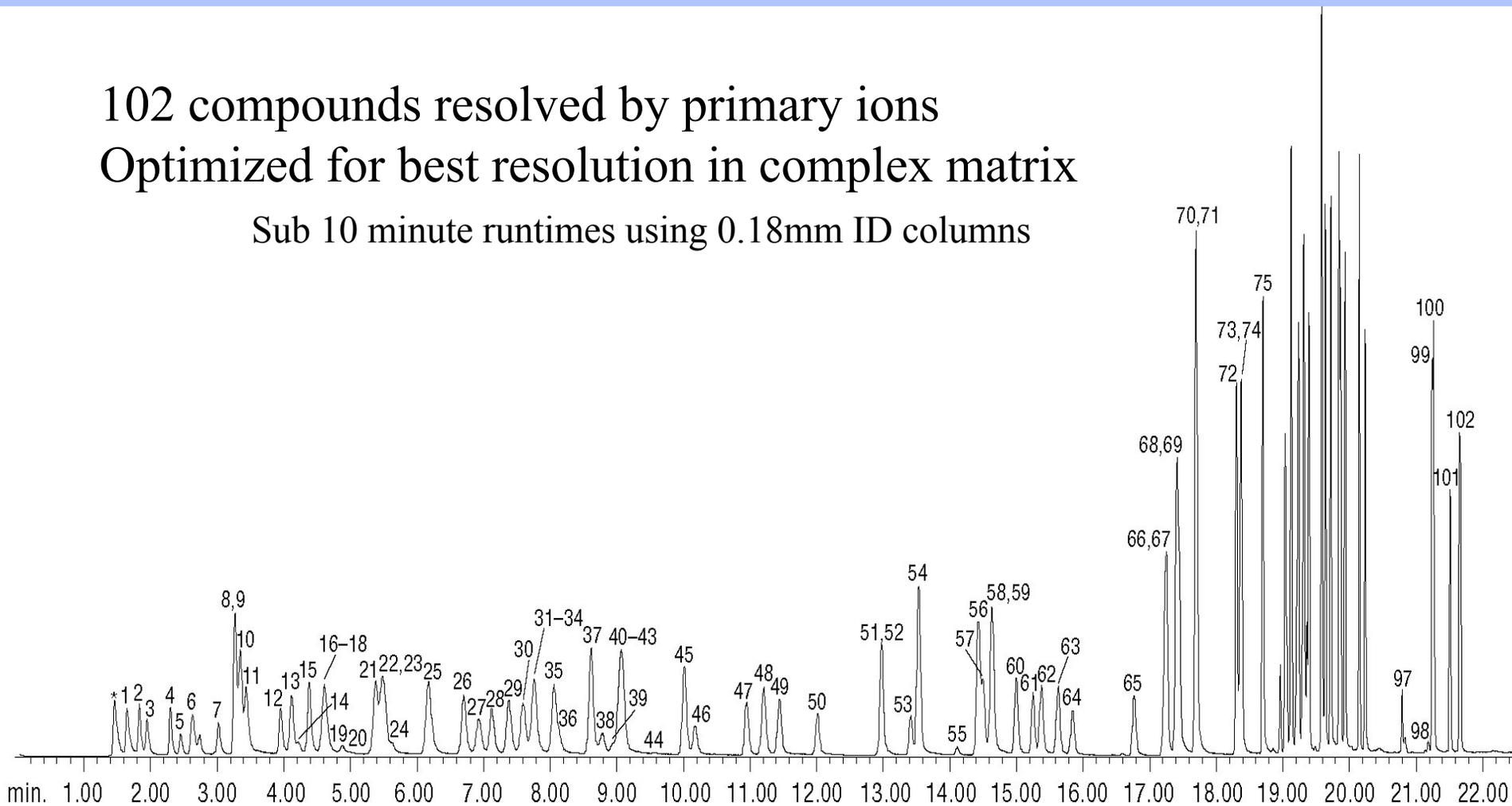
- Competitive Columns
  - DB-VRX, DB-624, DB-502.2, DB-VRX (Agilent/J&W)
  - HP-VOC
  - ZB-624 (Phenomenex)
  - SPB-624 & Vocol (Supelco)
  - VF-624ms & CP Select 624 CB (Varian)
- Other Restek Volatiles Columns
  - **Rxi-624Sil MS**
  - Rtx-502.2
  - Rtx-VRX
  - Rtx-Volatiles



# Rtx<sup>®</sup>-VMS 30m x 0.25mm x 1.4df

102 compounds resolved by primary ions  
Optimized for best resolution in complex matrix

Sub 10 minute runtimes using 0.18mm ID columns



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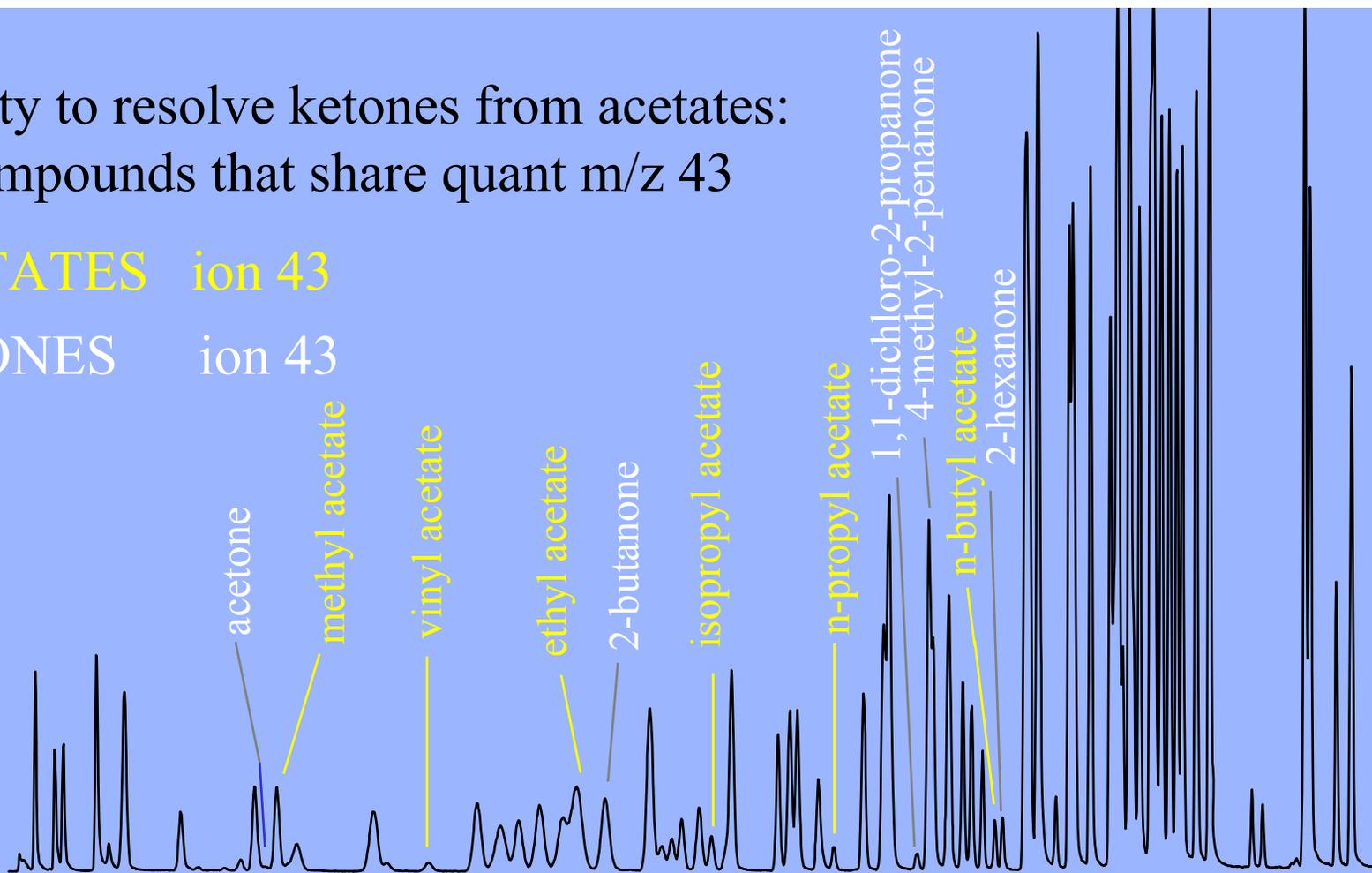


# Rtx<sup>®</sup>-VMS

Ability to resolve ketones from acetates:  
compounds that share quant m/z 43

ACETATES ion 43

KETONES ion 43



# Rtx-VMS advantages over other phases

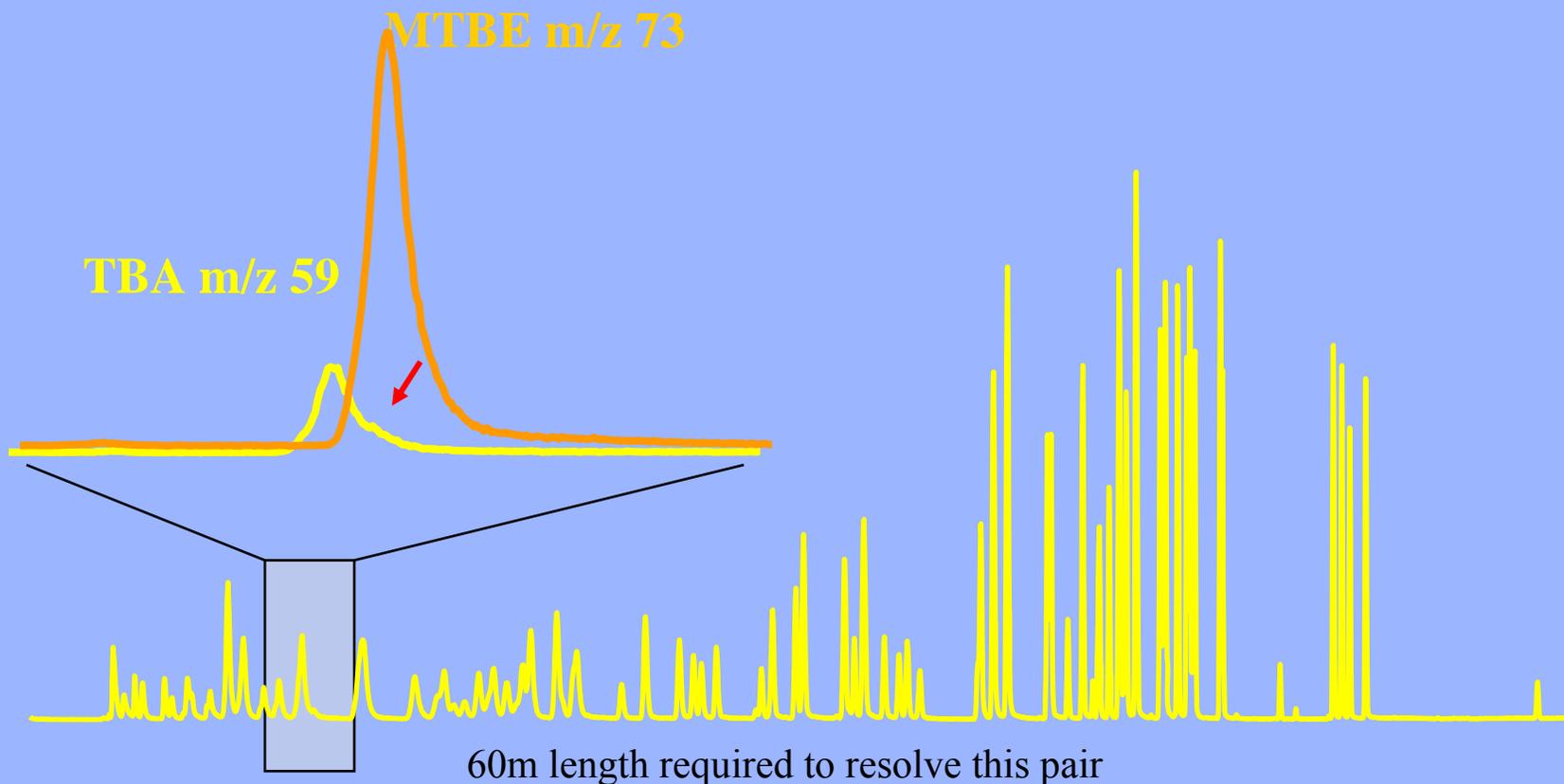
- “624 type phases”
  - Resolution of 2/4-chlorotoluene
  - TBA & MTBE resolution (m/z 59)
  - J&W type: Benzene & 12DCA (m/z 62)
  - GC: C12DCE & 22DCPropane
  - **Rxi-624Sil MS designed not to have these disadvantages**
- “phenyl type phases”

Includes HP-VOC, Rtx-Volatiles, Rtx-502.2, VOCOL

  - Poor resolution of bromomethane & chloroethane
  - Water/Methanol: results in poor gas peak shape
  - Plus: Generally lower bleed & withstand small leaks
- “VRX type phases”
  - Chlorobenzene-D5 (IS) / 1112TCA resolution
  - Bleed similar to 624, VMS
  - GC: Poor resolution of the trihalomethanes

# 624 Phase Disadvantage

MTBE's minor m/z 59 ion contributes to area of TBA

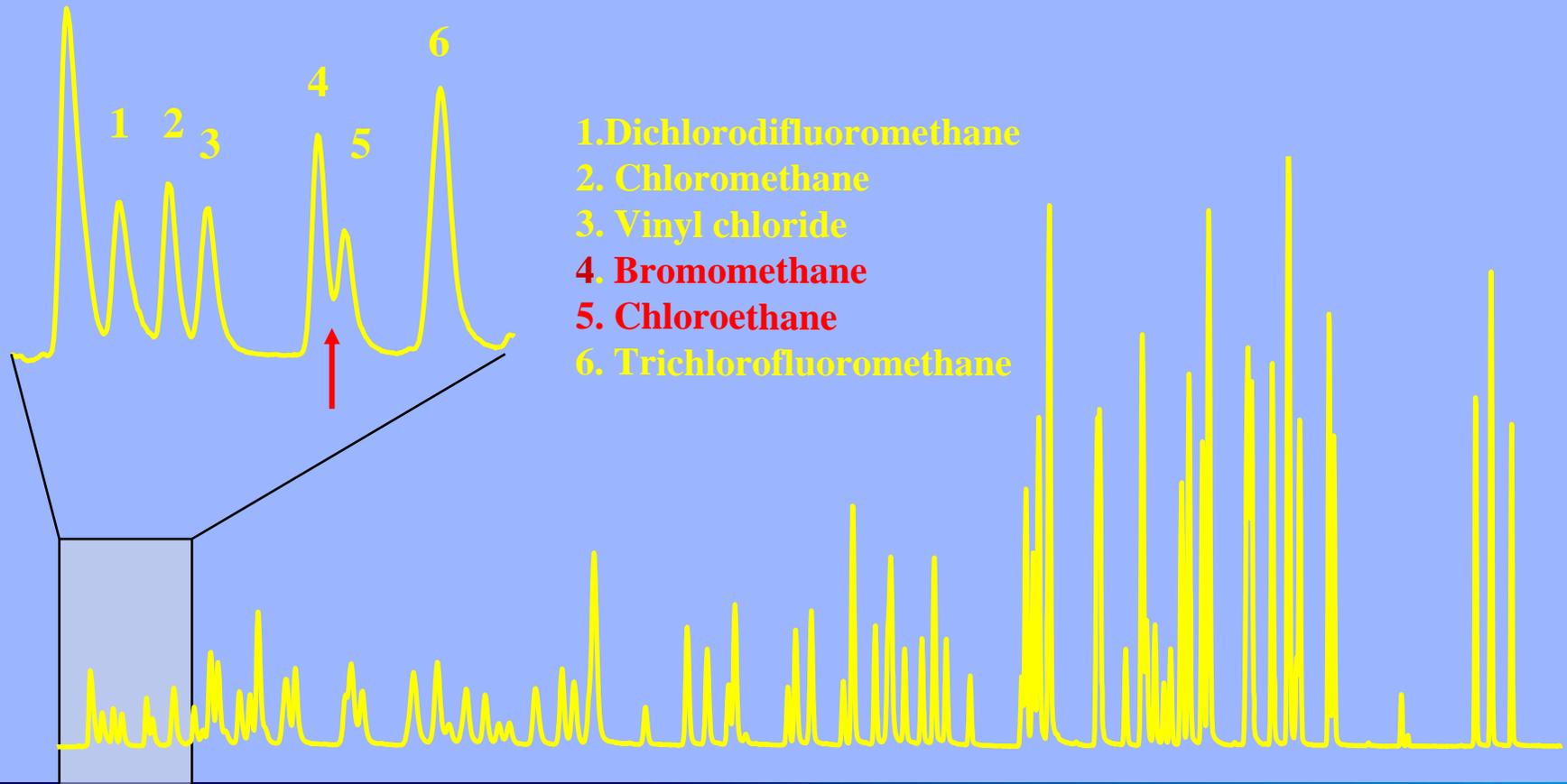


# Rtx-Volatiles (phenyl type phase)

All phenyl type phases have this disadvantage

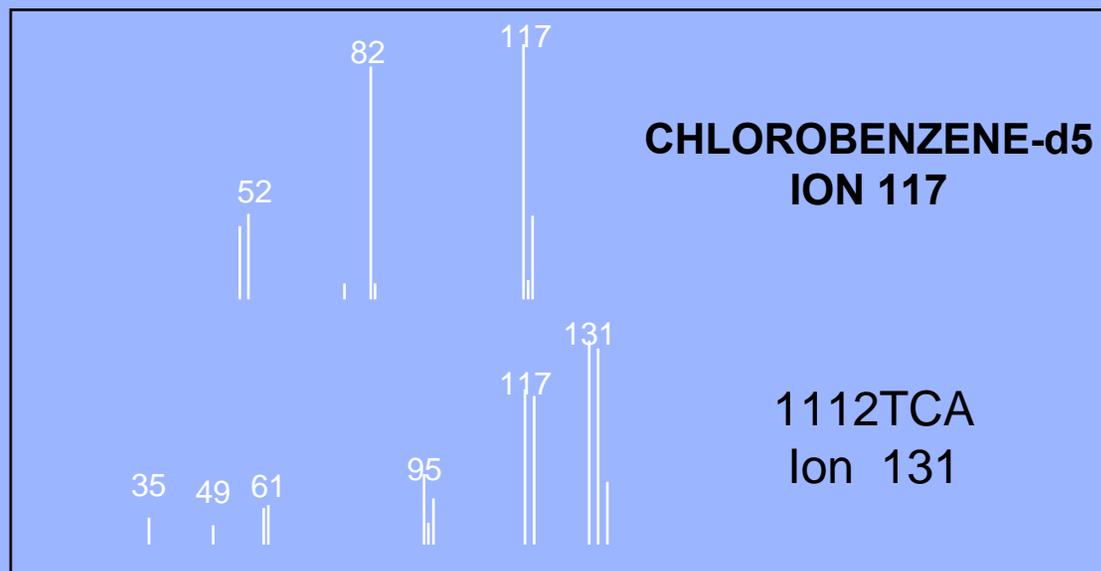
VOA Methods Require 90% Resolution of the Gases

:



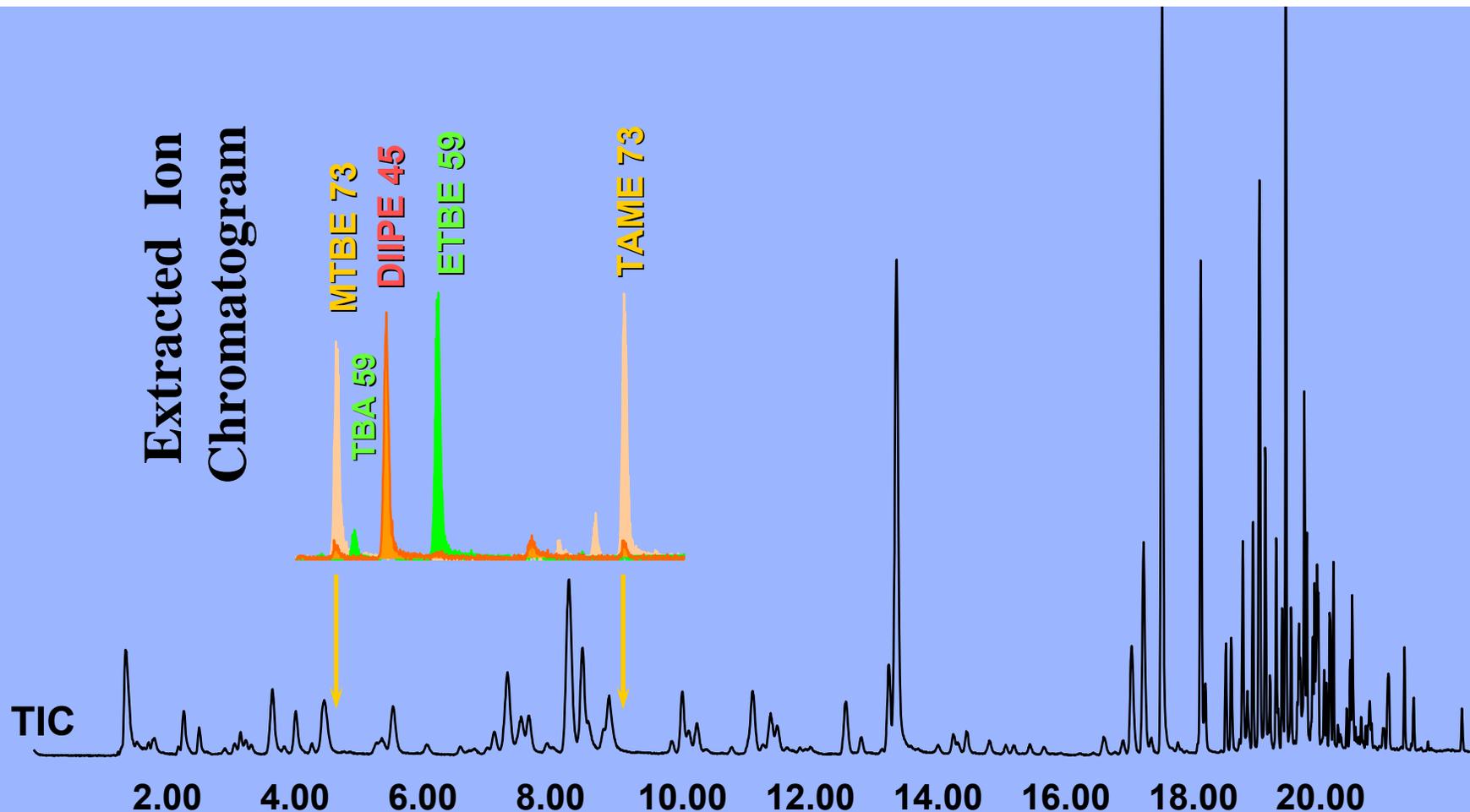
# VRX Phase Disadvantage

Coelution of Internal Standard (IS) and Target Compound,  
where primary ion for IS is  $m/z$  117



Customers *may* be able to use  $m/z$  82 ion

# Rtx-VMS Quantify Target Compounds in a High Concentration Gasoline Matrix: 1ppm Non-Oxygenated Gasoline Standard Spiked with 5ppb Oxygenates

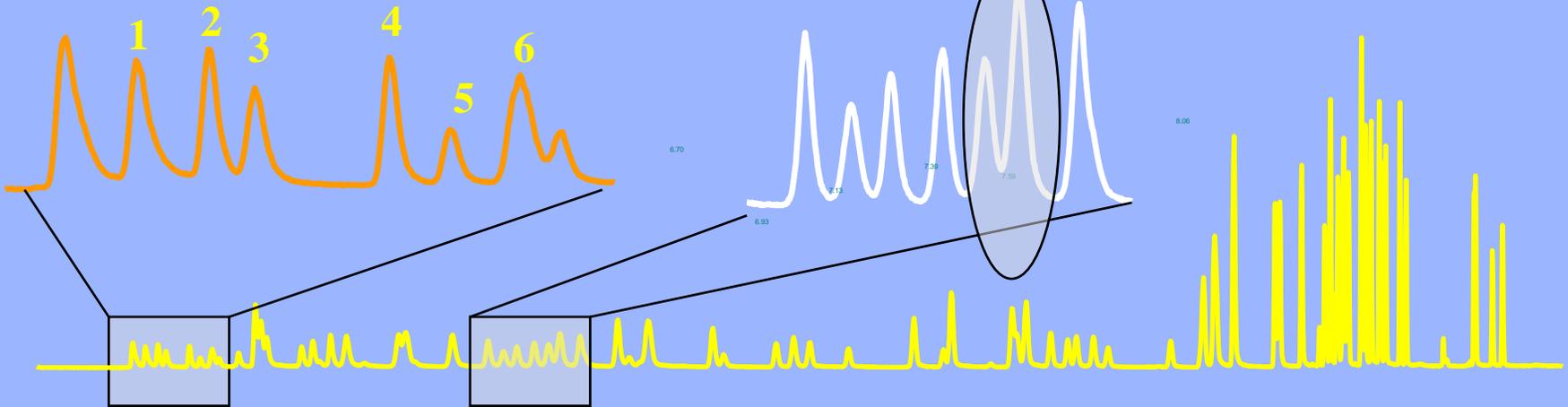


# Rtx-VMS Critical Resolutions

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane

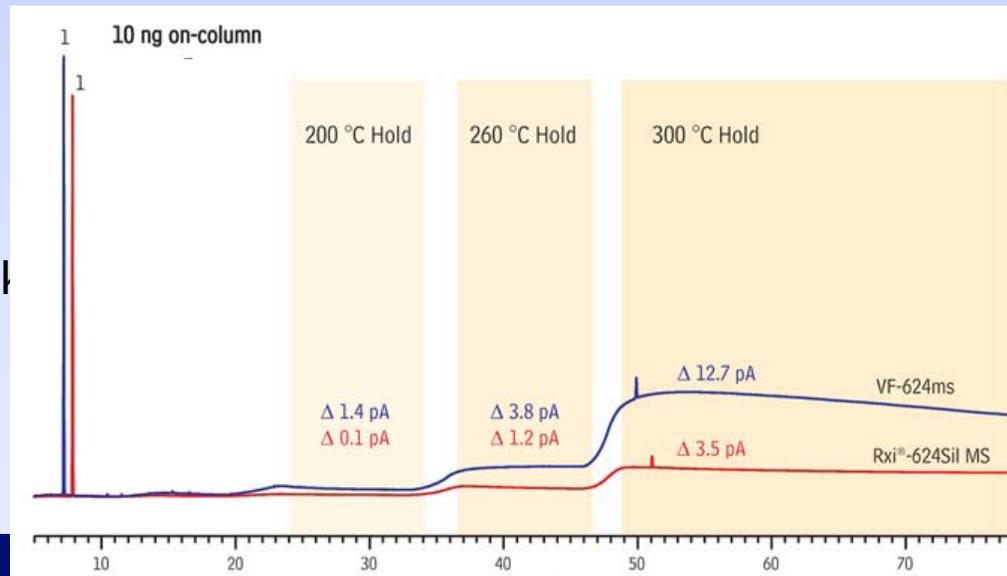
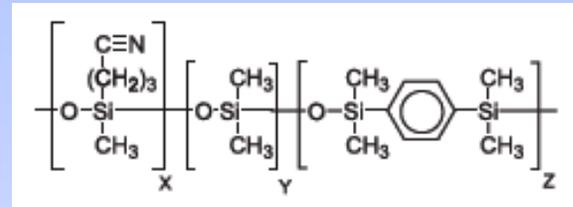
Carbon Tetrachloride  
m/z 117

1,1,1-Trichloroethene  
m/z 97



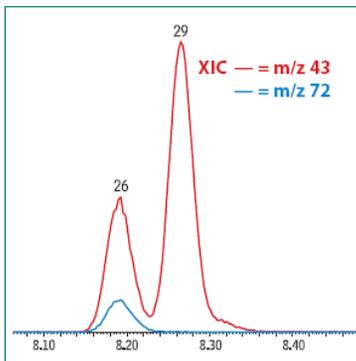
# Volatiles Analysis with **NEW** Rxi-624Sil MS

- New Rxi-624Sil MS
  - Silarylene in backbone of polymer
  - Selectivity similar to DB-625, VF-624ms, ZB-624
- What is different?
  - Max. temp of 320C
  - Lowest bleed column on market



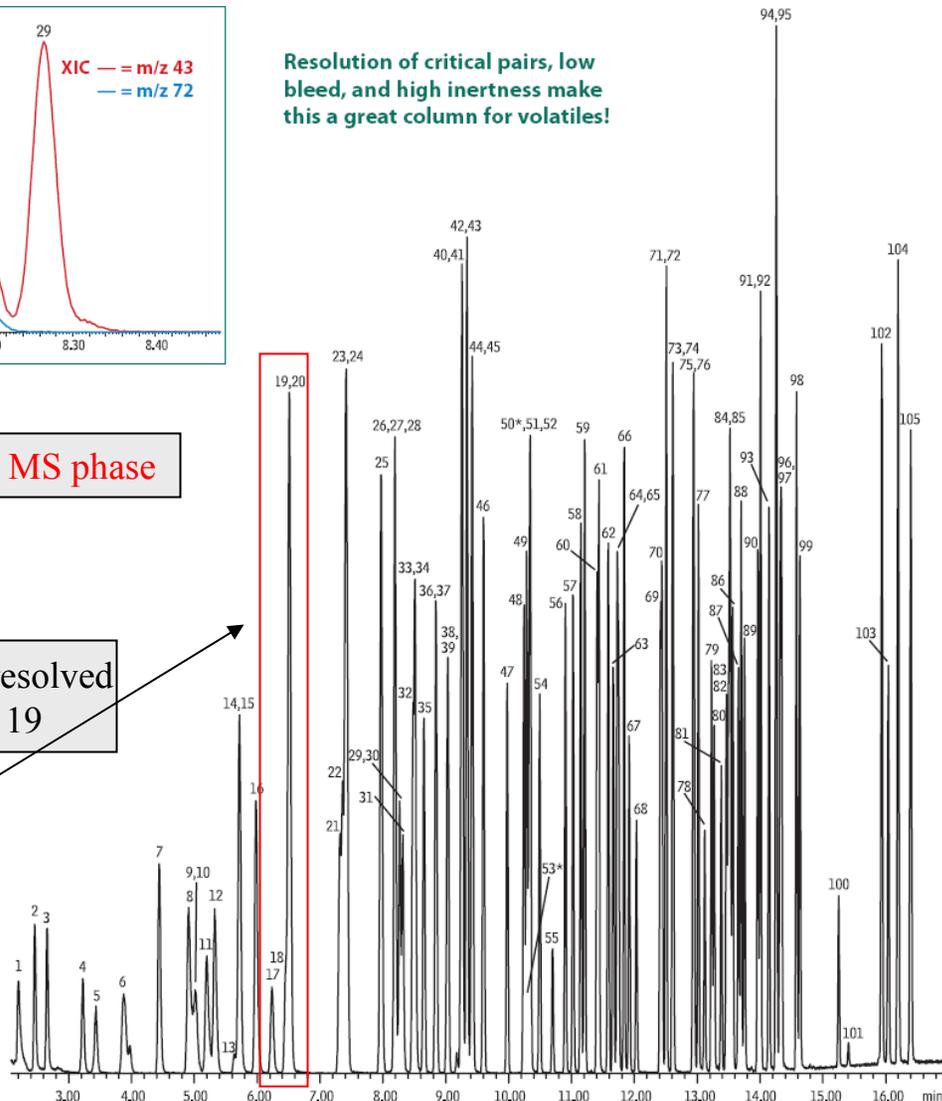
# Volatile Analysis – Rxi-624Sil MS

- 30m x 0.25mm, 1.4um
- Resolves 2-butanol & ethyl acetate
- Less than 17 minute analysis time
- Complete Pro EzGC library for modeling



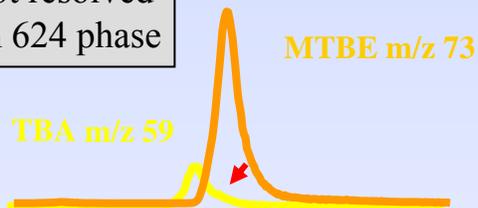
Resolution of critical pairs, low bleed, and high inertness make this a great column for volatiles!

624Sil MS phase



MTBE & TBA resolved  
Peaks 17 & 19

Not resolved  
on 624 phase



# Standards for Volatiles

- Mixtures available for all volatile meth  
  - Mega Mixes (502.2, 624, & 8260 compound list)
    - Less expensive, fewer ampoule alternative
    - Reduces cost and preparation time
    - Designed for maximum stability
  - Individual Components
  - Excellent Selection of Oxygenates & alcohol standards
  - Surrogate & Internal Standards
  - QA Documentation Available on all stock products
    - On-line data packs
- Custom Mixtures Available
  - Designed for customer specific requirements
  - Hybrid quotes (Mega Mix plus lab specific compounds)



# Sample Preparation for Volatiles

- Vials

- Used for making dilutions
- Soil analysis



- Headspace vials

- Syringes

- Used for making dilution
- Spiking standards



- Standards

- Surrogates
- Matrix spikes



5mL
10mL
25mL
50mL
100mL

\*Order needles separately.

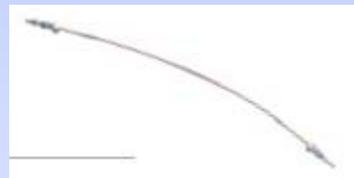
# Other Products for Volatiles

- **Purge & Trap Supplies**

- Spargers for Tekmar systems
- Silcosteel Transfer Lines
- Moisture Control Bypass Lines for Tekmar



- **PID Lamps**



- **ELCD Nickel reaction tubes & other "things"**



# General Chromatography Supplies

- Ferrules



- Ferrule removal kit



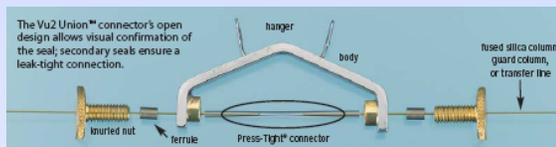
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

# Competitive Positioning for Selling into the Volatiles Lab

- Lead with:
  - Rtx-VMS columns (best overall separation, fast analysis time)
  - Now have direct replacement for DB-624, ZB-624 or ZB-624ms with the Rxi-624Sil MS
  - Mega Mixes (fewest ampoules and lowest overall cost for standards)
- Follow with:
  - Purge & Trap Supplies
  - VOA Vials, etc.



# Pesticide Analysis



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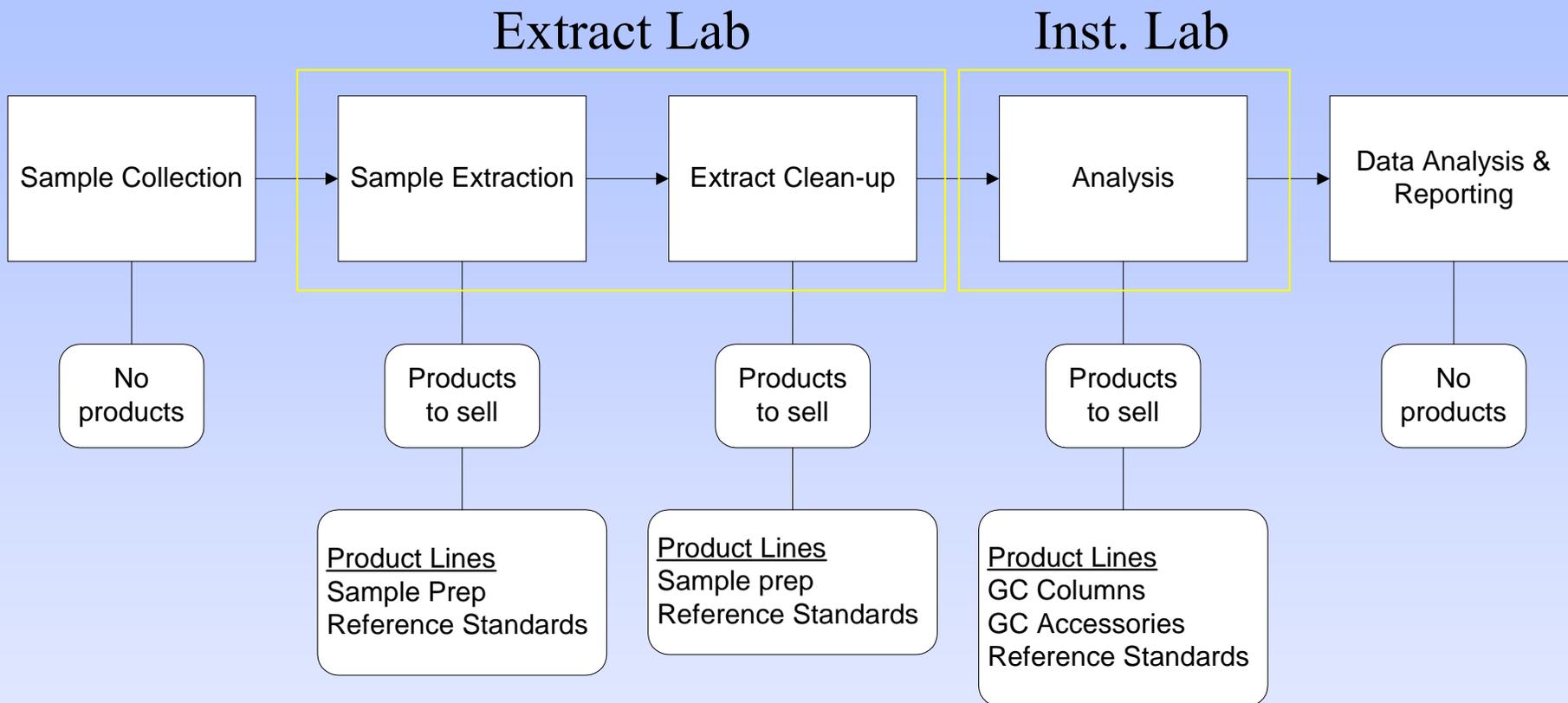
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# Chlorinated Pesticide Overview

- Method Summary:
  - Pesticides are extracted from sample matrix using an appropriate solvent. The extract is concentrated and cleaned to remove interferences. An aliquot of the extract is typically analyzed by GC with MS detection.
- Products offered:
  - GC Columns (Rxi-5Sil MS)
  - Reference Standards
  - Inlet Supplies (liners, inlet seals, septa)
  - Sample prep products (SPE Tubes, Adsorbents, ASE Parts)
  - Autosampler Vials & Syringes

# Sample Process for Pesticides



# Sales Strategy for Pesticides

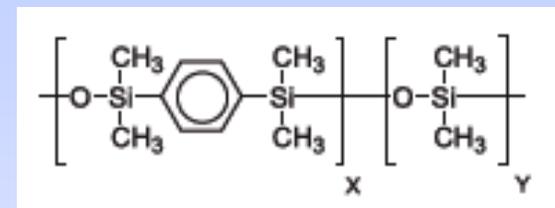
- Lead with:
  - Rxi-5Sil MS columns
    - 30m x 0.25mm, 0.25um
  - Sky Inlet Liners
    - First suggestion is single gooseneck w/ wool
      - Cyclo double gooseneck
      - Drilled Uniliner for maximum transfer of compounds and inertness
  - Calibration mixes
    - Multi component mixes
    - Surrogate & matrix spikes
    - Custom mixes
  - Flip Seal (Reversible Dual Vespel Inlet Seal)
  - CarboPrep SPE tubes for cleaning extracts
    - Chlorinated and organophosphorus pesticides evaluated
- Follow with:
  - Other instrument supplies
  - Sample Prep products
  - Vials & Syringes

# Rxi-5Si1 MS

## GC Column Technology



- Highest inertness
- Lowest bleed
- Exceptional reproducibility

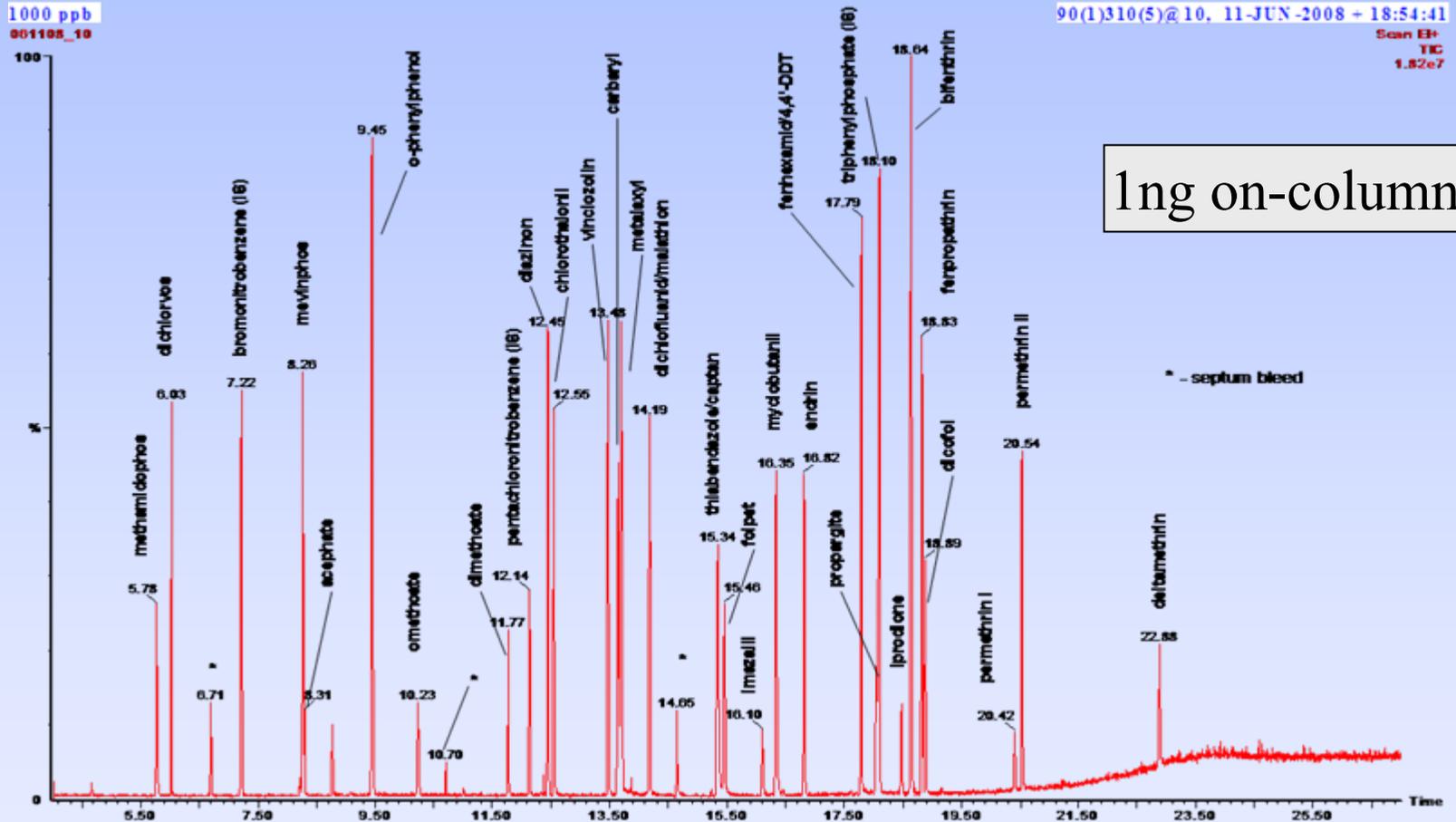


Rxi-5Si1 MS

- Combination of features are what is important for environmental analysis of pesticides

# Pesticide Analysis by GC/MS

Figure 1. Total Ion Chromatogram of Pesticides @ 1ppm using Rxi-5sil ms



**Table 1. MS Acquisition Parameters Rxi-5sil ms**

Column: 30m x 0.25mm x 0.25µm (cat.# 13623) serial # 880676

Instrument: Perkin Elmer Clarus 500 GC/MS

Conditions: Oven program: 90°C (1 min.) → 310°C (5 min.) @ 10°C/min.

Inj: 250°C 0.5µL; 1.5 ml/min. constant flow - He; Splitless injection with Uniliner

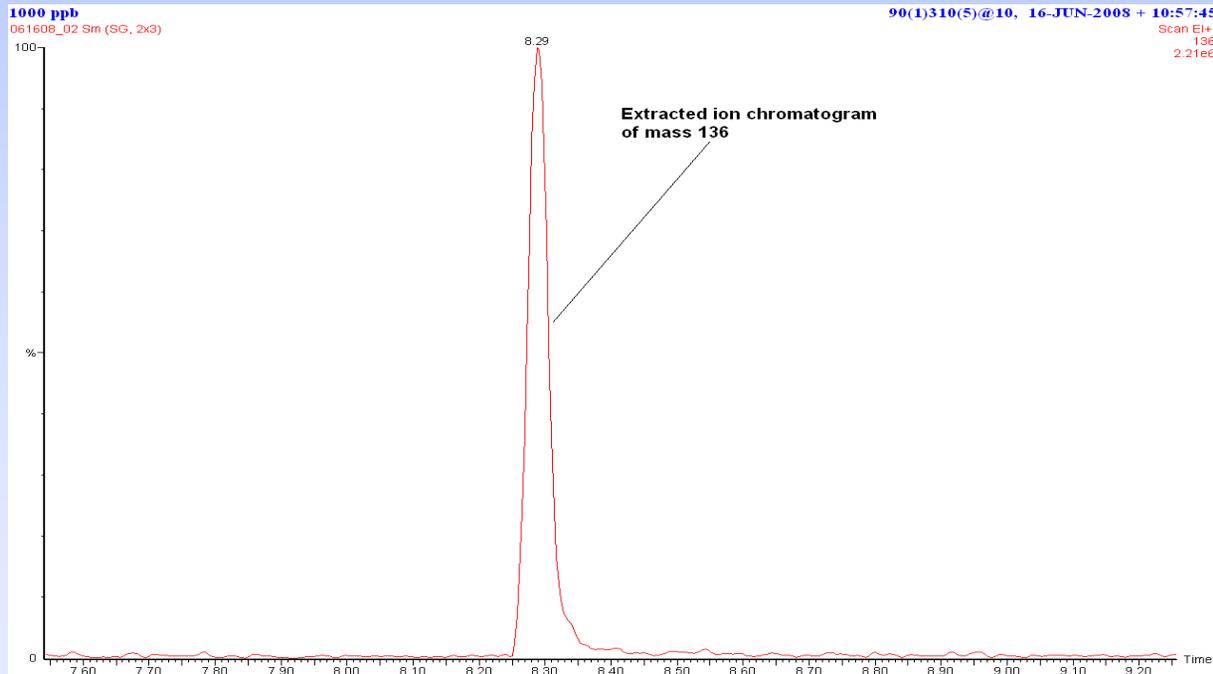
Det: Interface: 300°C; Source: 280°C; SIR mode

Compound	rt	Quantr	Qual 1	Qual 2	CAS#	MW	func#	start	end	IS	r <sup>2</sup> (0.01 - 1 ppm)
methamidophos	5.77	141	95	94	10265-92-6	141	1	5.67	5.87	BNB	0.997
dichlorvos	6.02	185	79	109	62-73-7	220	2	5.92	6.12	BNB	0.998
bromonitrobenzene (IS)	7.21	203	201	157	577-19-5	201	3	7.11	7.31	IS	—
mevinphos	8.26	192	127	109	7786-34-7	224	4	8.16	8.36	BNB	0.995
acephate	8.30	136	95	94	30560-19-1	183	5	8.20	8.40	BNB	0.982
o-phenylphenol	9.44	170	169	141	90-43-7	170	6	9.34	9.54	BNB	0.997
omethoate	10.23	156	110	109	1113-02-6	213	7	10.13	10.33	BNB	0.976
dimethoate	11.77	125	143	93	60-51-5	229	8	11.67	11.87	BNB	0.981
pentachloronitrobenzene (IS)	12.13	295	249	237	82-68-8	293	9	12.03	12.23	IS	—
diazinon	12.45	179	304	137	333-41-5	304	10	12.35	12.55	PCNB	0.994
chlorothalonil	12.55	266	264	268	1897-45-6	264	11	12.45	12.65	PCNB	0.983
vinclozin	13.48	285	198	212	50471-44-8	285	12	13.38	13.58	PCNB	0.998
carbaryl	13.65	144	116	115	63-25-2	237	13	13.55	13.75	PCNB	0.996
metalaxyl	13.69	206	160	132	57837-19-1	279	14	13.59	13.79	PCNB	0.997
dichlofuanid	14.17	123	167	224	1085-98-9	332	15	14.07	14.27	PCNB	0.954
malathion	14.19	173	125	127	121-75-5	330	16	14.09	14.29	PCNB	0.992
thiabendazole I	15.34	201	202	174	148-79-8	201	17	15.24	15.44	PCNB	0.958
captan	15.34	79	119	149	133-06-2	299	18	15.24	15.44	PCNB	0.987
folpet	15.46	260	130	104	133-07-3	295	19	15.36	15.56	PCNB	0.964
imazalil	16.10	215	175	173	35554-44-0	296	20	16.00	16.20	PCNB	0.982
myclobutanil	16.34	206	179	150	88671-89-0	289	21	16.24	16.44	PCNB	0.973
endrin	16.82	265	279	317	72-20-8	378	22	16.72	16.92	PCNB	0.989
fenhexamid	17.79	177	179	97	126833-17-8	302	23	17.69	17.89	PCNB	0.969
4,4'-DDT	17.79	237	235	165	50-29-3	352	24	17.94	18.14	PCNB	0.998
propargite	18.04	173	150	135	2312-35-8	350	25	17.94	18.14	PCNB	0.999
triphenylphosphate (IS)	18.09	325	215	326	115-86-6	326	26	17.99	18.19	IS	—
iprodione	18.47	314	316	187	36734-19-7	329	27	18.37	18.57	TPP	0.991
bifenthrin	18.64	181	166	165	82657-04-3	423	28	18.54	18.74	TPP	0.998
fenprothrin	18.82	265	208	181	39515-41-8	349	29	18.72	18.92	TPP	0.985
dicofol	18.89	139	251	253	115-32-2	368	30	18.79	18.99	TPP	0.788
permethrin I	20.41	183	165	163	52645-53-1	390	31	20.31	20.64	TPP	0.998
permethrin II	20.54	183	163	165	52645-53-1	390	31	20.31	20.64	TPP	0.995
deltamethrin	22.87	253	251	181	52918-63-5	505	32	22.77	22.97	TPP	0.995

# Acephate @ 1ng on-column

- Very sensitive to active sites in the injection port and analytical column

Figure 3. EIC of acephate @ 1 ppm



# Injection Ports

- Activity from heated inlet systems
  - Some pesticide compounds more active than others
  - Endrin & DDT are 2 compounds sensitive to inlet activity
- How to prevent activity
  - Sky Inlet liners
  - Flip Seals
  - Replacement parts

# Injection Ports

## • Sky Inlet Liners

- Many geometry of liners used
- Recommend single gooseneck w/ wool



- Inert for active compound
- Wool traps nonvolatile materials

Wool

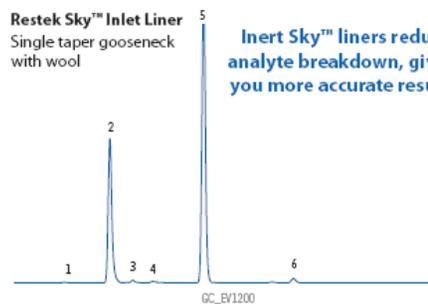
Gooseneck at bottom of liner



**Figure 1** Endrin and DDT breakdown is significantly reduced with Sky™ liners, demonstrating higher inertness.

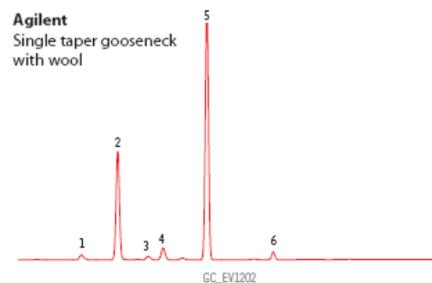
Restek Sky™ Inlet Liner  
Single taper gooseneck  
with wool

Inert Sky™ liners reduce  
analyte breakdown, giving  
you more accurate results.



	% Breakdown	
	endrin	DDT
Restek	4.8	1.3
Agilent	16.5	4.2

Peaks  
1. DDE\*  
2. Endrin  
3. DDD\*  
4. Endrin aldehyde\*  
5. DDT  
6. Endrin ketone\*  
\*breakdown products

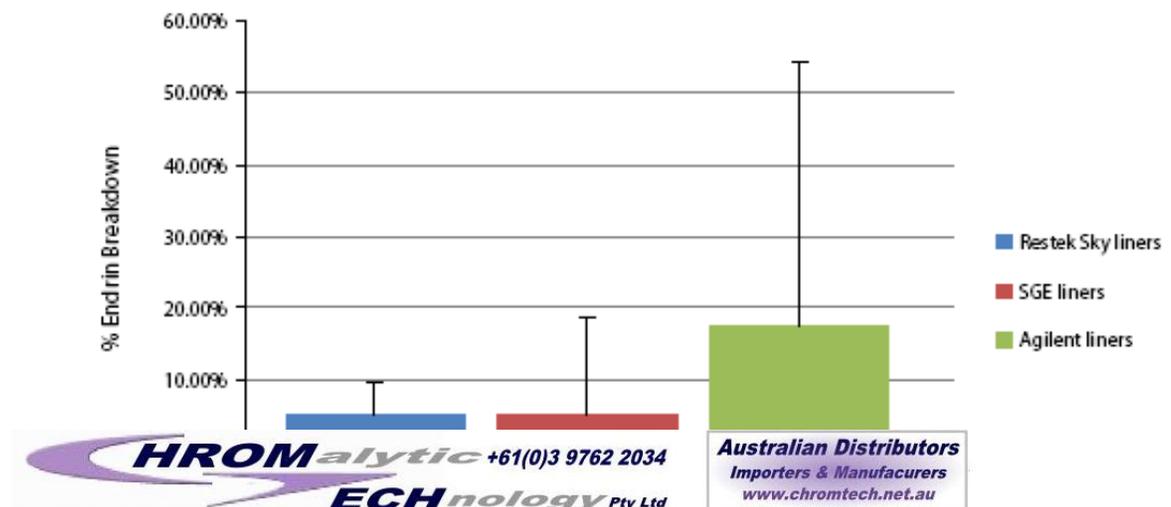


Column Rxi™-5Sil MS, 15 m, 0.25 mm ID, 0.25 μm (cat.# 13620); Sample endrin (50 ng/mL) and DDT (100 ng/mL) in hexane; Injection Inj. Vol.: 1 μL splitless (hold 0.75 min.); Liner: Comparison of Sky™ Single Taper Gooseneck Liner with Wool (cat.# 23303.5) and Agilent Single Taper Gooseneck Liner with Wool (cat.# 5062-3587); Inj. Temp.: 250 °C.

# Variation of Inlet Liners

- Due to activity of some of the pesticide compounds variation from liner to liner is important
- The Sky Inlet Liner deactivation has the least variation of liners in the market

**Figure 4** Sky™ inlet liners from Restek consistently show less endrin breakdown than comparable liners from other sources.



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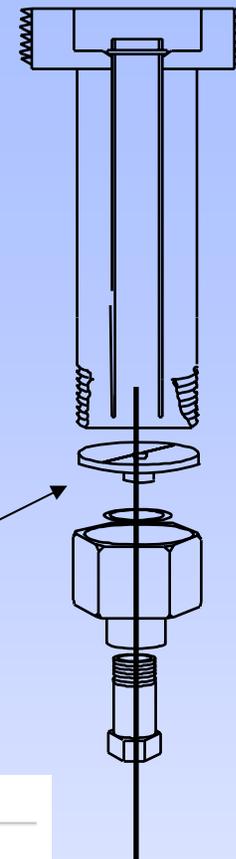
# Injection Port

- Inlet seals

- Recommend Gold Flip Seal
- Double use
- Gold plating is inert for active compounds
- Note: Do need adaptor, 1 time purchase



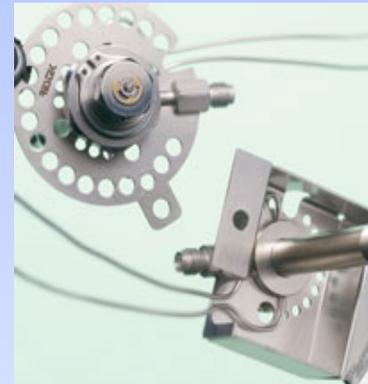
Inlet seal



Feature	Benefit
Reversible design.	Allows twice as many uses as other seals, at the same cost.
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.

# Injection Port

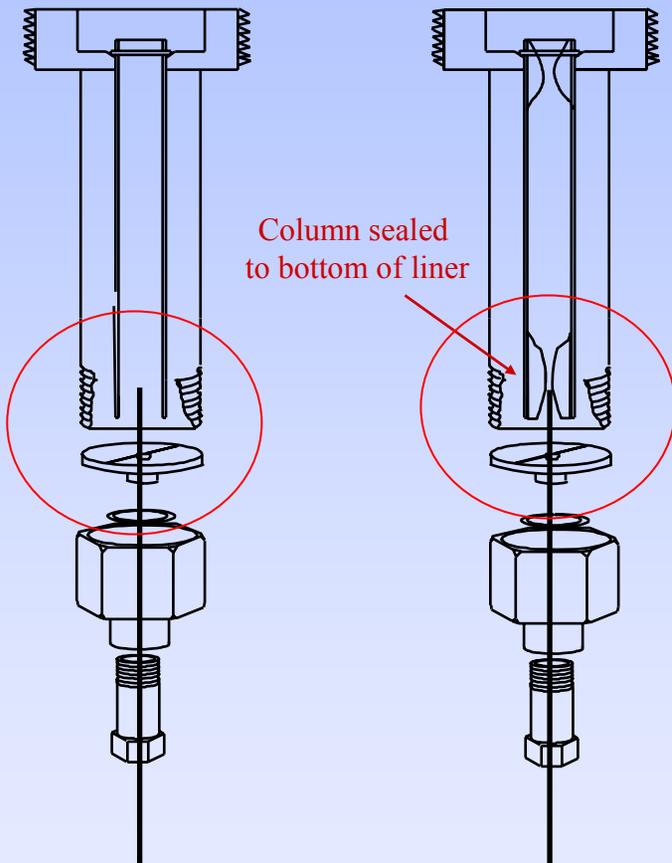
- Replacement Injection Ports
  - EZ Twist Top
  - Not available in Germany, Japan
- Other instrument replacement parts offered
  - Support Agilent, Shimadzu, Perkin Elmer, Thermo



# For Most Inert Inlet System

Gooseneck Liners

Drilled Uniliner



## Affect of Drilled Uniliner

- Very inert sample pathway
- “Funneling” all sample to column
- Most effective inlet system
- No inlet seal exposure



# Consumable Products for Pesticides

- Autosampler Vials

- Screw Cap
- Crimp Top
- Certified Vials – New



- Autosampler Syringes

- SGE
- Hamilton



# Sample Extraction for Pesticides

- Extraction Disks
  - 47mm C18, C8, and DVB
  - Drinking water methods
- ASE Replacement Parts
  - Models 200 & 300
  - Cells, PEEK Washers, Filters, tools
  - Used with Dionex automated solvent extractor
    - Soil extraction
- Surrogate & matrix spikes
  - Added to samples prior to extraction
  - Higher concentration & larger package size for lower spike per sample cost
  - QA documentation available
  - On-line data pack



# SPE Products for Specific Compounds

Description	Applications	Tube Volume, Bed Weight	qty.	cat.#
EPH Fractionation	Extraction of hexane-extractable petroleum hydrocarbons from soil and waste samples. Specially treated to reduce contaminants and increase capacity. Silica packing.	20mL, 5g	20-pk.	26065
EPA Methods 521 & 522	For use in EPA Method 521: Nitrosamines in Drinking Water and EPA Method 522 for 1,4-Dioxane in Drinking Water. Activated charcoal packing.	6mL, 2g	30-pk.	26032
EPA Method 548.1	Extraction of endosulfan from aqueous samples. Weak anion exchange resin (BioRex 5) packing.	6mL	30-pk.	26063
Ultra Quat SPE	For use in HPLC analysis of paraquat/diquat, as an alternative to EPA 549.2. For an HPLC column developed specifically for this application, see page 189.	6mL, 500mg	30-pk.	25499
EPA Method 8321 (AH SPE)	For use in HPLC analysis of phenoxy acid herbicides.	6mL, 500mg	30-pk.	26029
Organo Tin	High-capacity clean-up of butyl and phenyl tin compounds from soil, water, and biota. Mixed bed.	60mL	16-pk.	24049
Drug Prep I	Suitable for acidic, basic, and neutral drugs. Mixed-mode hydrophobic and ion exchange sites provide selective, reproducible extraction of biological samples containing therapeutic or illegal drugs.	3mL, 200mg 10mL, 200mg	50-pk. 50-pk.	26044 26045
Drug Prep II	Extraction of THCA from biological samples. Copolymeric anion exchange sites provide selective, reproducible extraction of THCA from urine.	3mL, 200mg 10mL, 200mg	50-pk. 50-pk.	26046 26047
GHB	Extraction of $\gamma$ -hydroxybutyric acid from biological samples. Provides reproducible extraction of GHB from urine, blood, or tissue without conversion to GBL ( $\gamma$ -butyrolactone).	10mL, 200mg	50-pk.	26208
RDX	Extraction of explosive compounds (similar to EPA Method 8095 and 8330 list) from water samples.	6mL, 500mg	30-pk.	26093

# Extract Clean-up for Pesticides

- Typically clean-up is performed

- Florisil & Silica gel tubes
- CarboPrep



- Bulk Adsorbents

- Florisil PR
- Activated Copper
- Sodium Sulfate



# Reference Materials for Pesticides

- Mixtures available for all US EPA Methods
  - Pesticide Mixes
    - QA documentation available on all stock products
    - On-line data-pack
  - Individual Pesticides
  - Surrogate & Internal Standards
- Custom Mixtures Available
  - Designed for customer specific compound lists



# Sales Strategy for Pesticides Summary

- Lead with:
  - Rxi-5Sil MS columns
    - 30m x 0.25mm, 0.25um
  - Sky Inlet Liners
    - First suggestion is single gooseneck w/ wool
      - Cyclo double gooseneck
      - Drilled Uniliner for maximum transfer of compounds and inertness
  - Calibration mixes
    - Multi component mixes
    - Surrogate & matrix spikes
    - Custom mixes
  - Flip Seal (Reversible Dual Vespel Inlet Seal)
  - CarboPrep SPE tubes for cleaning extracts
    - Chlorinated and organophosphorus pesticides evaluated
- Follow with:
  - Other instrument supplies
  - Sample Prep products
  - Vials & Syringes

# General Chromatography Supplies

- Ferrules



- Ferrule removal kit



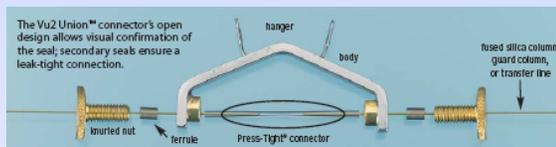
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

# AIR SAMPLING



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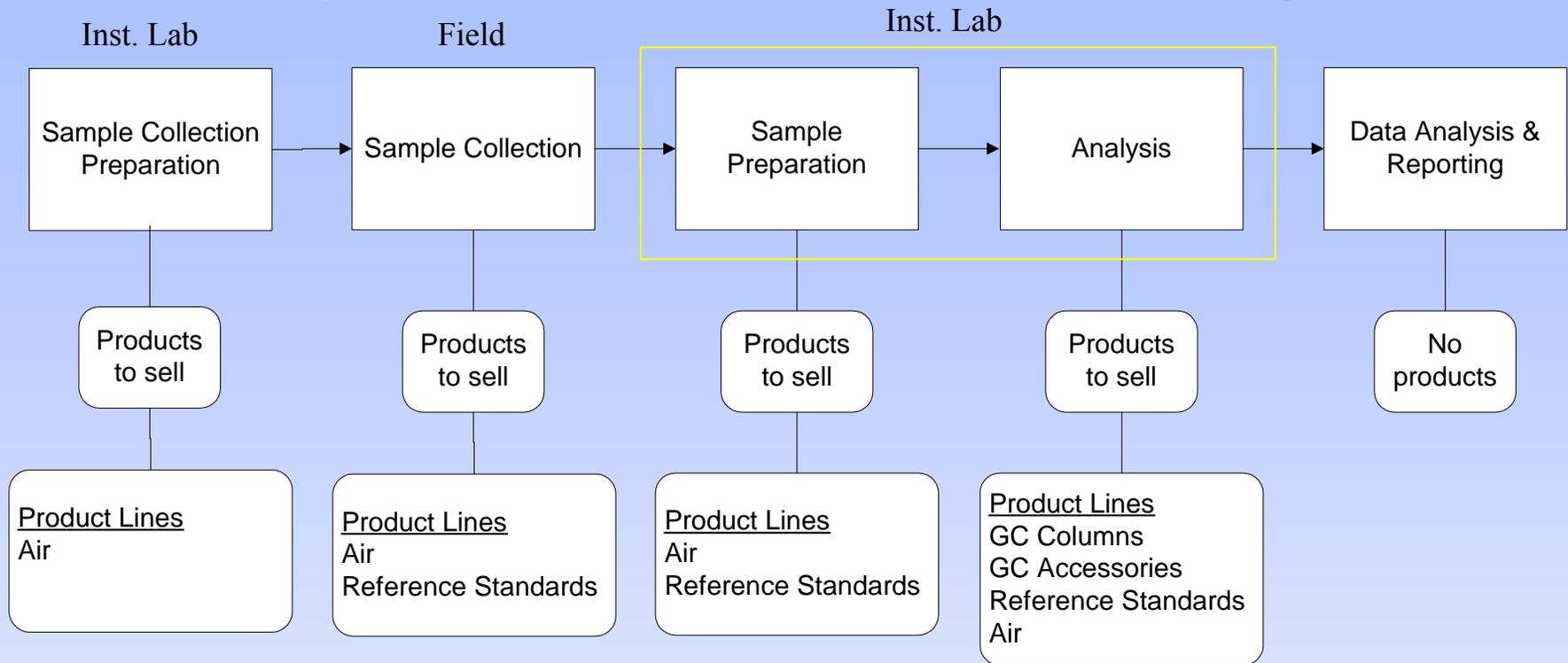
Website NEW : [www.chromalytic.com.au](http://www.chromalytic.com.au) E-mail : [info@chromtech.net.au](mailto:info@chromtech.net.au) Tel: 03 9762 2034 . . . in AUSTRALIA



# Air Monitoring Overview

- Method Summary:
  - Volatile organic contaminants in air samples are collected in canisters or on thermal desorption tubes. The collected volatile compounds are transferred to a GC or GC/MS system using a sample concentrator or thermal desorption system.
  - Semi-volatile contaminants are collected on adsorbents which are extracted using an appropriate solvent. The solvent is concentrated and an aliquot of the extract is typically analyzed by GC with a selective detector, or by GC/MS.
- Methods: TO-13 (SV), TO-15 & TO-17 (VOCs)
- Products offered:
  - Canisters (Silcocans & TO-Cans)
  - Passive sampling Kits
  - Sample Timer
  - Thermal Desorption Tubes
  - Gas Sampling Bags
  - Gas Standards
  - Semi-volatile sampling products

# Sample Process for Air Analysis



## Three sampling techniques supported

1. Canisters (Volatiles)
2. Thermal desorption tubes (Volatiles)
3. High volume sampling (semi-volatiles)

# Competitive Positioning for Selling into the Air Lab

- Lead with:
  - Canisters
    - Large inventory for fast Delivery
    - Available Siltek coated and electro-polished
    - 3 valve options
  - Passive sampling kits
    - Available Siltek coated and stainless steel
    - Replaceable orifices
  - Ultra-Clean Resin
- Follow with:
  - Sampling Bags
  - Thermal Desorption Tubes
  - Gas Standards



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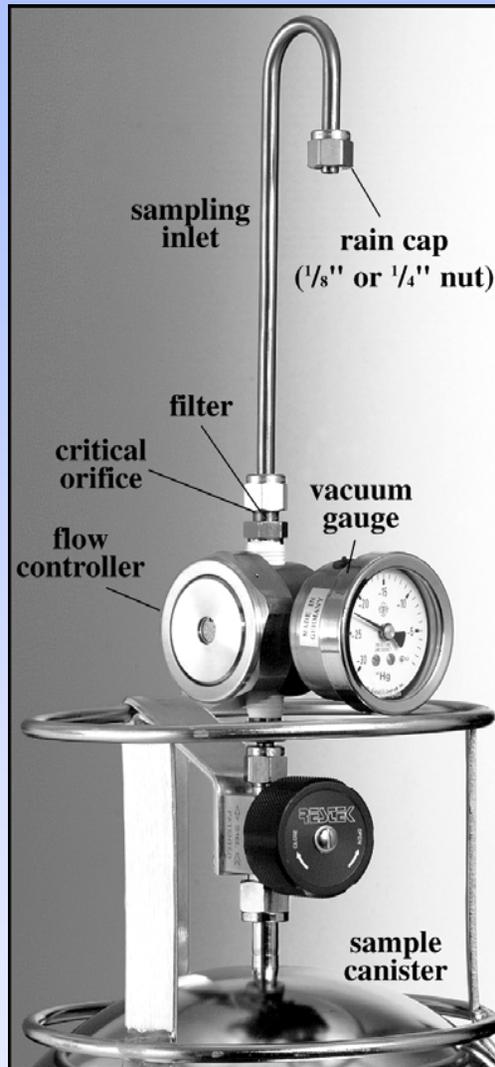


# Sample Collection Preparation (Canisters)

- Air canister heating jacket
  - Heats canister and valve
  - Mini Cans can be cleaned the same way
    - Although there are better systems on the market
  - Sampling kit
    - Heat with flow
- Humidification chamber
  - Need to use humidified nitrogen for best cleaning results



# Passive Sampling w/Canisters



- No sampling pump required
- Time-integrated sampling using flow controller
- Multiple orifices for broad range of flow rates
  - 0.5 – 350 cc/min

# Sample Collection - Canisters



- “Gold Standard” for ambient air sampling of VOCs
  - Whole air sampling
- Variety of sizes
  - 1, 3.2, 6 and 15L
- Grab & time-integrated sampling
  - No sampling pump
- Valve options now available
  - Swagelok SS4H same as Entech
- Large inventory for fast delivery



# Sample Collection – Canisters Timer

- Automated valve open/close for grab and time-integrated sampling
  - 12 timed events
  - Manual operation also available
- Long battery life – months
- Recharge with USB port on PC
- ¼" inlet/outlet compatible with any canister and flow controller



# Sample Preparation - Canisters

- Canister comes into lab under vacuum
  - Nitrogen is added to pressurize sample
  - Need to measure vacuum and pressure to determine sample volume
- Ashcroft Test Gauges



# Analysis

- GC Columns

- Rxi-1ms, 60m x 0.32mm, 3um

- Standards

- TO-14, TO-15, and Ozone Precursor Standards

- DIN EN ISO 16017
- 12 month stability
- Includes Certificate of Analysis

- PI Marked Cylinders (Scotty)

- Meets EU Requirements
- 110L size
- CGA 180



In nitrogen, 104 liters @ 1,800psi
1ppm cat. # 34400 (ea.)
100ppb cat. # 34421 (ea.)
In nitrogen, 110 liters @ 1,800psi (PI-marked Cylinder)
1ppm cat. # 34400-PI (ea.)
100ppb cat. # 34421-PI (ea.)

## cylinder design

Spectra 104L Cylinders:

Aluminum construction.

Size: 8 x 24 cm.

Volume/Pressure:

104 liters of gas

@ 1,800psi

CGA-180

outlet fitting.

Weight:

1.5 lbs./0.7 kg



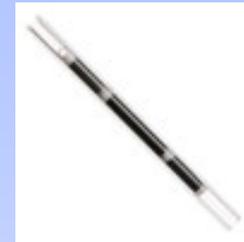
# Sample Collection – Mini Canisters

- 400cc & 1000cc
- Electro-polished & Sulfinert treated
- Both quick-connect fitting and diaphragm valve
- Uses: indoor air, personal, emergency response, soil gas sampling
- Miniature sampling kit components now available
  - Shorter sampling inlet
  - Smaller gauge



# Sample Collection Preparation (Thermal Desorption Tubes)

- Alternative technique to canisters
- SS & glass tubes compatible with Perkin Elmer and Markes instruments
- Active (requires sampling pump) and passive sampling modes
- Written in several methods
  - EPA TO-17, NIOSH 2549, ASTM D6196
  - Widely accepted technique in Europe, particularly for personal monitoring (DIN EN ISO 16017)
- Major competitors
  - Perkin Elmer, Supelco, SKC
- Pre-cleaned tubes available
  - Pricey!!! (\$585 vs \$950)
- Do not offer tube cleaning system



# Sample Collection – TDU Tubes

- Need pumps, orifice, etc.
  - We do not offer at this time
  - Looking into



# Sample Collection Preparation (Gas Sampling Bags)

- ALTEF Gas Sampling Bags

- Replacement for Tedlar bags
- SS & polypropylene septum/valve fitting
- Inert
- Sizes from 0.5 to 100 liters



- Cali-5-Bond™ Bags (New!)

- Opaque for light sensitive compounds
- 5-layer inert construction
- Rugged, reusable
- Hose barb valve with separate septum port
- Commonly used for emission sampling & petroleum industry
- Sizes from 0.5 to 44 Liters



# Semivolatile Air Sampling (XAD2 & PUFs)

- XAD2
  - Precleaned material
- XAD2 – Not cleaned
  - 1kg quantities
- Polyurathane Foam (PUFs)
  - Do not offer equipment for re-use
  - XAD2 – Soxhlet extractor
  - Tubes – heat with flow



# Other Air Sampling Products

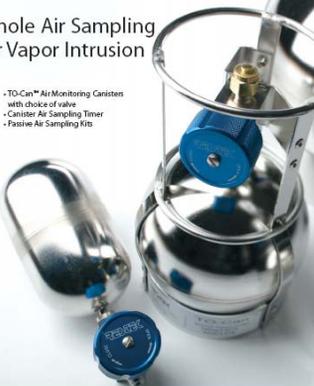
- Large volume syringes
- Custom standards
- Carrying boxes/cases
  - Use for shipping



# Marketing Literature

Whole Air Sampling for Vapor Intrusion

- TO-Can™ Air Monitoring Canisters with choice of valve
- Canister Air Sampling Timer
- Passive Air Sampling Kits



**RESTEK** Chromatography Products  
www.restek.com

Thermal Desorption Unit (TDU) Tubes  
Variety of sorbents to collect a wide range of VOCs



**RESTEK** Chromatography Products  
www.restek.com

Air Monitoring Products

- All sampling canisters
- Thermal desorption units/tubes
- Gas sampling bags
- Sample cylinders
- Gas standards
- Accessories
- Applications

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**RESTEK AIRMAIL** VOL. 6 2008

Meet the Air Team!

**How to Prolong Canister Life**  
What reduces canister performance and is costly? Leakage is the most common reason for canister failure, but contamination and damage to the fused silica lining can also lead to canister leakage problems. Here are some ways to protect your investment:

- Prevent leaks**
  - Use proper handling to avoid these 3 leak-prone areas of leaks.
    - Pinch connections**  
You can prevent part of the stress exerting the valve by always using a 2 or 3 jaw pliers. Be sure to use the correct size pliers on your canister fitting equipment. Also, prevent the valve from being fully closed that applies most stress. The 20% recommended metal-to-metal seating when possible prevents the greatest stresses, but avoid too much contact to produce damage that other valve type.
    - Canister fused fittings**  
Avoid gasket fused fittings by using a plug cap to prevent over-tightening of compressed fittings. Starting with 1/2 turn past finger tight is another rule of thumb to prevent over-tightening. Likewise compressed fittings on stainless steel, high-pressure fittings, such as desiccant or addition, to minimize thermal damage. Canister threads may also cause a poor connection to measurement probe, resulting in inaccurate measurement and misleading conclusion that canister leakage exists.
    - Over-tightened valve**  
Canister valves are designed to close securely with hand tightening only. Over-tightening a valve device with a wrench can damage the valve seat when the seal is made.
- Reduce contamination**
  - Storage high concentration (ppm) cans and trace concentration (ppb) cans.** Use dual-use canisters, or gas sampling bags, for your level sampling. Since it is somewhat difficult to remove impurities from your sampling to a level suitable for trace sampling.
  - Check the entire sampling train** as you would the can to minimize introduction of contaminants into the can. Maintain temperature at 10°C on the probe and 10°C on the can's valve (the controls).
  - High temperature (>100°C) humidified air (trace cleaning) provides the most effective way to remove contamination from desiccant-lined cans (TO-Can™ or SDBM-A canisters), but can damage fused silica lined cans.** See #1 below for proper cleaning of fused silica lined cans.
- Avoid damage to fused silica lined cans**
  - In cases of 100% humidified trace concentration (ppb) during your canister to avoid damage to the fused silica lining. Cleaning residue of 20°C canisters using hand held air can be 10°C and 12°C have the most critical moisture of water compounds, when compressed and nitrogen on the same conditions. This causes the damage to the fused silica surface, using water that may affect the recovery of volatile or polar compounds. Strong acids and bases may also result in damage to the fused silica surface.

**Upcoming Events**

**Pittcon, Booth # 2411**  
March 2-7  
New Orleans, LA

**ASTM Meeting**  
Committee D22 on Air Quality  
April 6-9  
Anaheim, CA

**ASTM Intertek Round Workshop**  
New ASTM Standard on Vapor Intrusion  
April 8  
Anaheim, CA

**American Industrial Hygiene Association 8th Exhibition**  
May 21-24  
Memphis, TN

**AIH & WSHA Management Association Annual Conference**  
June 24-27  
Portland, OR

**New Products!**  
Thermal Desorption Tubes, p. 2  
Canister Air Sampling Timer, p. 4  
GIS-S-Band Gas Sampling Bags, p. 4

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  - Gas Standards
- Diverse product line offering many options for VOC and SVOC sampling



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