Keep your GC Column Alive: Tips and Tricks for Extending Column Lifetime

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Application Engineers
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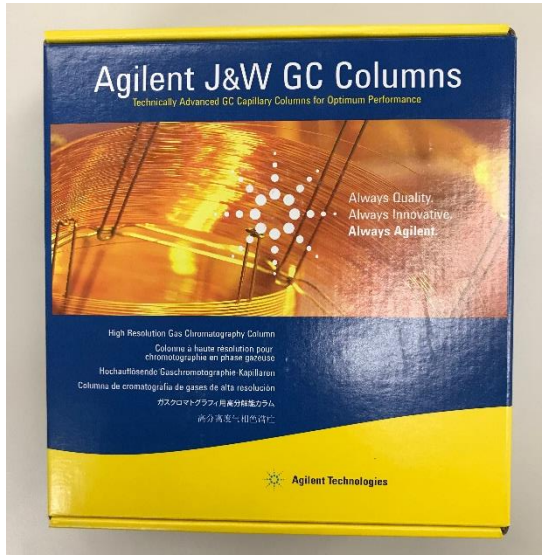
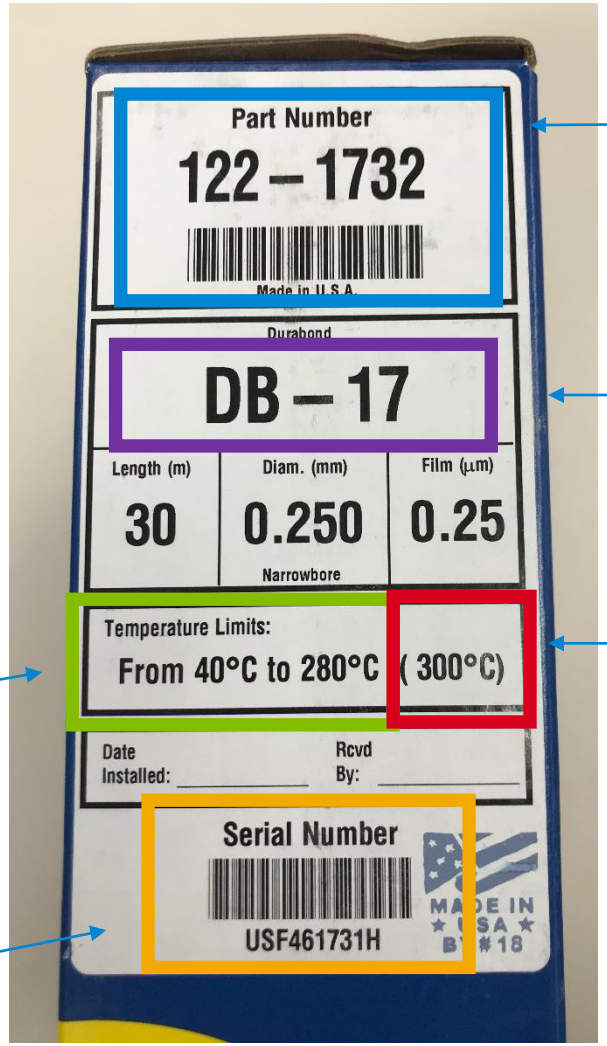
Agenda

- Review – get to know your column
- Signs and symptoms of an unhealthy column
- How to know if your column is healthy?
- What can you do to bring your column back to life?
- Extending the lifetime of your column – sample preparation options
- Extending the lifetime of your column – backflush techniques

The “Unboxing” of the GC Column





Important for identification and re-ordering

Column stationary phase

Programmed temperature limit (<10 min)

Isothermal temperature limits

Unique to each column (identification)

What's Inside?

Column tag contains useful information

Column plug holds column ends together and protects against contamination. To put the column in storage, use this plug again or a piece of septa over the ends of the column.

Column Performance Summary

Catalog: 19091S-433UI
Serial: F542252H

Stationary Phase: HP-5MS UI
Description: 30m x 0.250mm x 0.25µm
Temperature Limits: -60°C to 325°C (350°C Pgm)

Performance Results

Theoretical Plates/Meter:

n-DECANE	3208

Retention Index:

n-PROPYLBENZENE	953.110
1-HEPTANOL	967.660

Resolution:

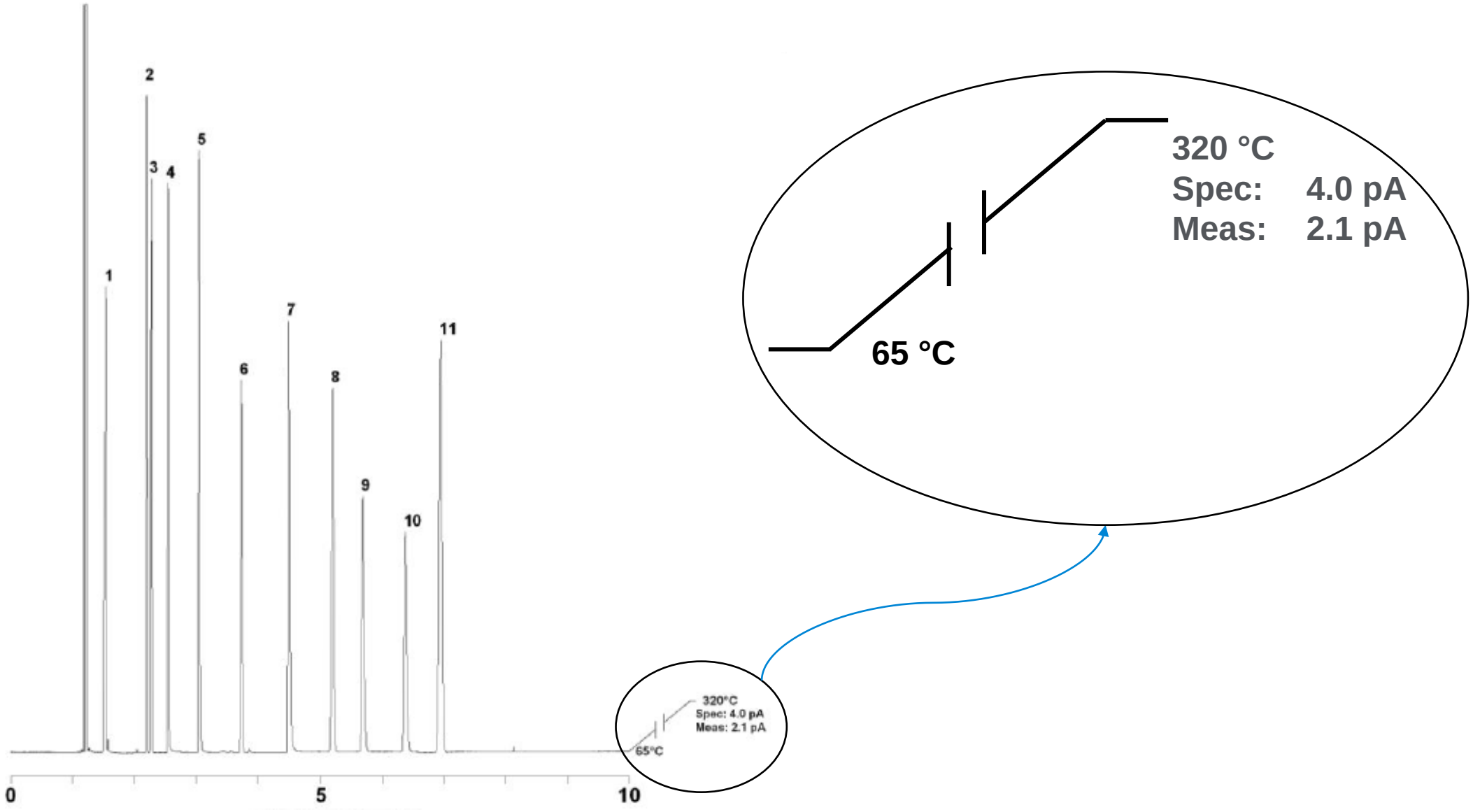
1-OCTENE, n-OCTANE	2.97

Compound Identification	Retent. Time	Part. Ratio	1/2-Width
1. PROPIONIC ACID	1.543	0.30	0.027
2. 1-OCTENE	2.203	0.86	0.015
3. n-OCTANE	2.282	0.92	0.016
4. 1,3-PROPANEDIOL	2.552	1.15	0.020
5. 4-METHYLPYRIDINE	3.051	1.57	0.021
6. n-NONANE	3.738	2.15	0.027
7. TRIMETHYLPHOSPHATE	4.482	2.78	0.033
8. n-PROPYLBENZENE	5.193	3.38	0.038
9. 1-HEPTANOL	5.682	3.79	0.041
10. 3-OCTANONE	6.368	4.37	0.047
11. n-DECANE	6.940	4.85	0.053

Test Conditions

Inlet: **Split** (250°C) **Detector:** FID (325°C)
Carrier Gas: Hydrogen **Flow:** 42.1 cm/sec (1.2 ml/min)
Holdup Compound: Pentane (1.187-min)
Temperature Program: Isothermal at 65°C

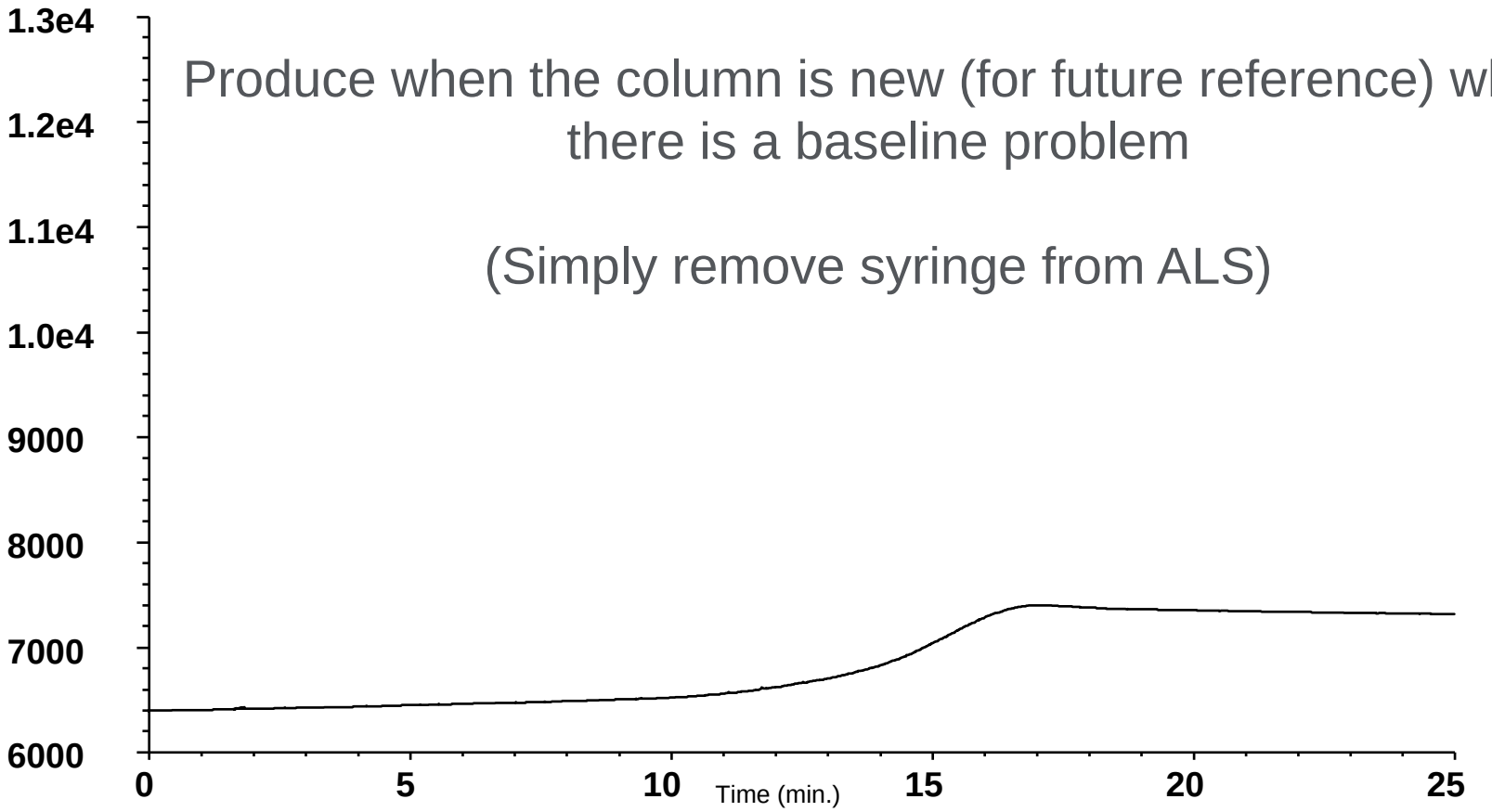
Chromatographic Performance



Test Mixture Components

Compounds	Purpose
Hydrocarbons	Efficiency
	Retention
FAMEs, PAHs	Retention
Alcohols	Activity
Acids	Acidic character
Bases	Basic character

Generating a Bleed Profile



*Agilent J&W DB-1 30 m x 0.32 mm id, 0.25 µm
Temperature program // 40 °C, hold 1 min // 20 °C/min to 320 °C, hold 10 min

Common Peak Shape Issues

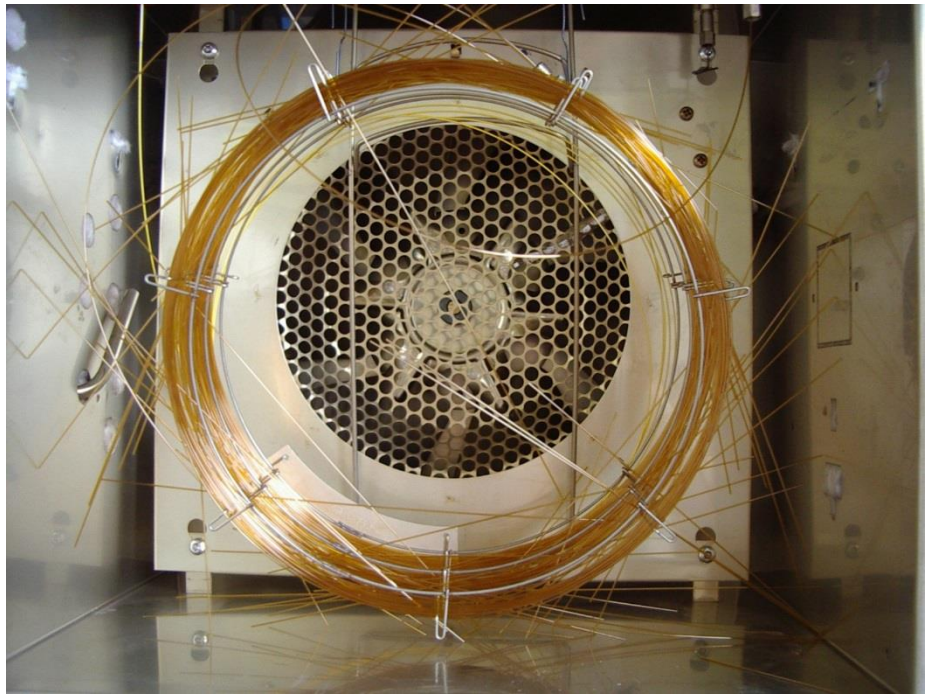
- **Peak tailing** – flow path or activity
- **Bonus peaks** – in sample or backflash (carry over)
- **Split peaks** – injector problems, mixed solvent
- **No peaks** – wasn’t introduced, wasn’t detected
- **Response changes** – activity, injector discrimination, detector problem
- **Peak fronting** – overload or solubility mismatch, injector problems
- **Shifting retention** – leaks, column aging, contamination or damage
- **Loss of resolution** – separation decreasing, peak broadening
- **Baseline disturbances** – column bleed, contamination, electronics
- **Noisy or spiking baseline** – electronics or contaminated detector
- **Quantitation problems** – activity, injector, or detector problems

Common Causes of Column Performance Degradation

- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O_2 damage)
- Chemical damage by samples
- Contamination

Physical Damage to the Polyimide Coating

- The smaller the tubing diameter, the more flexible it is
- Avoid scratches and abrasions
- Immediate breakage does not always occur upon physical damage



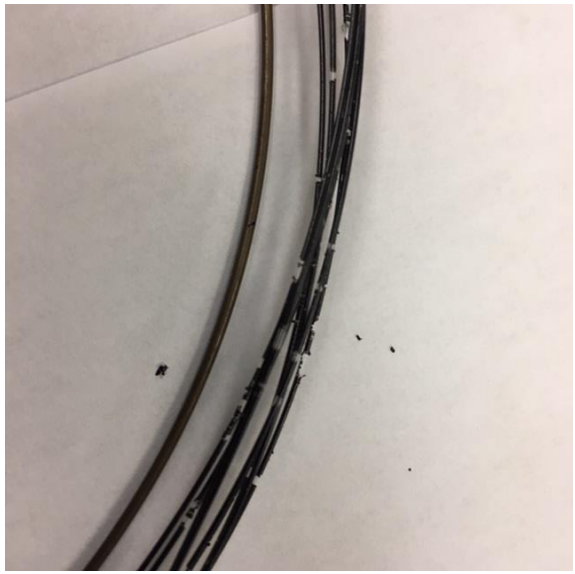
Thermal Damage

Degradation of the stationary phase is increased at higher temperatures

- Rapid degradation of the stationary phase (breakage along the polymer backbone) caused by excessively high temperatures

  Isothermal limit = indefinite time
  Programmed limit = 5–10 minutes

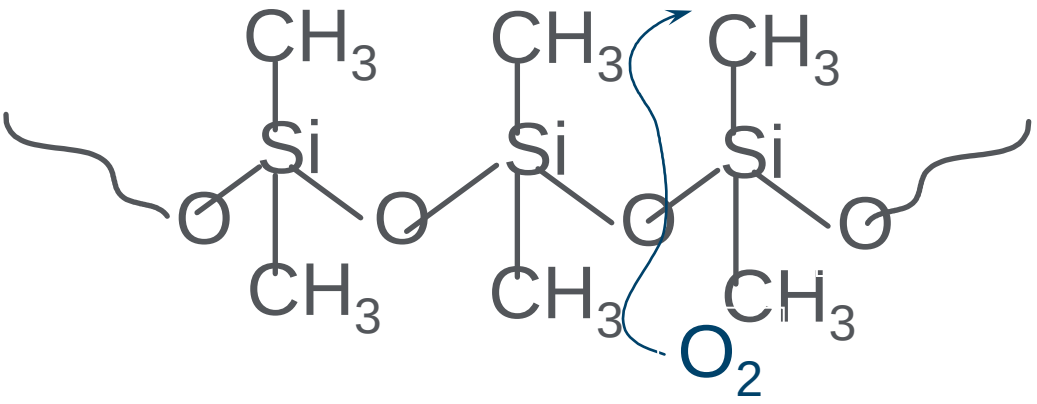
- Temporary "column failure" below lower temperature limit
- If this happens:
  - Disconnect column from detector
  - “Bake out” overnight at isothermal limit
  - Remove 10–15 cm from column end



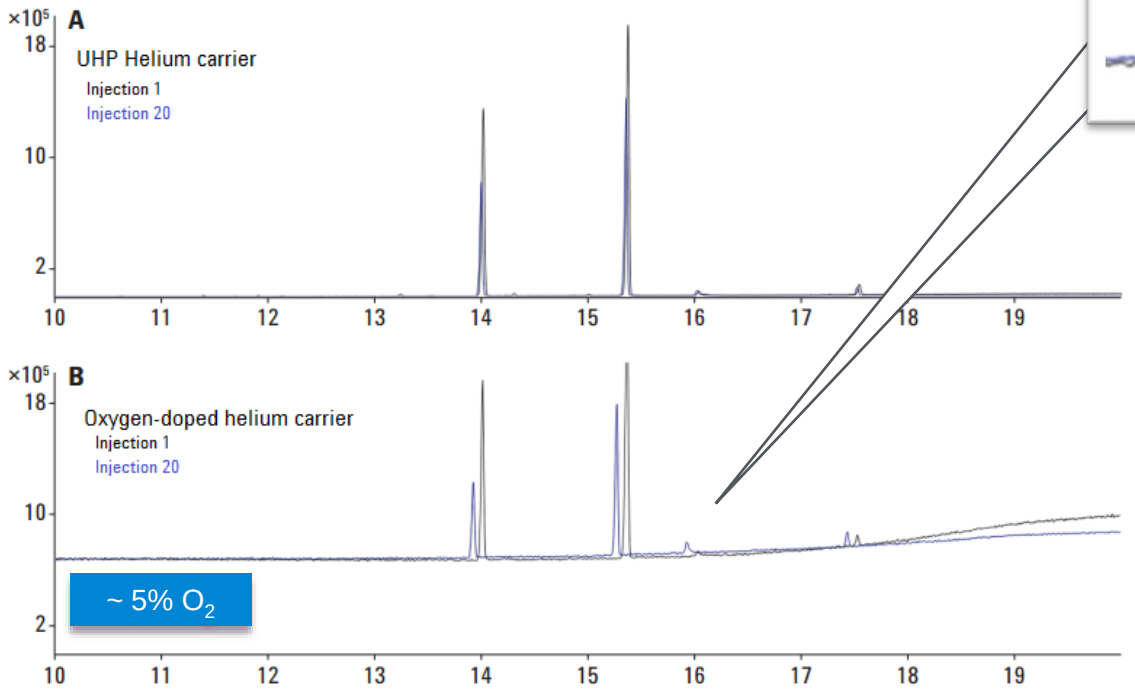
Column continuously exposed to temperatures above its temperature limit

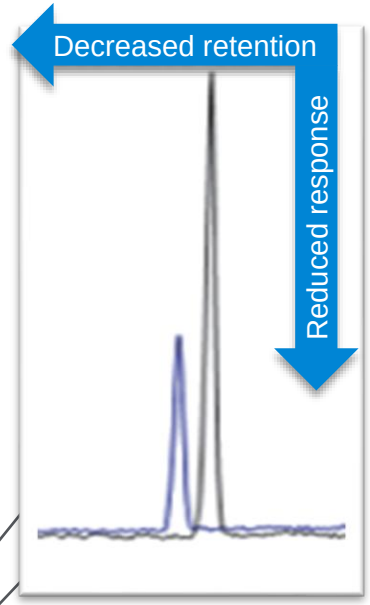
Oxidation (O_2 Damage)

Oxygen in the carrier gas rapidly degrades the stationary phase. The damage is accelerated at higher temperatures. Damage along the polymer backbone is irreversible. (Premature filament failure/excessive source maintenance.)



Dimethylpolysiloxane

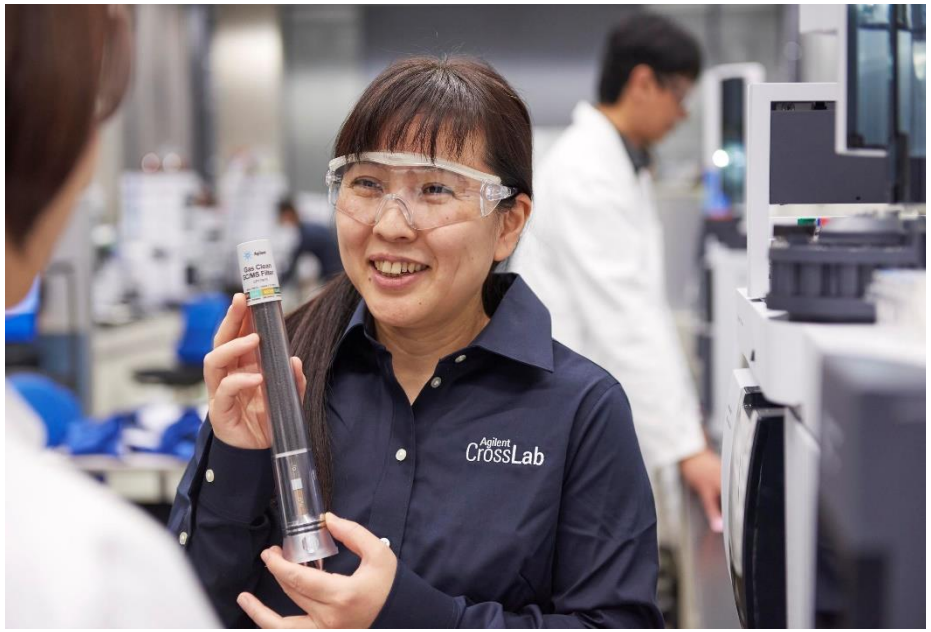




Higher bleed

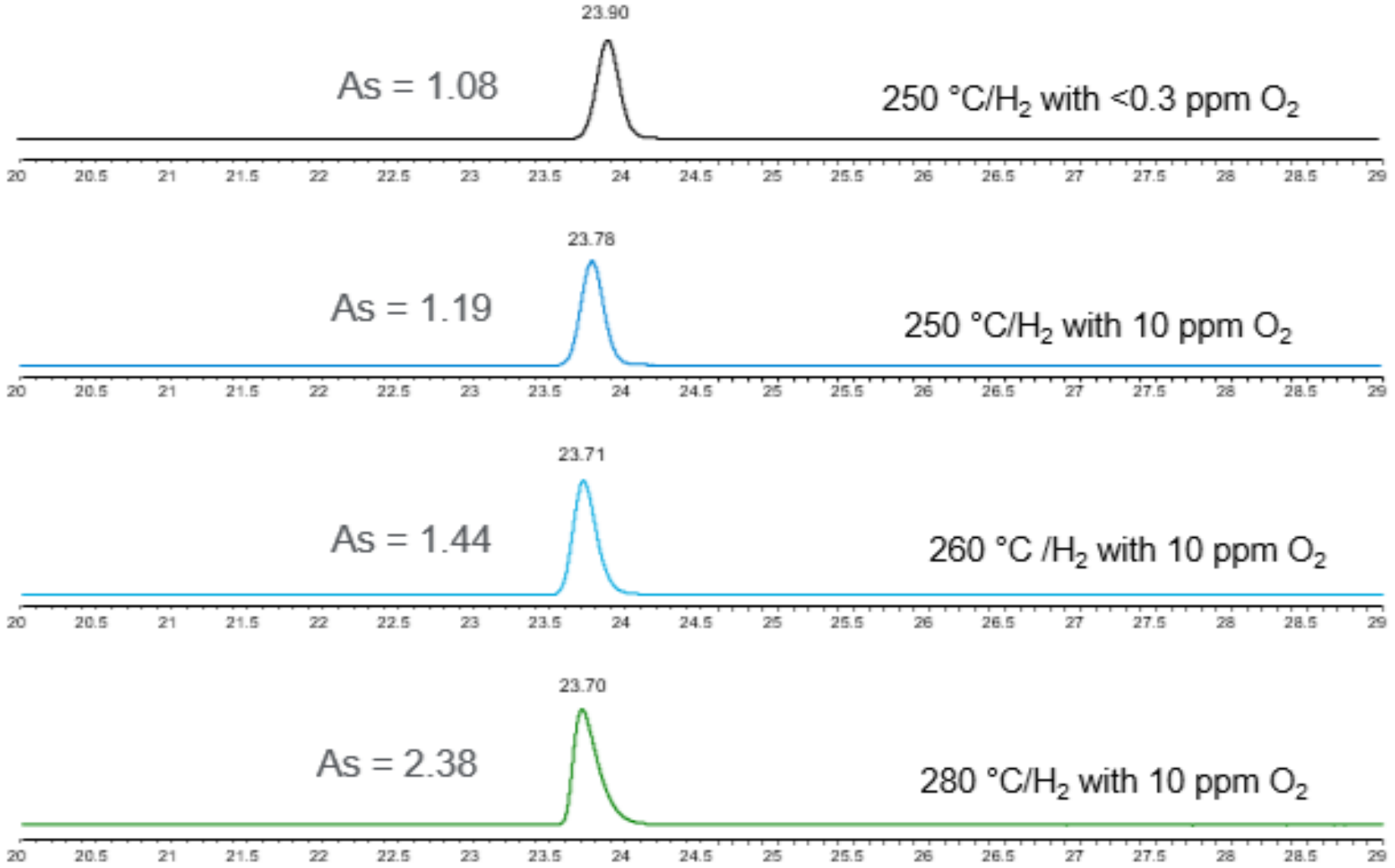
How to Prevent Column Damage by Oxygen

- High-quality carrier gas (four 9s or greater)
- Leak free injector and carrier lines
  - Change septa
  - Maintain gas regulator fittings
- Appropriate impurity traps



Efficient, fast, easy

Effect of Oxygen on Peak Shape of 2-ethylhexanoic Acid



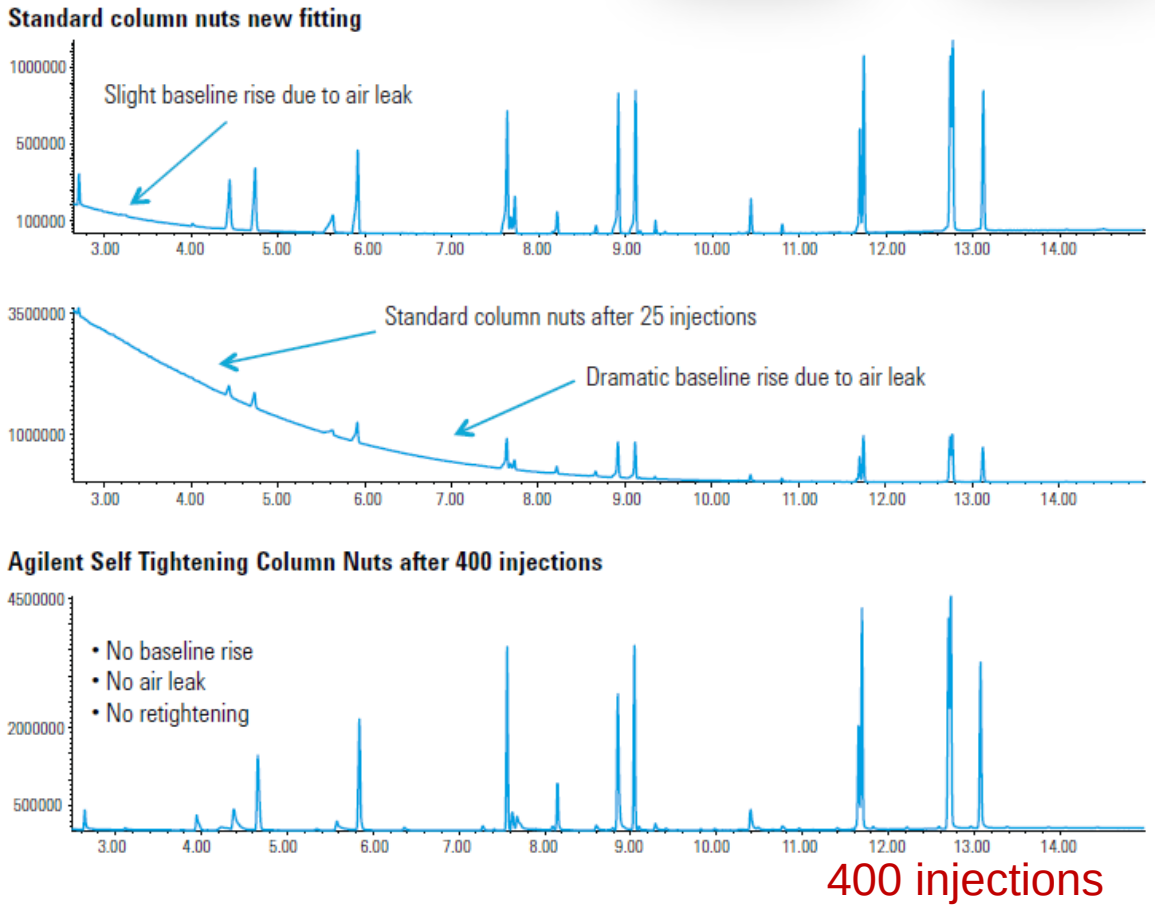
Self Tightening Nuts: No Leaks, No Downtime, No Frustration

- Spring-driven piston continuously presses against ferrule
- Automatically retightens when ferrule shrinks
- Wing design for finger tightening
- No tools needed
- Works only with graphite/Vespel ferrules

Part Number	Description
G3440-81013	Column Nut, Collared Self-Tightening MSD
G3440-81011	Column nut, Collared Self Tightening Inlet/Detect
G3440-81012	Collar for Self Tigthening Nut

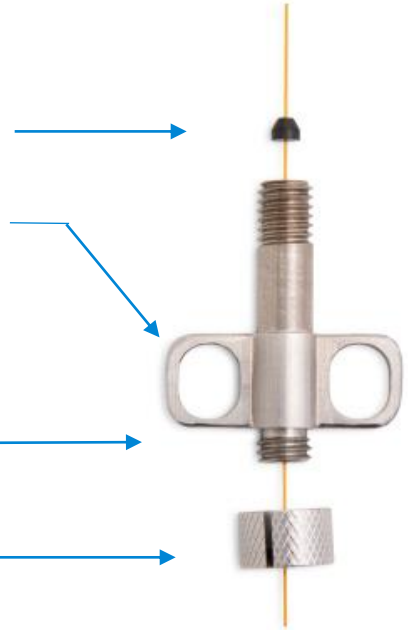
https://www.agilent.com/en/video/gc-supplies-innovation

https://www.agilent.com/en/video/stcn-inlet-detector
https://www.agilent.com/en/video/stcn-mass-spec



New Agilent Standard Winged Nut and Depth Guide

- Compatible with Agilent/HP style compact ferrules, including graphite ferrules
- Winged fastener design for easy engagement and tool free install
- Hollow-body design with low thermal mass mitigates thermal lag during temperature cycling within the GC oven
- Removable locking-collar with soft-PTFE insert to secure column placement during install without damaging the analytical column



Inlet / Detector
G3440-81018

MSD
G3440-81019

Agilent Capillary Column Depth Guide – G3440-88000

- Easy-to-use template provides critical capillary column installation for the most popular Agilent GC-configurations
  - SSL, MMI, purge-packed inlets
  - FID, TCD, NPD detectors
  - EI MSD source
- Compatible with the Agilent Self Tightening and winged column nuts

The Cost of Leaks

- Cost of gases
- Contamination from exposure
- Reduced consumable lifetime
- Reduced productivity from downtime
- Detector noise and elevated baselines
- Time in troubleshooting

It is critical that every customer checks for leaks. They should have the best tool for the job.

Check valves, fittings, and traps for leaks after every piece of maintenance, and after thermal cycling, as these can loosen some types of fittings.

Agilent CrossLab CS (Cartridge System)

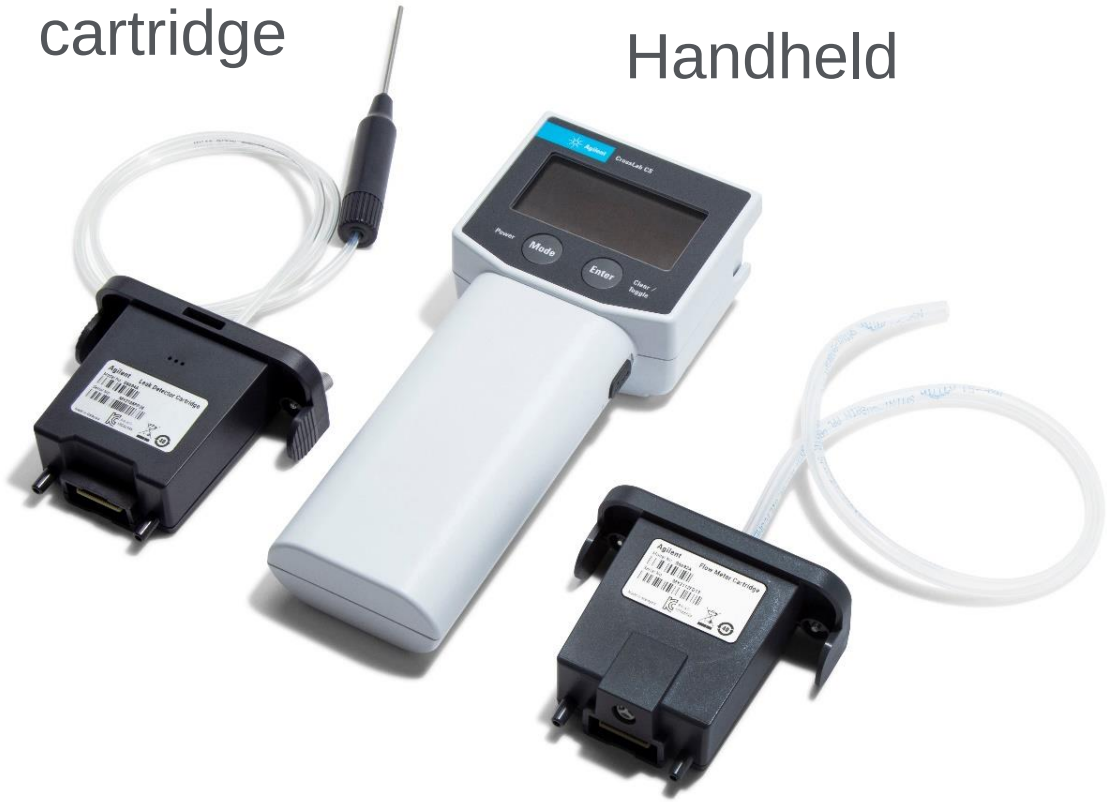
No peaks from leaks

Features:

- Exchangeable cartridge with ADM Flow Meter
- Automatic notification of probe filter replacement
- Ergonomic and robust design
- Universal 3AA or USB power
- USB connects to web interface for added functionality and firmware updates
- Easy to view OLED screen
- Kickstand

Leak detector cartridge

Handheld



ADM Flow Meter cartridge

More Information

- **Agilent.com CrossLab CS Leak Detector**

www.agilent.com/chem/gas-leak-detector

- **Agilent.com – ADM Flow Meter**

https://www.agilent.com/en/product/gas-purification-gas-management/gas-management/adm-flow-meter

- **Installation manual**

Agilent CrossLab CS
Electronic Leak Detector manual
Part number: G6693-90000

The installation manual is available on Agilent.com.

- **Innovation minute video**

https://www.agilent.com/en/video/crosslab-cs-innovation-minute

- **Technical overview**

Agilent CrossLab Cartridge System
(CS) Electronic Leak Detector
Publication number: 5994-4262EN

The technical overview is available on Agilent.com.

- **Brochure**

GC Troubleshooting in
the Palm of Your Hand
Publication number: 5994-3607EN

The brochure is available on Agilent.com.

- **Flyer**

Is a Leak Causing Your
Inaccurate Results?
Publication number: 5994-4202EN

The flyer is available on Agilent.com.

Ordering Guide

One year warranty

- G6693A – CrossLab CS Electronic Leak Detector
- G6694A – Electronic Leak Detector Cartridge
- G6699A - CrossLab CS Bundle: ADM Flow Meter and Electronic Leak Detector
  - The bundle will include one handheld, two cartridges, and a **free** carrying case.

- G6694-60005 – Replacement probe filter
- G6691-40500– Carrying case

Existing products:

- G6691A – CrossLab CS ADM Flow Meter
- G6692A – ADM Flow Meter cartridge*
  - Note that the ADM Flow Meter cartridge is ordered annually for calibration.
    The Electronic Leak Detector does not need to be recalibrated

Chemical Damage

Bonded and crosslinked columns have excellent chemical resistance except for inorganic acids and bases.

HCl NH_3 KOH $NaOH$

H_2SO_4 H_3PO_4 HF

Chemical damage will be evident by excessive bleed, lack of inertness, or loss of resolution/retention.

Chemical Damage

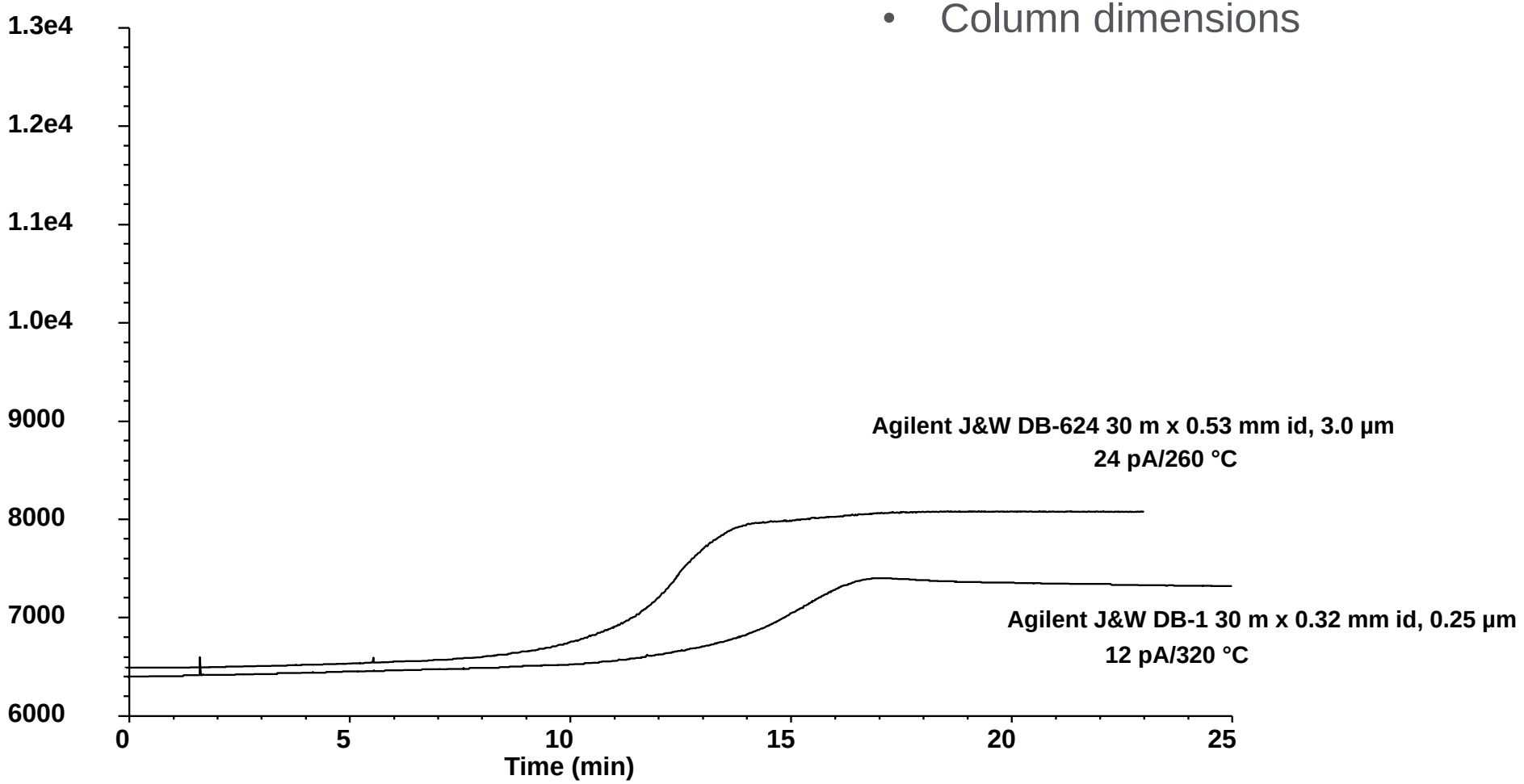
What to do if it happens

- Remove 0.5 to 1 m from the front of the column
- Severe cases may require removal of up to 5 m

What is Normal Column Bleed?

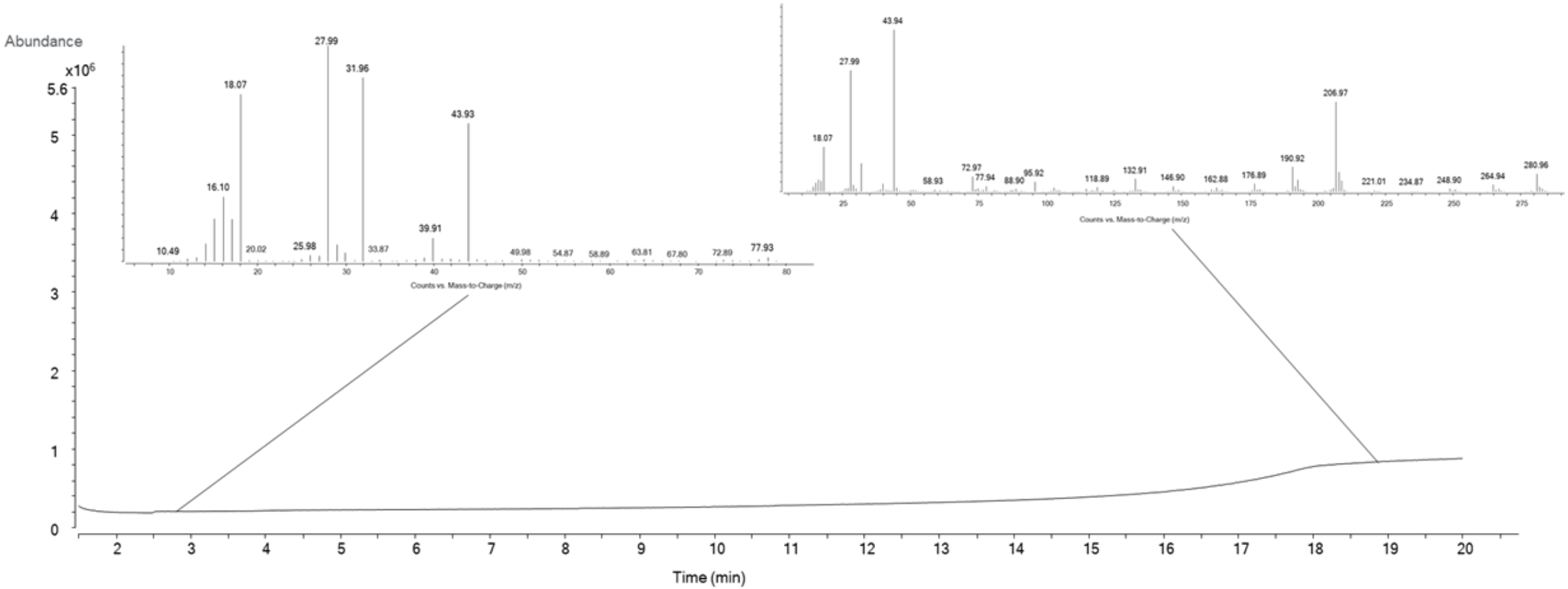
Normal background signal generated by the elution of normal degradation products of the column stationary phase. Column bleed is influenced by:

- Phase type
- Temperature
- Column dimensions



Mass Spectrum of Phenylmethylpolysiloxane Column Bleed

Normal background (HP-5ms UI)



What is a Bleed Problem?

An abnormal elevated baseline at high temperature

It is **not**

- A high baseline at low temperature
- Wandering or drifting baseline at any temperature
- Discrete peaks

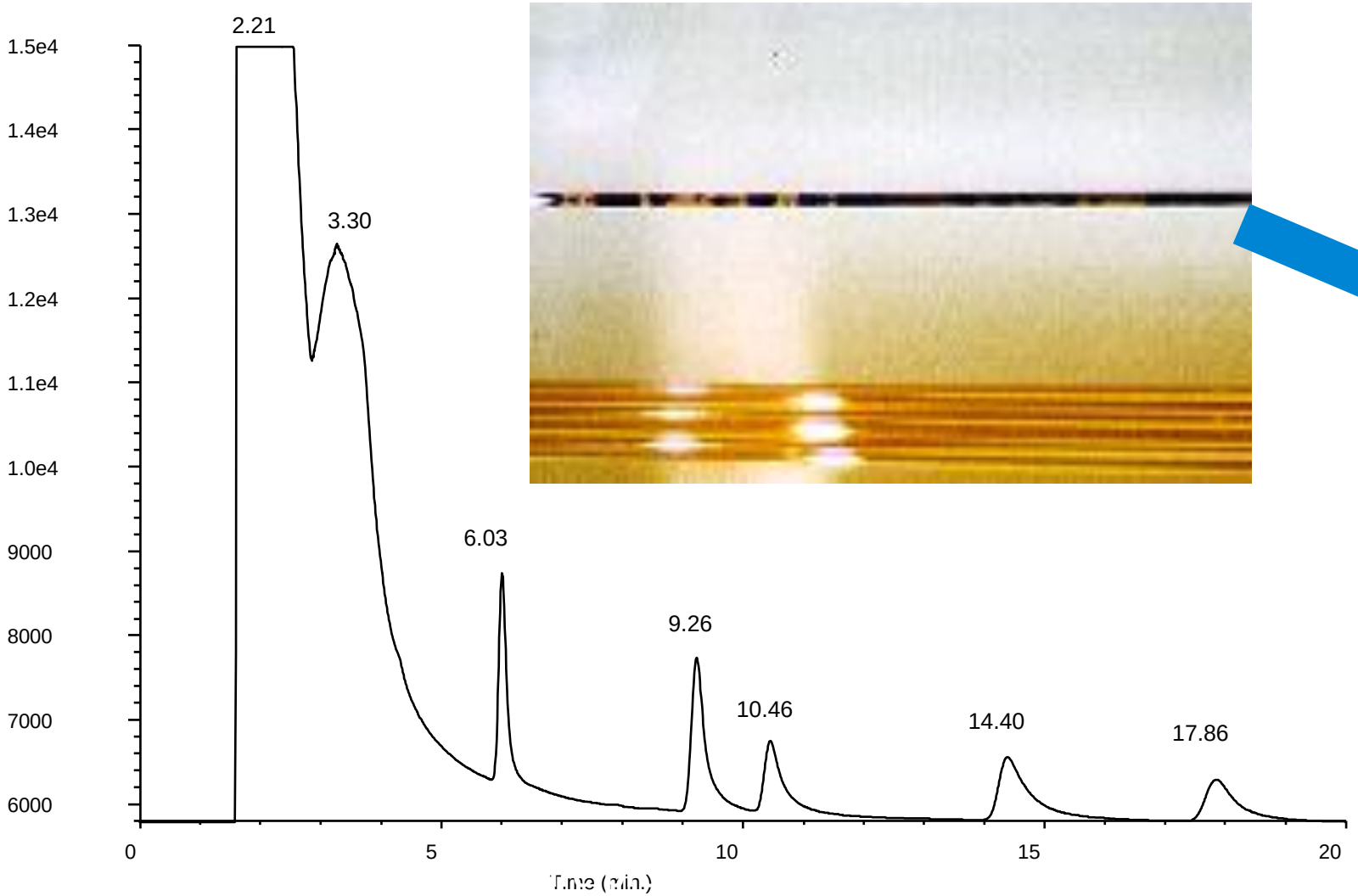
Column Contamination and Symptoms

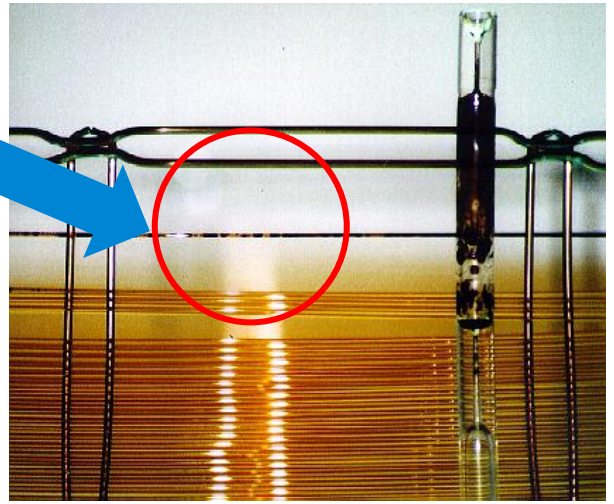
- Fouling of GC and column by contaminants
- Mimics nearly every chromatographic problem

- Poor peak shape
- Loss of separation (resolution)
- Changes in retention
- Reduced peak size
- Baseline disturbances (semivolatiles only)

Example of Column Contamination

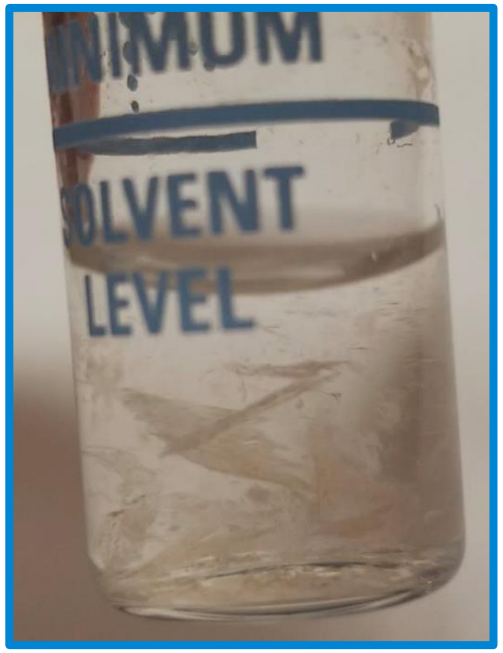
Agilent J&W DB-624 QC test mix*
After 75 injections of oily sample





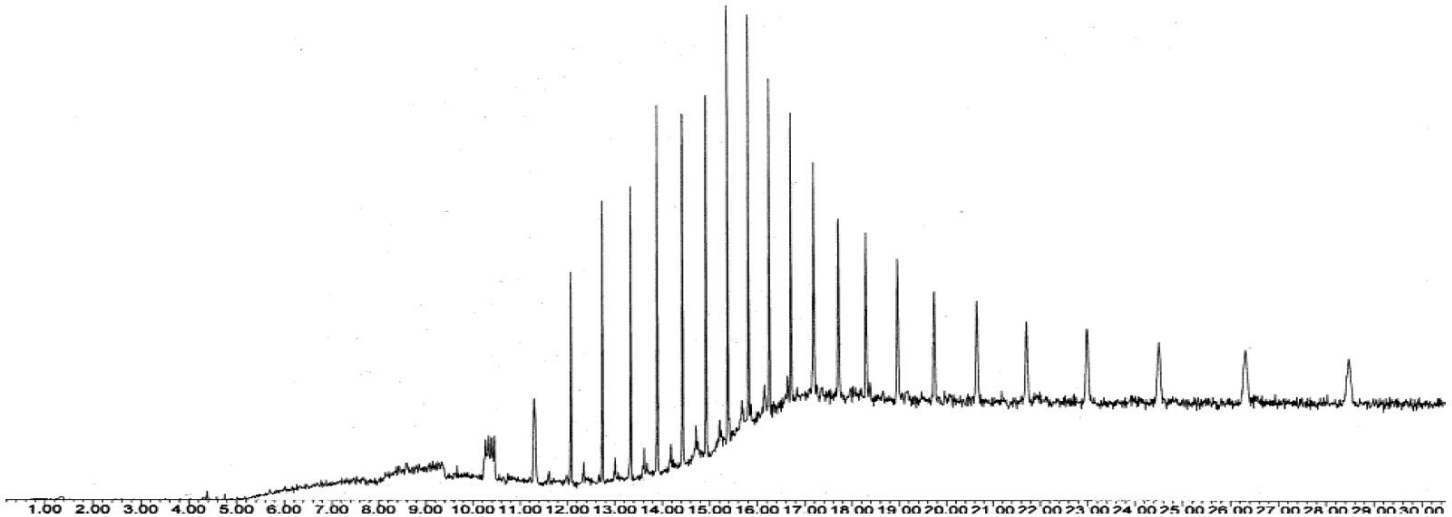
Sources of Contamination

- Septum and ferrule particles
- Gas and trap impurities
- Unknown sources (vials, syringes, and so on)



Contaminated wash solvent

Sample vial septum bleed profile:



Typical Samples That Contain a Large Amount of Residues

Biological
Soils
Foods
Wastewater
Sludges

All samples contain residues (even standards)

Types of Residues

Nonvolatile residues

- Any portion of the sample that does not elute from the column or remains in the injector.

Semivolatile residues

- Any portion of the sample that elutes from the column after the current chromatographic run.

Semivolatile Contamination

What to do if it happens

- “Bake out” the column
  - Limit to 1 to 2 hours
  - Longer times may polymerize some contamination and reduce column life
- Solvent rinse the column

Nonvolatile Contamination

What to do if it happens

- Do not “bake out” the column
- Front end maintenance
  - Clean or change the injector liner
  - Clean the injector
  - Cut off 0.5 to 1 m of the front of the column
- Turn the column around
- Cut the column in half

Methods to Minimize Nonvolatile Residue Problems

- Sample cleanup
- Packed injection port liners
- Guard columns
- Backflush techniques

Dilute and Shoot

Advantages

- Fast and easy
- High throughput

GC inlet liner

GC inlet seal

Limitations

- Interferences are not removed
- Analyte concentration is reduced
- Instrument and column contamination
- Matrix interferences – ion suppression or poor peak shapes

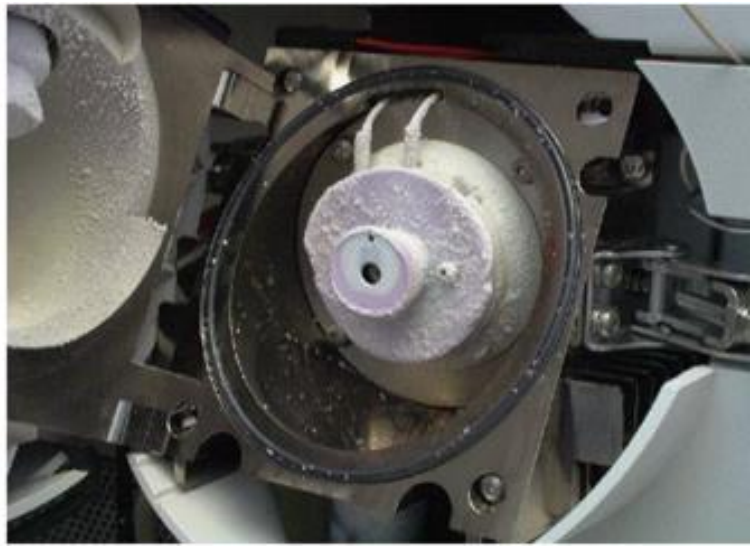


Image of salt buildup on an ESI-LC/MS inlet from unremoved salts.

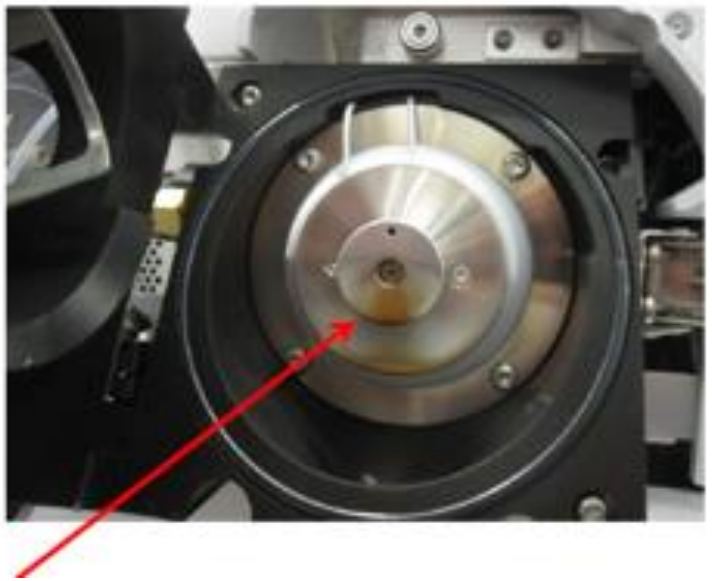


Image shows the build up on the ESI-MS inlet after 3000X urine dilute and shoot injections.

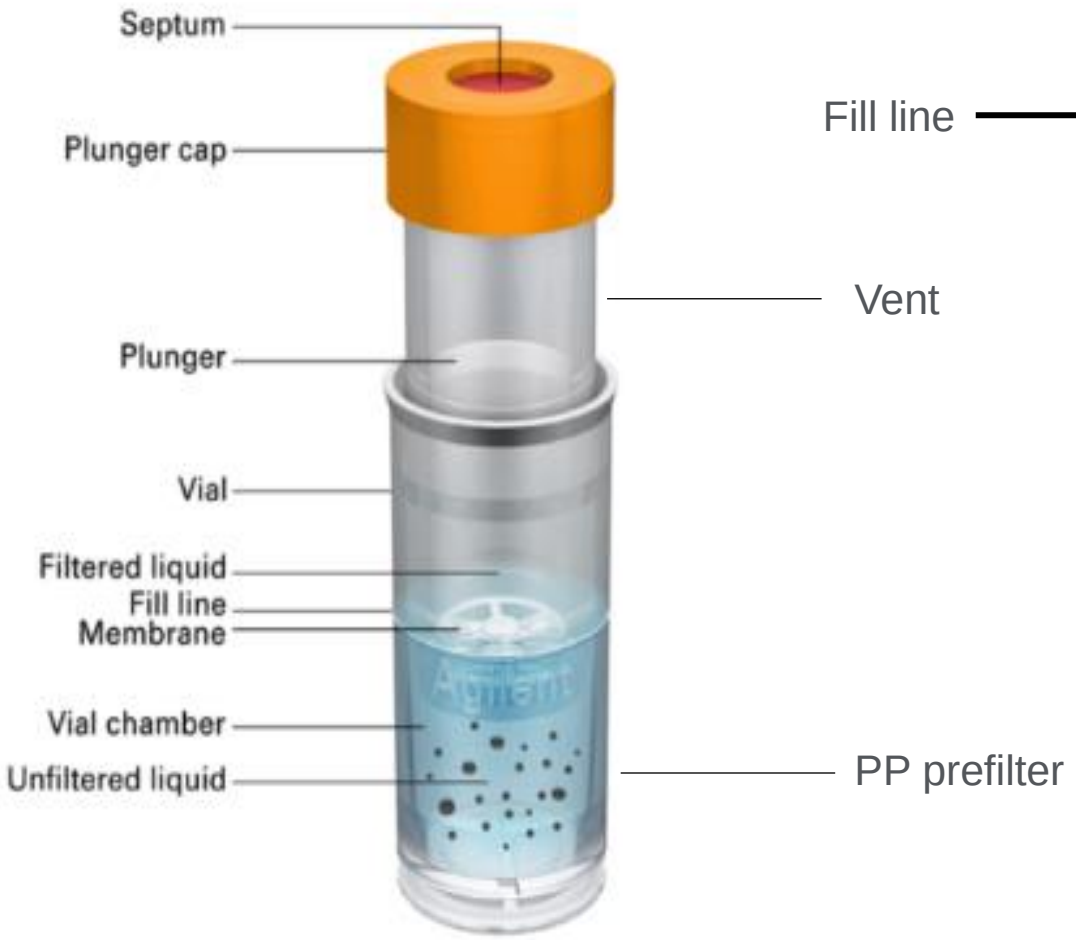
Physical Filtration

Captiva premium syringe filters

- Certified to be free of UV-detectable extractables on HPLC. PES and glass fiber also certified for LC/MS.
- Color-coded boxes for easy identification
- Comprehensive portfolio to meet all customers' needs

Premium Syringe Filters						
Membrane	Diameter/Pore Size					
	4 mm		15 mm		25 mm (28 mm)	
	0.2 µm	0.45 µm	0.2 µm	0.45 µm	0.2 µm	0.45 µm
PTFE	♦	♦	♦	♦	♦	♦
Nylon			♦	♦	♦	♦
PES	♦	♦	♦	♦	♦	♦
Regenerated cellulose	♦	♦	♦	♦	♦	♦
Cellulose acetate					♦	♦
Glass microfiber			♦		♦	
Depth filters: glass/PTFE			♦	♦	♦	♦
Depth filters: glass/nylon			♦	♦	♦	♦

Physical Filtration – Captiva Filter Vials



Part Number	Description
5191-5933	PTFE filter vial, 0.45 µm, 100/pk
5191-5934	PTFE filter vial, 0.20 µm, 100/pk
5191-5935	Nylon filter vial, 0.45 µm, 100/pk
5191-5936	Nylon filter vial, 0.20 µm, 100/pk
5191-5939	RC filter vial, 0.45 µm, 100/pk
5191-5940	RC filter vial, 0.20 µm, 100/pk
5191-5941	PES filter vial, 0.45 µm, 100/pk
5191-5942	PES filter vial, 0.20 µm, 100/pk
5191-5943	Vial closure tool

Preslit options available as well

Agilent.com/chem/filtervials
Filter vials user guide: 5994-0814EN

Chemical Filtration

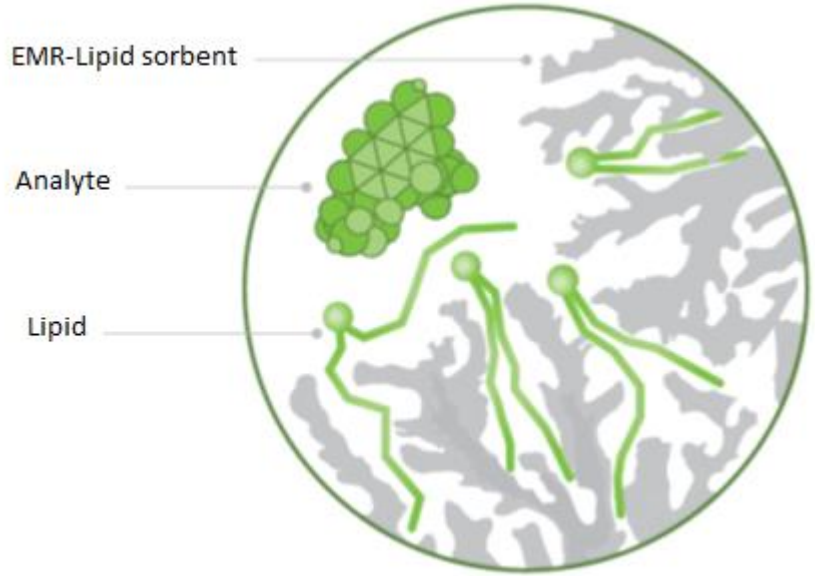
Captiva EMR-Lipid

- One of the newest Agilent sample cleanup products with a 2-in-1 benefit of removing lipids and fats selectively and efficiently.
- It reduces ion suppression, increases analyte sensitivity, improves peak shape, and extends the lifetime of your analytical column.
- Simple pass-through format, 96-well plate, 1 mL, 3 mL, and 6 mL cartridges
- Solvent-retention frit in 1 mL cartridge/96-well plate for in-well protein precipitation
- Unique chemistry and filtration ensures protein and lipid removal
- Depth filtration design allows for smooth elution
- Received the Analytical Scientist Innovation Award (TASIA) of 2017

Captiva EMR-Lipid Sorbent Technology

EMR-Lipid sorbent technology effectively traps lipids through two mechanisms:

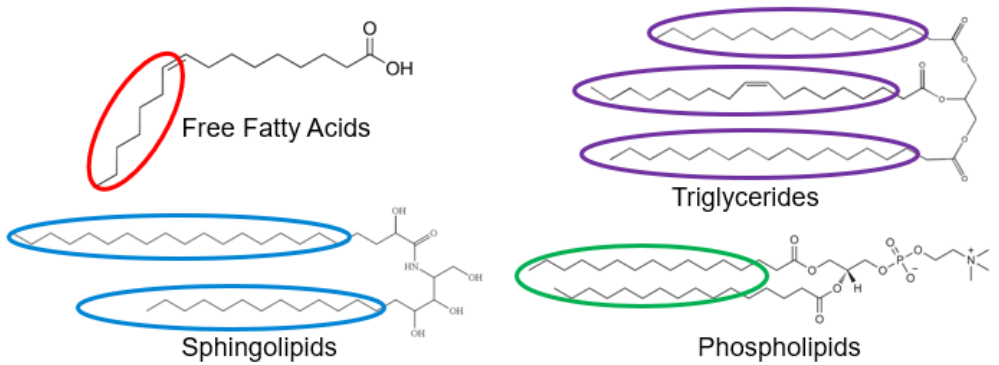
- Size exclusion – Unbranched hydrocarbon chains (lipids) enter the sorbent; bulky analytes do not
- Sorbent chemistry – Lipid chains that enter the sorbent are trapped by hydrophobic interactions



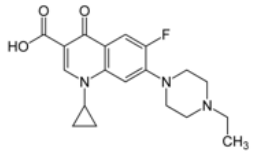
Captiva EMR-Lipid

Selective removal of lipids

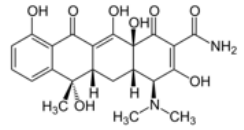
Removes lipids



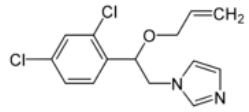
Does not remove target analytes



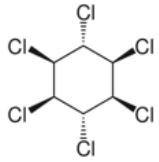
Fluoroquinolones



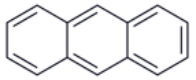
Tetracyclines



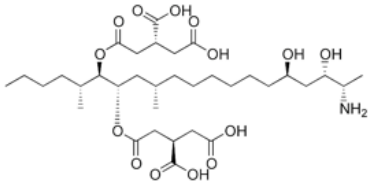
Imidazole pesticides



Organochlorine Pesticides



PAHs

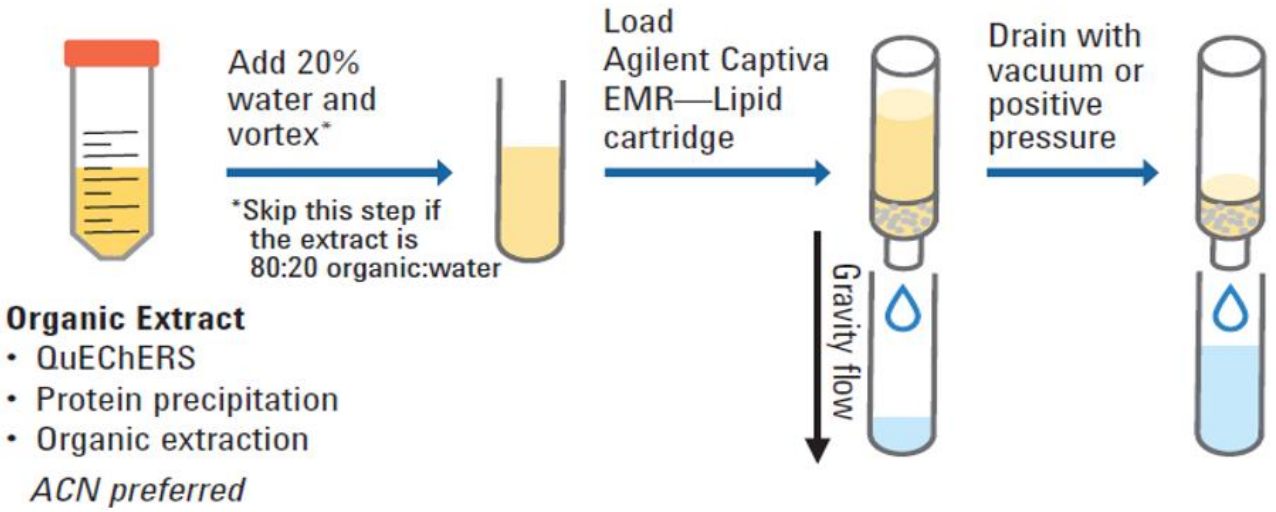


Fumonisin B2

Captiva EMR-Lipid

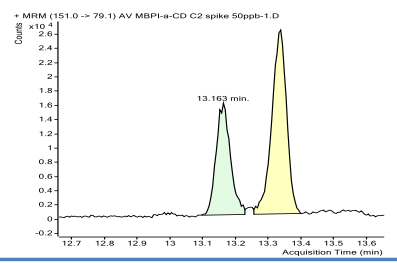
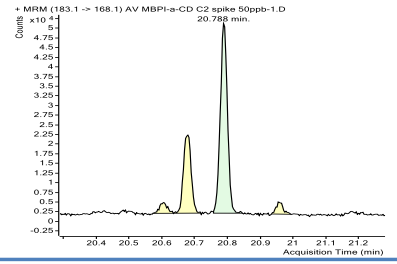
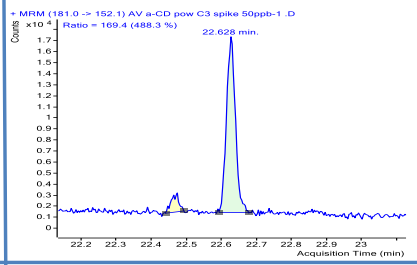
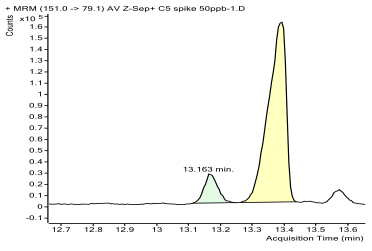
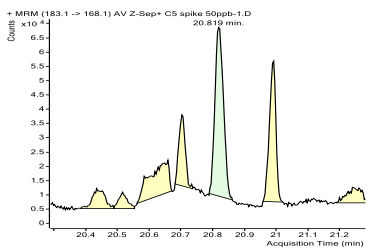
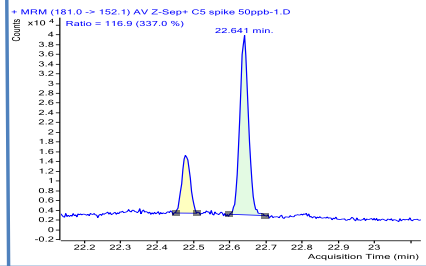
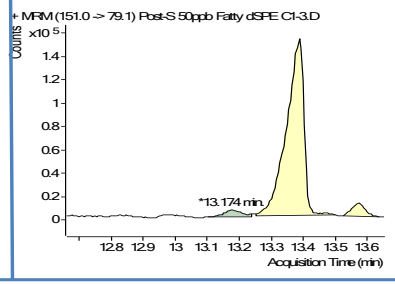
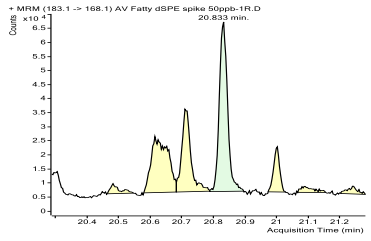
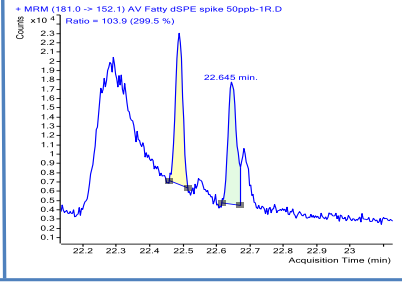
General protocol for food and food products using 3 mL and 6 mL cartridges

Operating instructions



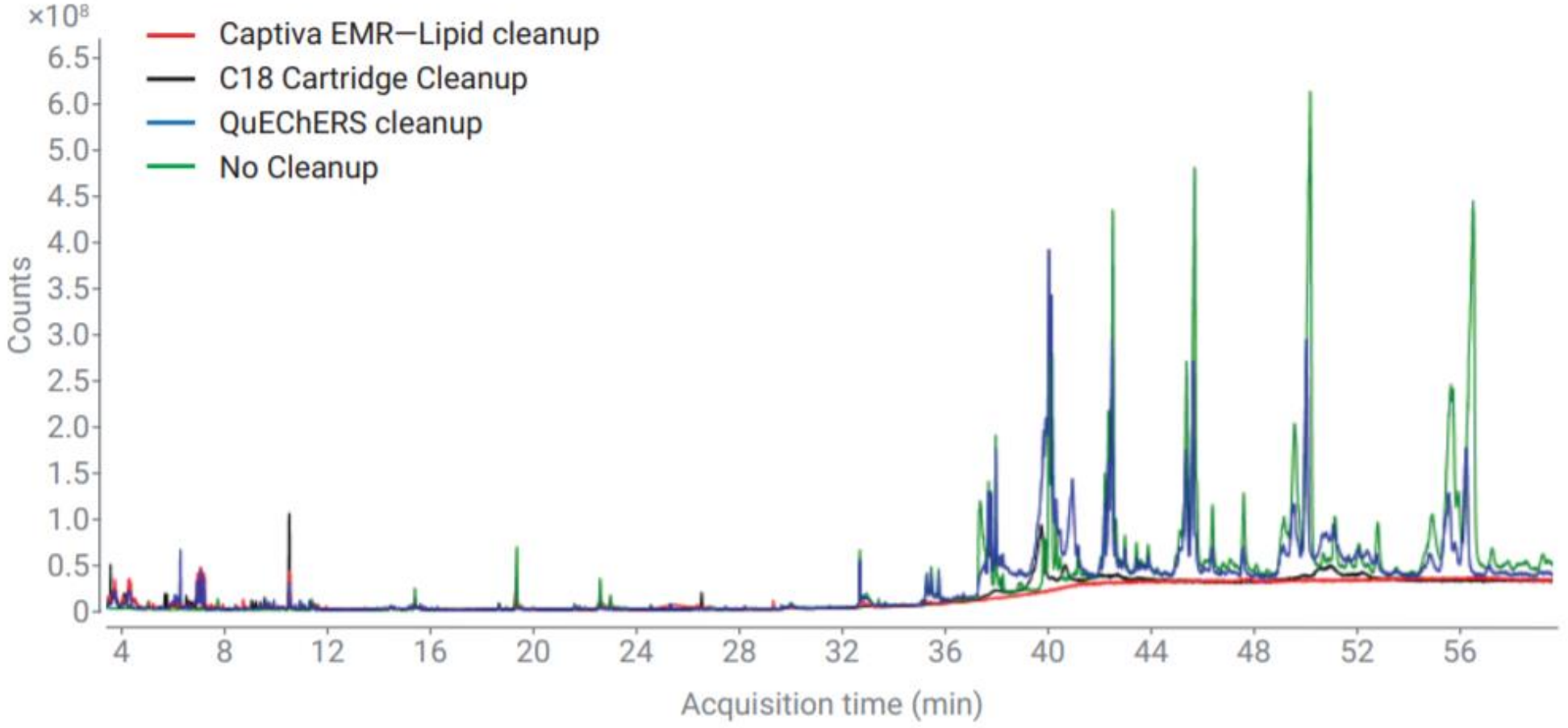
[Captiva EMR-Lipid method guide for 3 mL and 6 mL cartridges]

A Cleanup Step Improves Analytes, S/N Ratio, and Integration Accuracy on GC/MS(/MS)

Pesticide	Captan	Permethrin	Deltamethrin
EMR-Lipid cleanup			
Zirconia sorbent cleanup			
C18/PSA cleanup			

5994-0405EN

Analysis of Multiclass Multiresidue Pesticides in Milk Using Agilent Captiva EMR-Lipid with LC/MS/MS and GC/MS/MS (5994-2038EN)



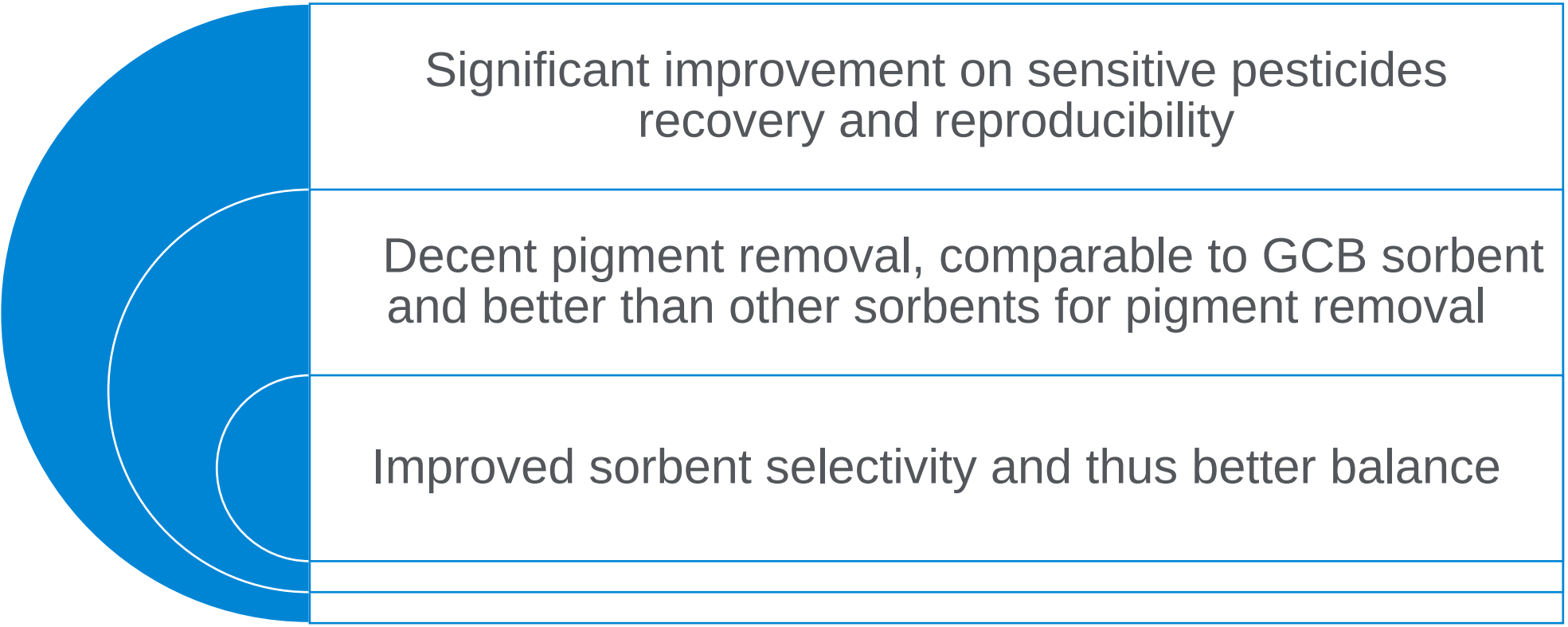
Carbon Material Used in Food Analysis

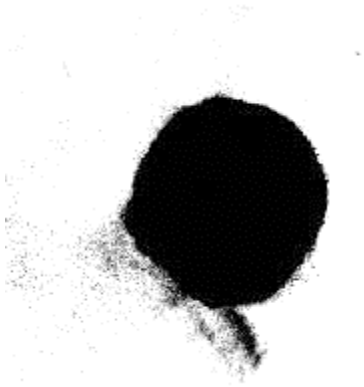
- The structure of carbon material, including graphitized carbon black (GCB), coconut carbon, and activated carbon, favors the retention of pigment components.
- Pigments are one of major matrix interferences in plant-origin food matrices
- Offers efficient pigment removal, but can also cause unwanted targets loss, such as planar compounds

Color	Pigments
Green	Chlorophyll Lutein
Red, blue, purple, black	Anthocyanidins Anthocyanins
Orange, yellow	Carotenoids Xanthophylls

Carbon S Sorbent

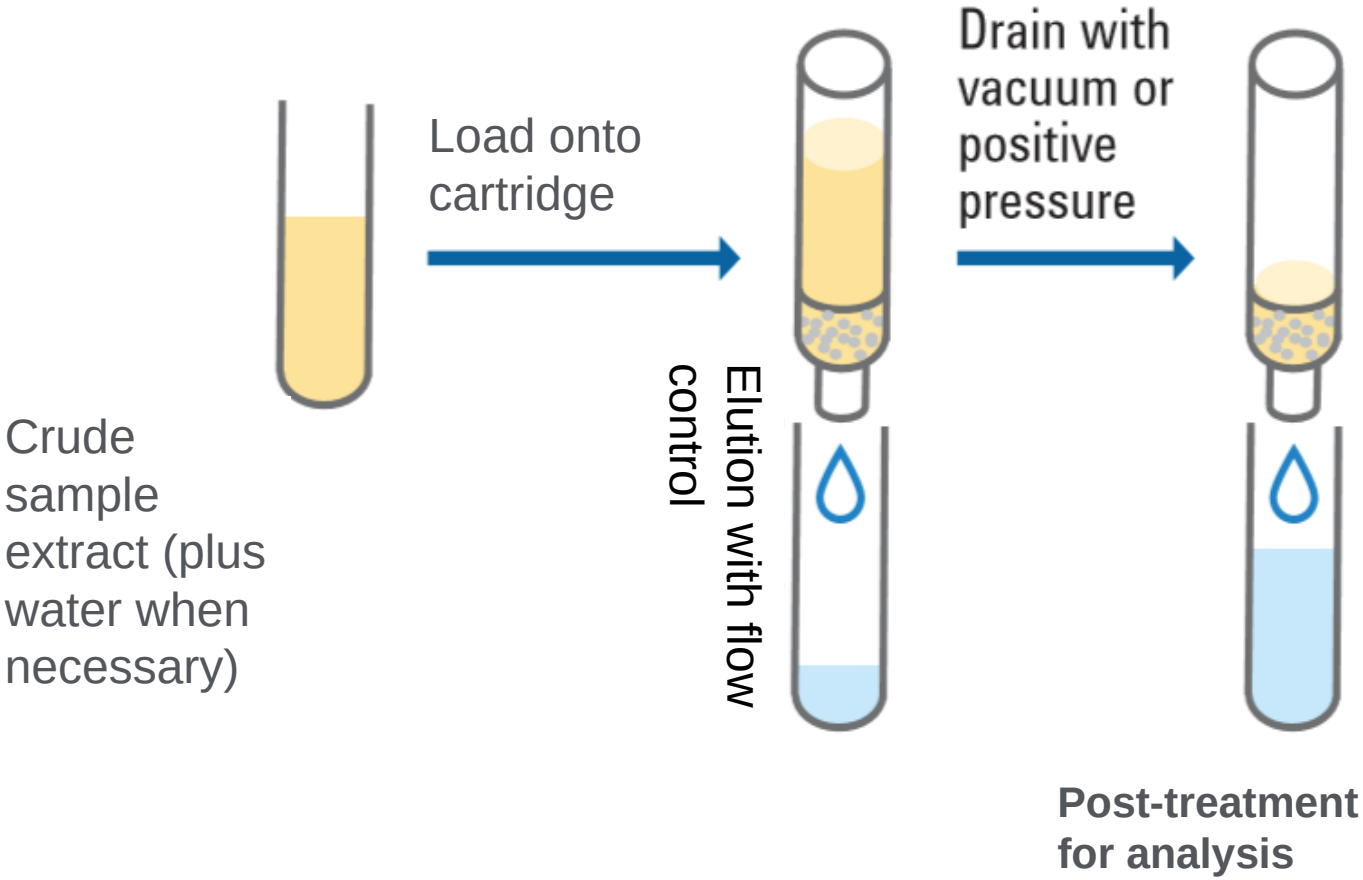
Agilent Carbon S sorbent is an *advanced hybrid carbon material* with **optimized carbon content and pore structure**





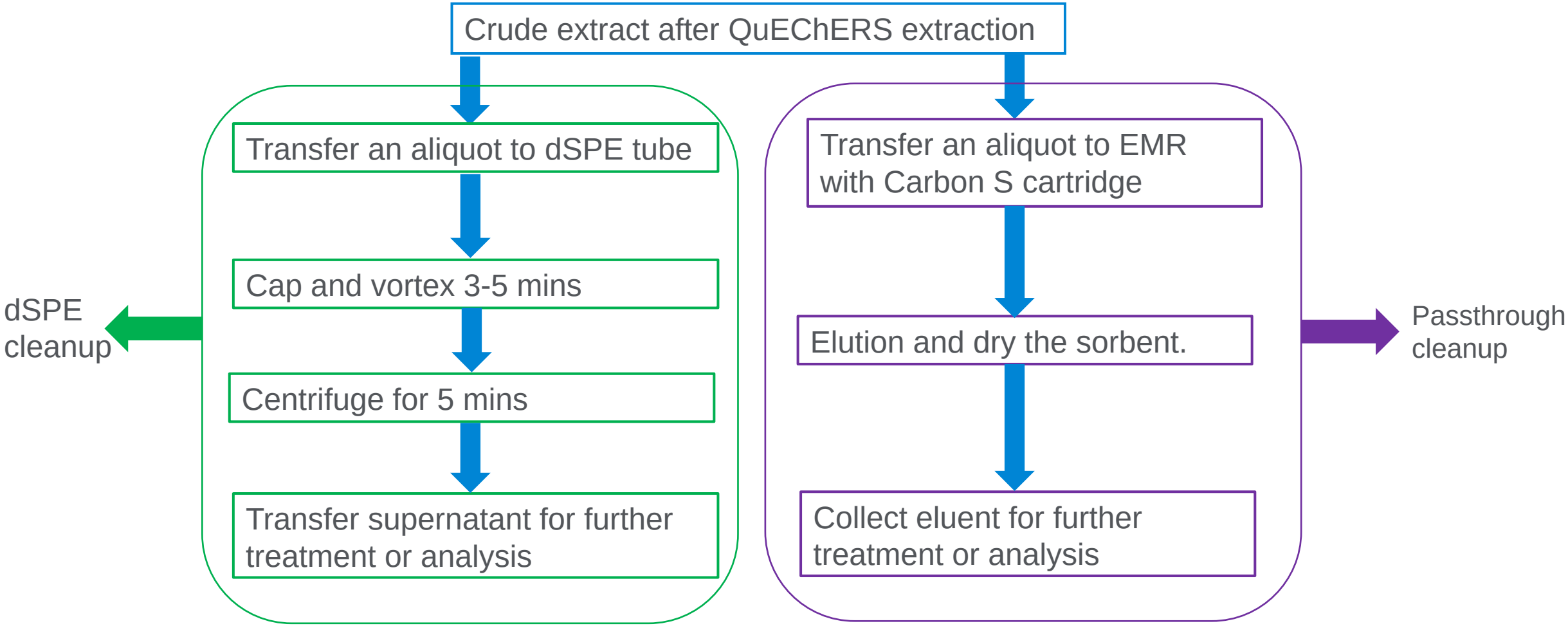
Carbon S sorbent

Chemical Filtration – Captiva EMR with Carbon S



- Sorbent interaction is targeted to unwanted matrix interferences
- Limited interactions with targets to prevent analytes loss
- Basic methodology
  - Load the sample crude extract to cartridge
  - Elution with flow control, either on gravity or low-level external force
  - Collect eluent for direct analysis or further post-treatment

Captiva EMR with Carbon S Passthrough Cleanup vs dSPE Cleanup



Passthrough cleanup saves 15 to 30% of processing time. The more samples to process, the more time can be saved.

Captiva EMR Passthrough Selection Guide

Captiva EMR-Lipid

High Lipid/Fats/Oils
- Meat, dairy, oils, eggs

Captiva EMR-HCF

High Chlorophyll Fresh
- Spinach, arugula, chard

Captiva EMR-GPF

General Pigmented Fresh
- Berries, peppers, broccoli

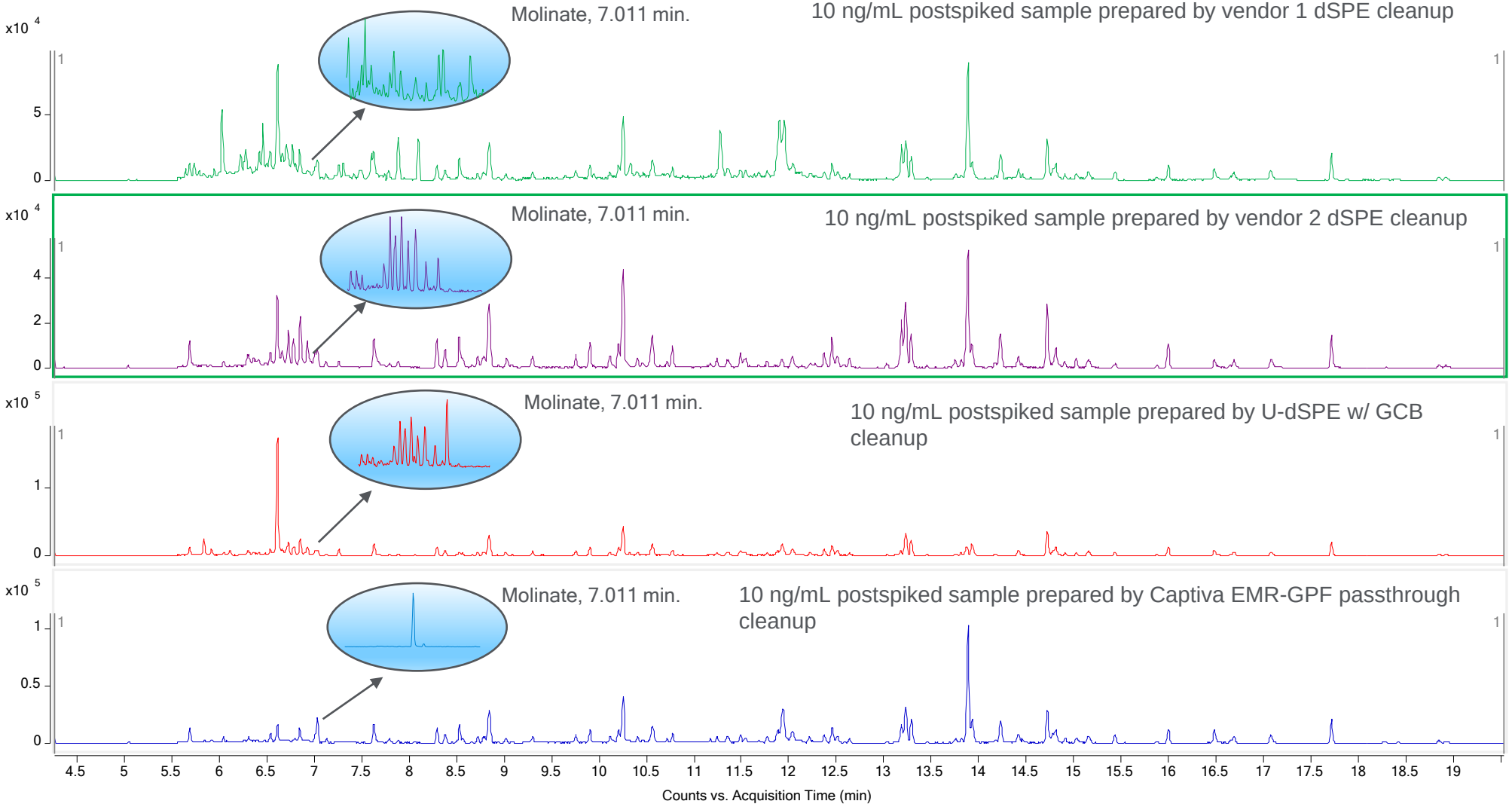
Captiva EMR-GPD

General Pigmented Dry
- Spices, seasoning, herbal medicine

Captiva EMR-LPD

Low Pigmented Dry
- Nuts, tobacco, light pigmented spices

Captiva EMR – Cleaner Matrix Background on GC/MS/MS



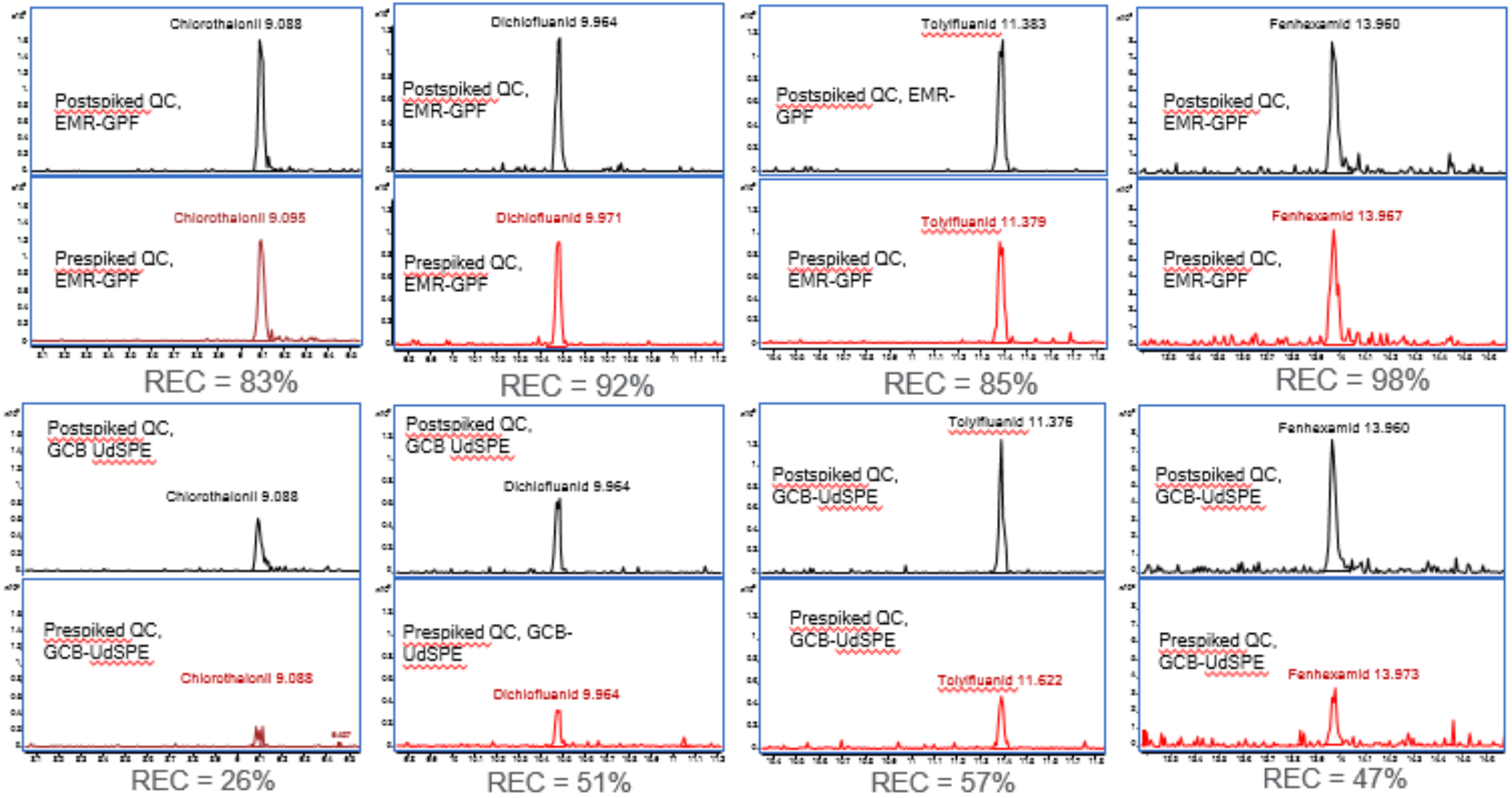
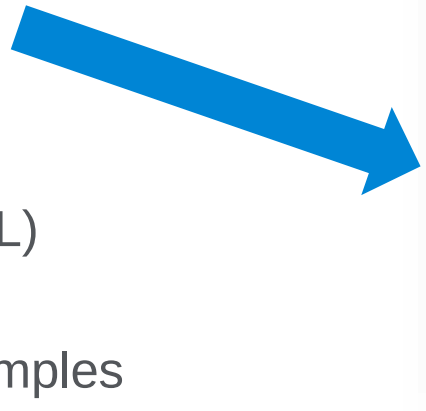
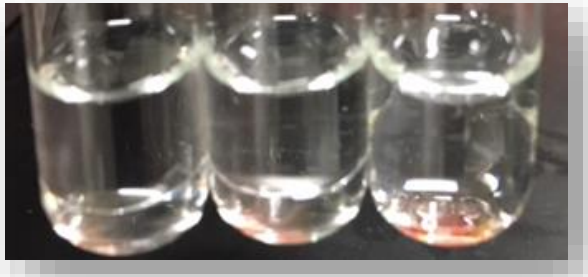


Figure 6. Chromatographic comparison of sensitive targets for samples prepared using different cleanup method.

Drawbacks of Liquid-Liquid Extraction

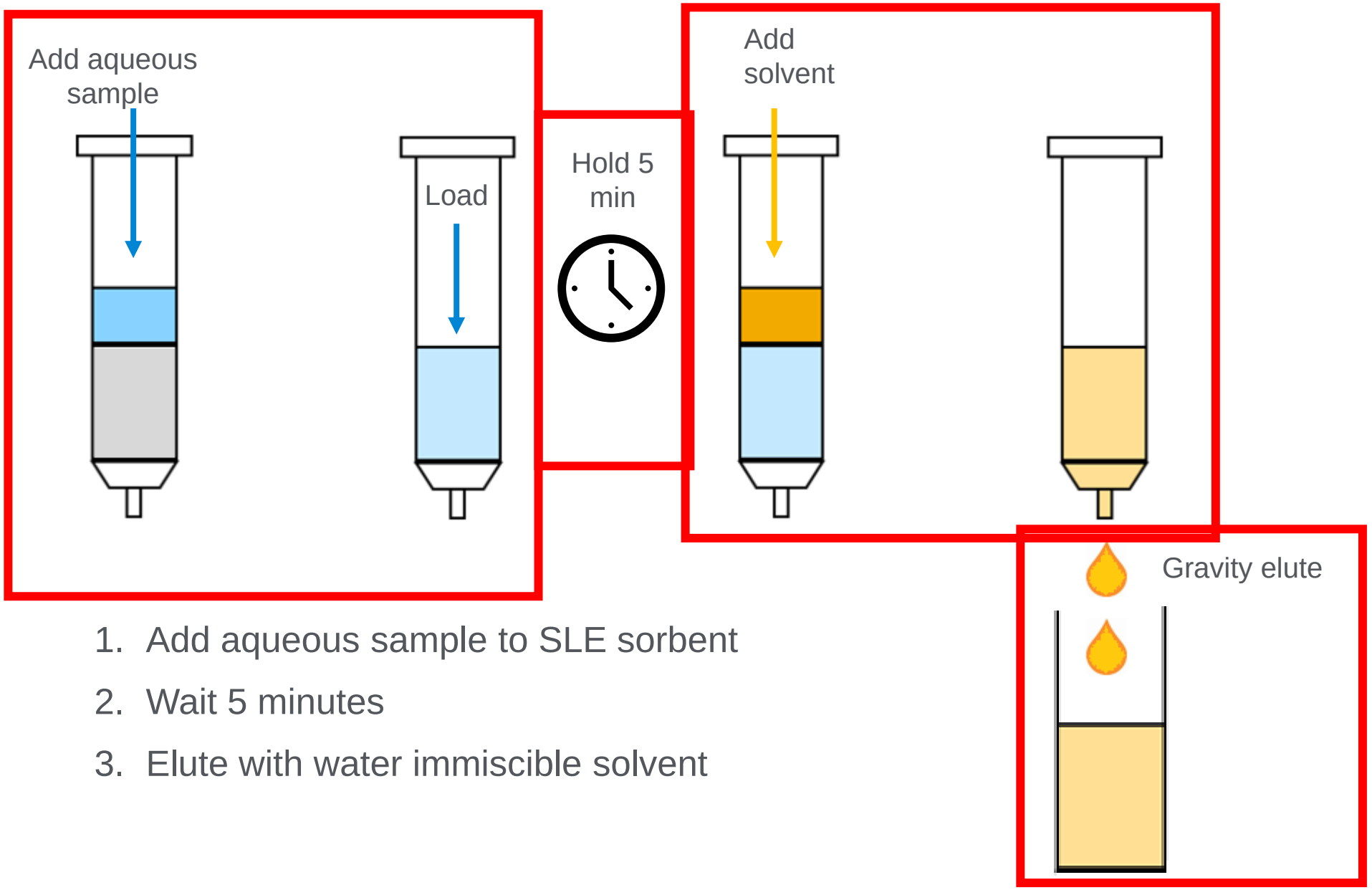
- LLE does have drawbacks
  - Inconsistent results from one analyst to another
    - Shaking time
    - Shaking motion
    - Determination of where to cut between layers
  - Emulsions
  - Labor intensive
  - Quite tedious with small sample sizes (<5 mL)
  - Challenging with large numbers of samples
  - Difficult to automate for large numbers of samples





How many of these problems can be fixed with solid supported liquid extraction?

How Does SLE Work?



1. Add aqueous sample to SLE sorbent
2. Wait 5 minutes
3. Elute with water immiscible solvent

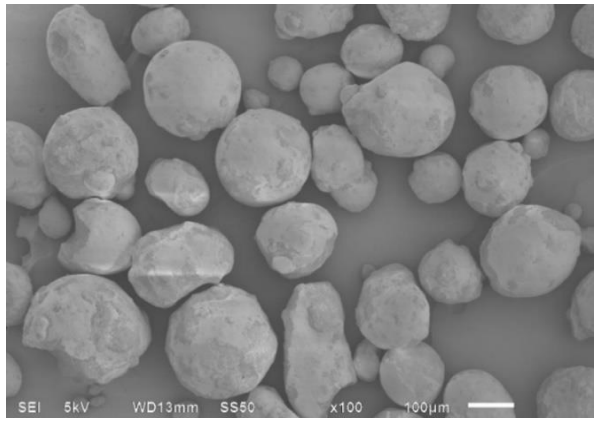
What is SLE Sorbent?

- There are two types of SLE media
  - Diatomaceous earth (DE) based products like our Chem Elut brand of SLE products
    - A mined fossil diatom material, which is heterogeneous and inconsistent from one mine to the next

Diatomaceous earth in Chem Elut

- ✗ Naturally occurring; mined
- ✗ Broad particle size distribution
- ✗ Supplier reliability issues
- ✗ Poor lot-to-lot consistency

  - Synthetic media we use in Chem Elut S
    - Controlled synthesis to be consistent batch after batch



Synthetic SLE sorbent

- ✓ Large scale synthesis
- ✓ Narrow particle size distribution
- ✓ Reliable supplier
- ✓ Controlled manufacturing

Supported Liquid Extraction (SLE)

Chem Elut S

- Same extraction mechanism as traditional liquid-liquid extraction (LLE)
- Cartridge and plate format, packed with proprietary synthetic sorbent – high surface area
- Simple method, gravity flow
- Smaller volume sample and solvent compared to LLE
- No emulsions

Cartridges for sample volumes 0.2 – 20 mL

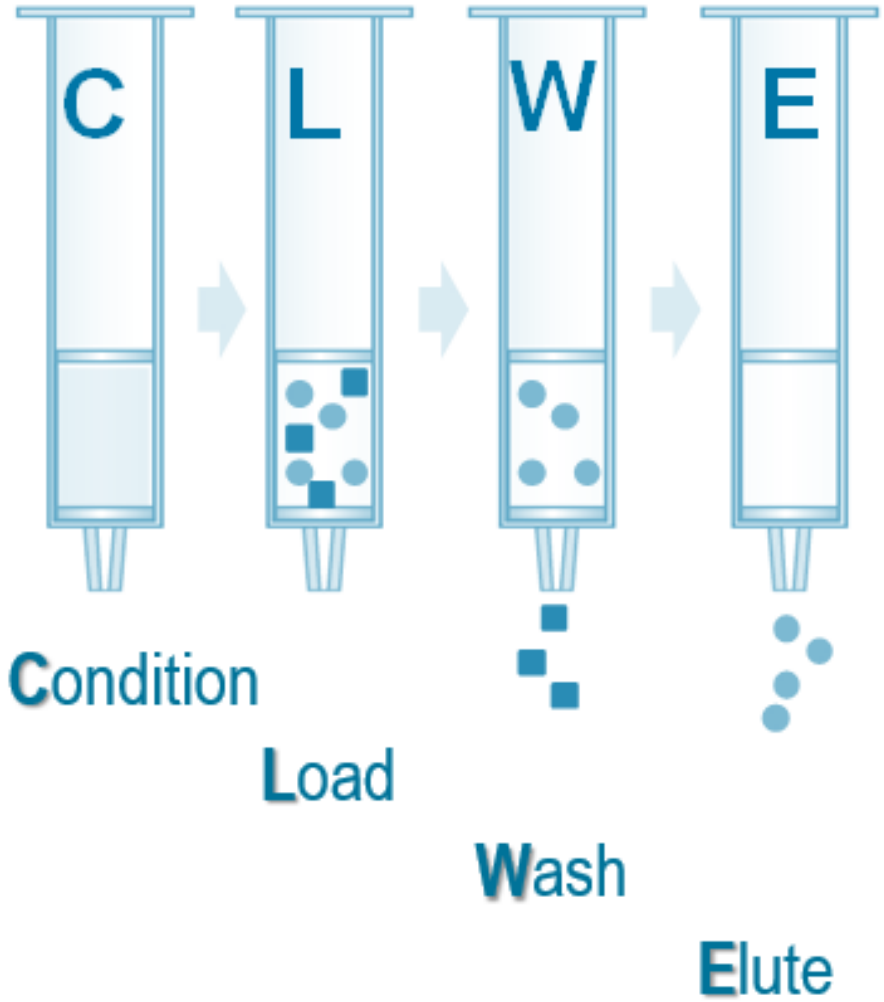
Bulk Chem Elut S
1 and 4 kg

96-well plate for sample volume 200 and 400 µL

Solid Phase Extraction (SPE)

- Capabilities
  - Very selective
  - Highly clean samples
  - Concentrated samples
  - Wide range of applicability
  - Automation friendly

- Types of SPE
  - Nonpolar (reversed phase) SPE
  - Polar (normal phase) SPE
  - Cation exchange SPE
  - Anion exchange SPE
  - Mixed mode SPE
  - Specialty SPE



Bond Elut:

Silica or polymer based, cartridge and 96-well plate format

Agilent SPE Offering

- Reliable SPE with a 30-year history
- Agilent offers the most comprehensive set of phases, sizes, and formats of any SPE provider (over 40 sorbent materials/phases available).
- Easy adoption of methods due to high number of publications and applications
- Includes packed bed silica and polymeric phases, and monolithic silica phases

Bond Elut Silica and polymer SPE

Bond Elut AccuCAT
Bond Elut Alumina (AL-A)
Bond Elut Alumina (AL-B)
Bond Elut Alumina (AL-N)
Bond Elut NH_2
Bond Elut C1
Bond Elut C2
Bond Elut C8
Bond Elut C18 .....
..... **40 phases**

Bond Elut Plexa polymer SPE

Bond Elut Plexa
Bond Elut Plexa PCX
Bond Elut Plexa PAX

OMIX monolithic silica tip SPE

OMIX C18
OMIX MP1
OMIX SCX

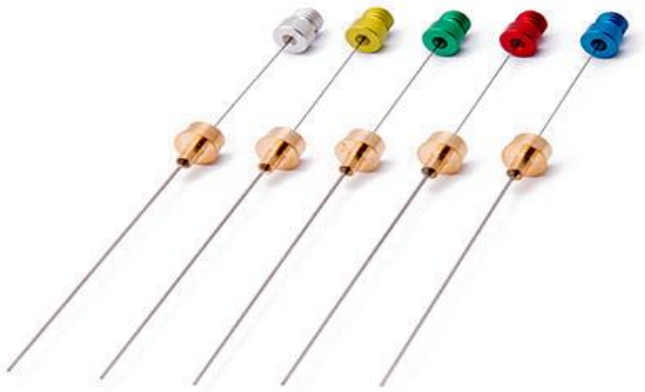
SPEC monolithic silica disk SPE

SPEC C2
SPEC C8
SPEC C18
SPEC C18AR
SPEC PH
SPEC NH2
SPEC CN
SPEC Si
SPEC PSA
SPEC SAX
SPEC SCX
SPEC MP1
SPEC MP3

SPME Fiber and Arrow Offering from Agilent

Solid phase microextraction (SPME)

- Environmental analyses of water samples
- Odor analyses (ppt)
- Flavor analyses of food products
- Surfactants, other industrial applications
- Trace analysis in food
- Herbicides/pesticides
- Trace impurities in polymers and solid samples
- Solvent residues in raw materials
- Explosives



SPME fibers

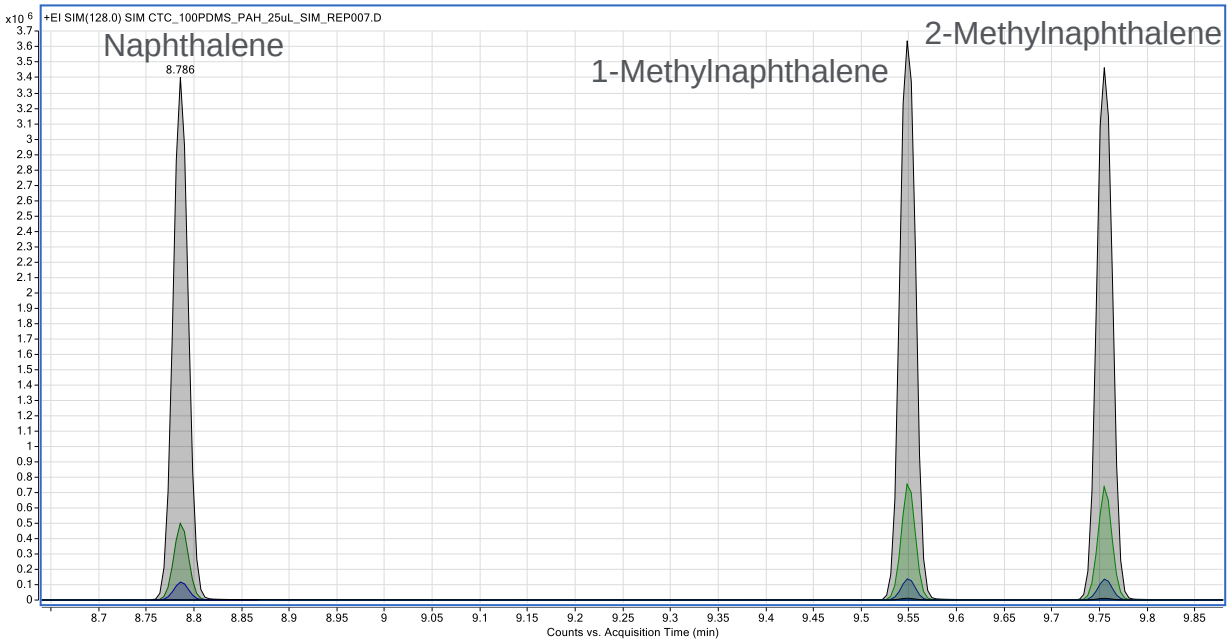
SPME arrows

Smart SPME Fibers and Arrows are now available for the smart rail systems

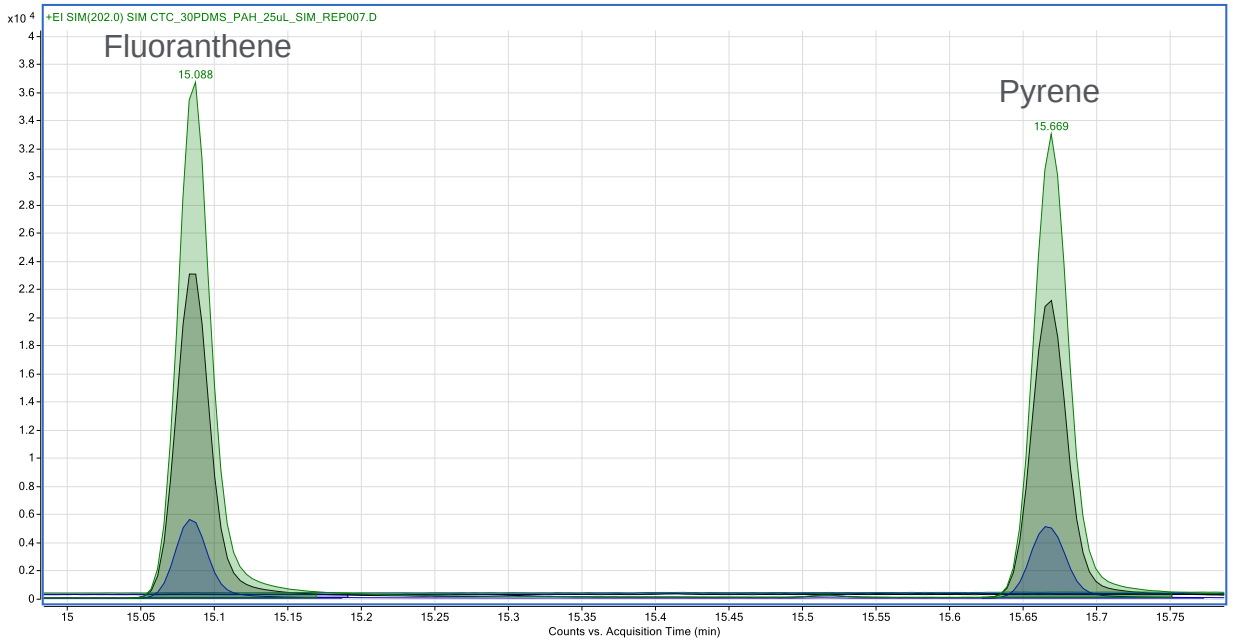
Examination of Lower Molecular Weight PAHs in Drinking Water Using Agilent PDMS SPME Fibers

Polycyclic aromatic hydrocarbons (PAHs) are a large class of organic compounds containing two or more fused aromatic rings. PAHs are considered compounds of concern by environmental organizations; their concentration in water is strictly regulated.

5994-1301EN



SIM chromatogram of naphthalenes with PDMS fibers (black trace = 100 µm; green trace = 30 µm; blue trace = 7 µm)



SIM chromatogram of fluoranthene and pyrene with PDMS fibers (black trace = 100 µm; green trace = 30 µm; blue trace = 7 µm)

Agilent Bond Elut QuEChERS

Quick Easy Cheap Effective Rugged and Safe

Initially developed for screening of pesticide residues in fruit and vegetables to make sample cleanup of food faster, simpler, less expensive, and greener.

Now, QuEChERS is used with other matrices and compound classes as well.

Consists of two steps, and therefore two kits:

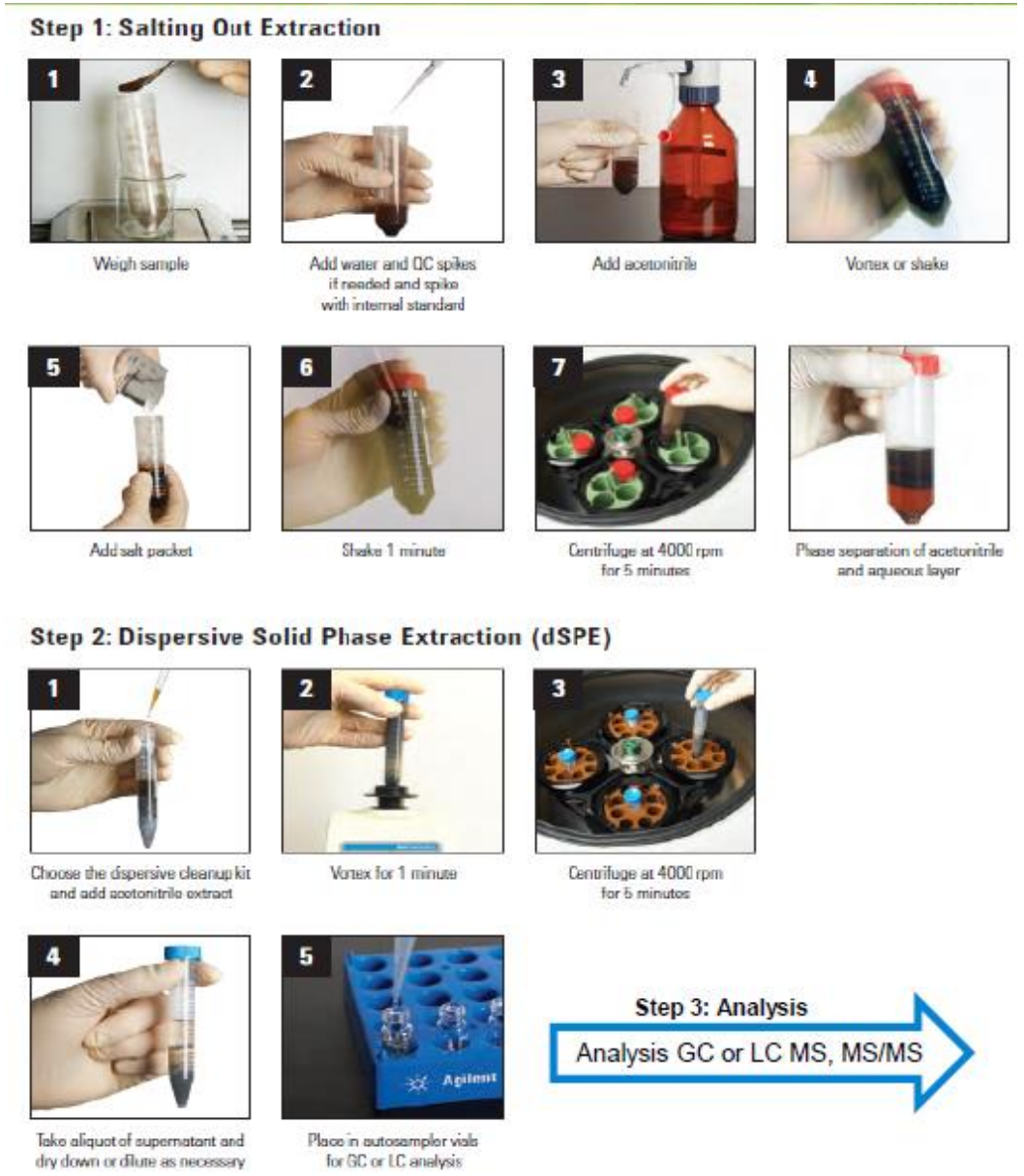
Step 1: Liquid extraction

Step 2: Dispersive SPE/ interference removal

QuEChERS Workflow

QuEChERS extraction salts

QuEChERS dispersive SPE sorbents



Bond Elut Dispersive SPE Kits

Dispersive kit

Centrifuge tubes containing preweighed SPE sorbent such as:

- C18: Removes residual fats and lipids
- PSA: ‘Primary/secondary amine’ for removal of organic acids and sugars
- Carbon S: Graphitized carbon black, removes pigments
- EMR-Lipid: Removes unbranched hydrocarbon chains (lipids)

Dispersive SPE kits are available for different food types.

They are for both AOAC (US) method and EN (Europe).

QuEChERS is a nonselective technique and does not remove **all** matrix, just enough.

Dispersive sorbents are also available as bulk material.

Sample Preparation Application Note Examples using GC Detection

Captiva EMR-Lipid

1. Determination of 14 Polycyclic Aromatic Hydrocarbon Compounds in Edible Oil, PN 5994-1483EN
2. Analysis of Multiclass Multiresidue Pesticides in Milk, PN 5994-2038EN

Captiva EMR with Carbon S

1. Determination of Over 300 Pesticides in Cayenne Pepper, PN 5994-5630EN
2. Determination of Multiclass, Multiresidue Pesticides in Bell Peppers, PN 5994-4767EN

Chem Elut S

1. Determination of Aromatic Amines Derived from Azo Colorants by GC/MS, PN 5994-0951EN

SPME

1. Analysis of Free Volatile Phenols in Smoke-Impacted Wines by SPME, PN 5994-3161EN

Manifolds for Processing Cartridges and 96-Well Plates

Captiva vacuum collar

Vac Elut 20 vacuum manifold

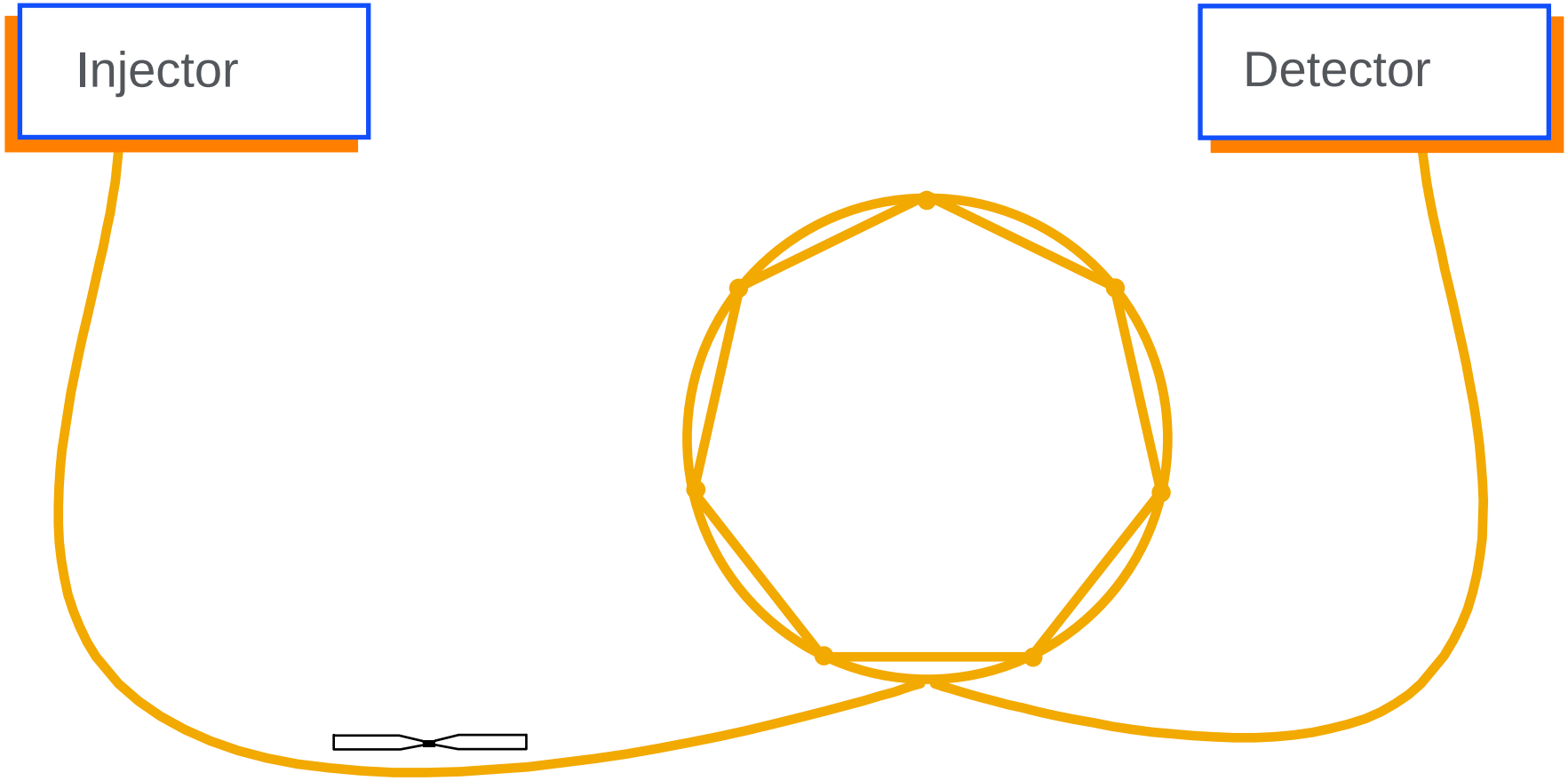
Vac Elut 12 vacuum manifold

SPS 24 vacuum manifold

96 well plate vacuum manifold

Positive Pressure Manifolds

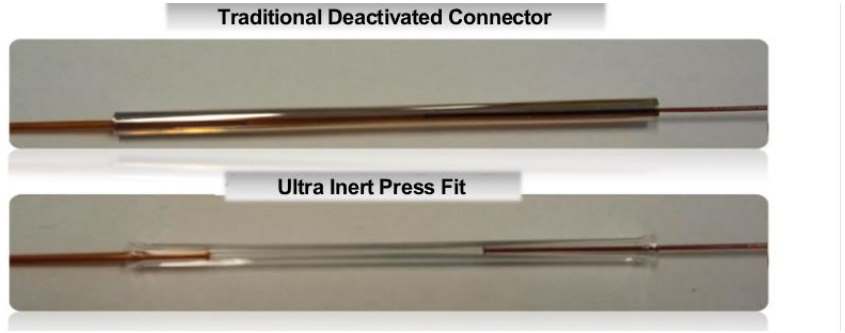
Guard Column or Retention Gap



The guard column is 3 to 5 m of deactivated fused silica tubing with the same diameter as the analytical column. It is connected with a zero dead volume union.

Better Connections: Ultra Inert Press Fits or Ultimate Union

- Ultra Inert press fits:
  - Join retention gap or guard column to analytical column
  - Dependable inertness performance at a lower cost
  - Batch certified inertness
  - Improved packaging and installation instructions
  - Easter to use – transparent deactivation gives visibility of the column connection
- Ultimate union
  - More robust
  - Reusable
  - Recommended for users with MS



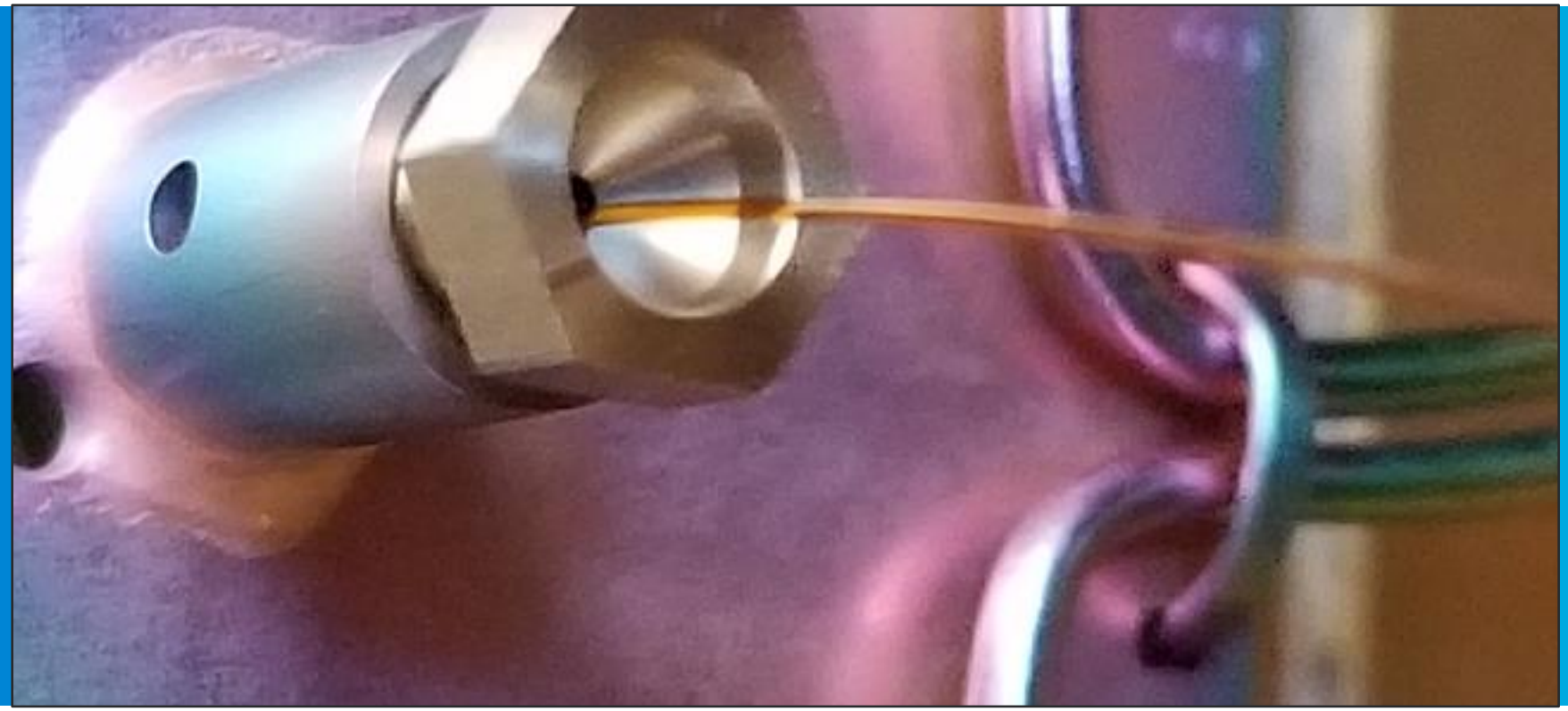
Integrated Guards – DuraGuard

- No union
- Possible for any DB column 0.18 mm and larger
- Limited offering “off-the-shelf”

DuraGuard

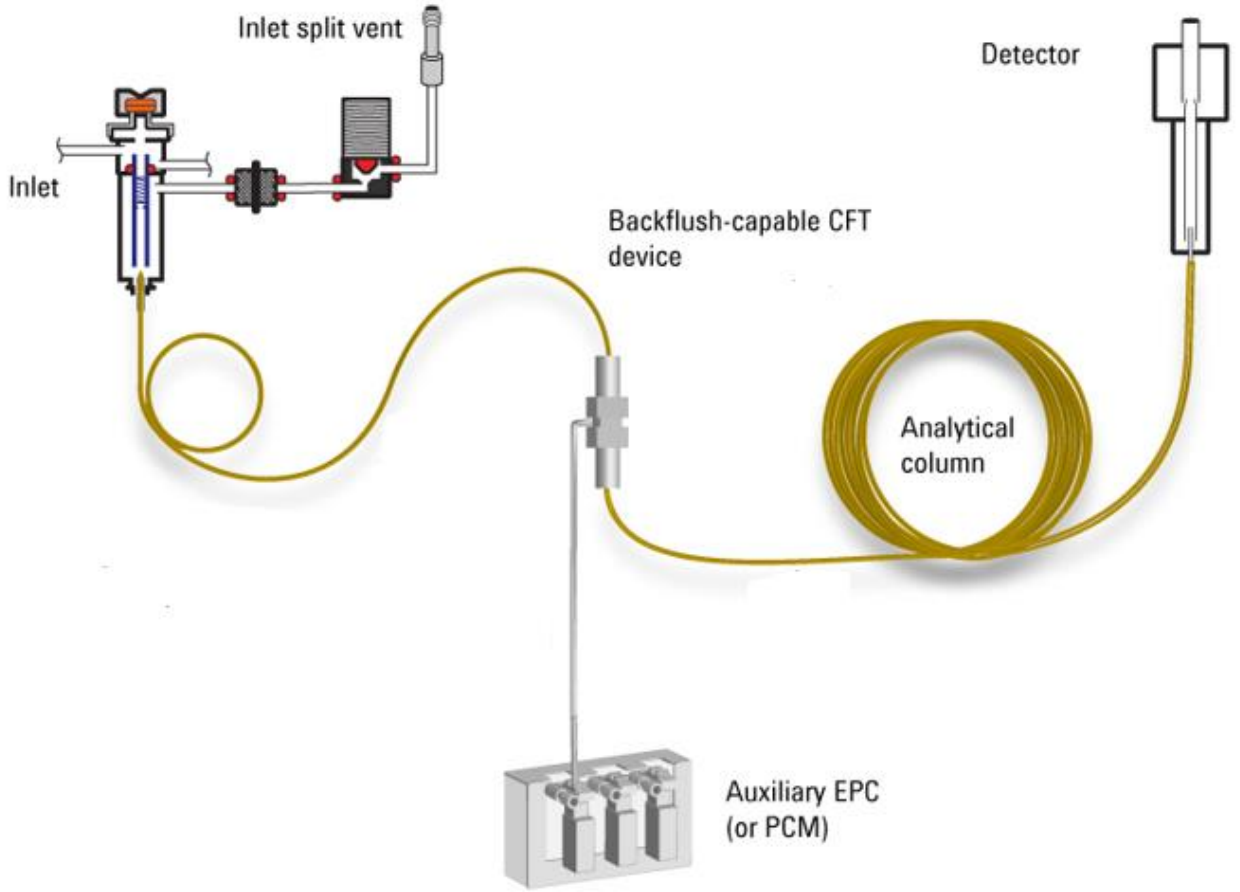
Phase	ID (mm)	Length (m)	Film (µm)	Guard Length (m)	Part No.
DB-1	0.25	30	0.25	10	122-1032G
DB-XLB	0.25	30	0.25	10	122-1232G
DB-5ms	0.25	30	0.25	10	122-5532G
			0.50	10	122-5536G
			1.00	10	122-5533G
		60	0.25	10	122-5562G
	0.32	30	1.00	10	123-5533G
	0.53	30	0.50	10	125-5537G
DB-5.625	0.18	20	0.36	5	121-5622G5
	0.25	30	0.25	5	122-5631G5
DB-1701	0.53	30	1.00	10	125-0732G
DB-624	0.53	30	3.00	5	125-1334G5

An Introduction to Backflush Techniques

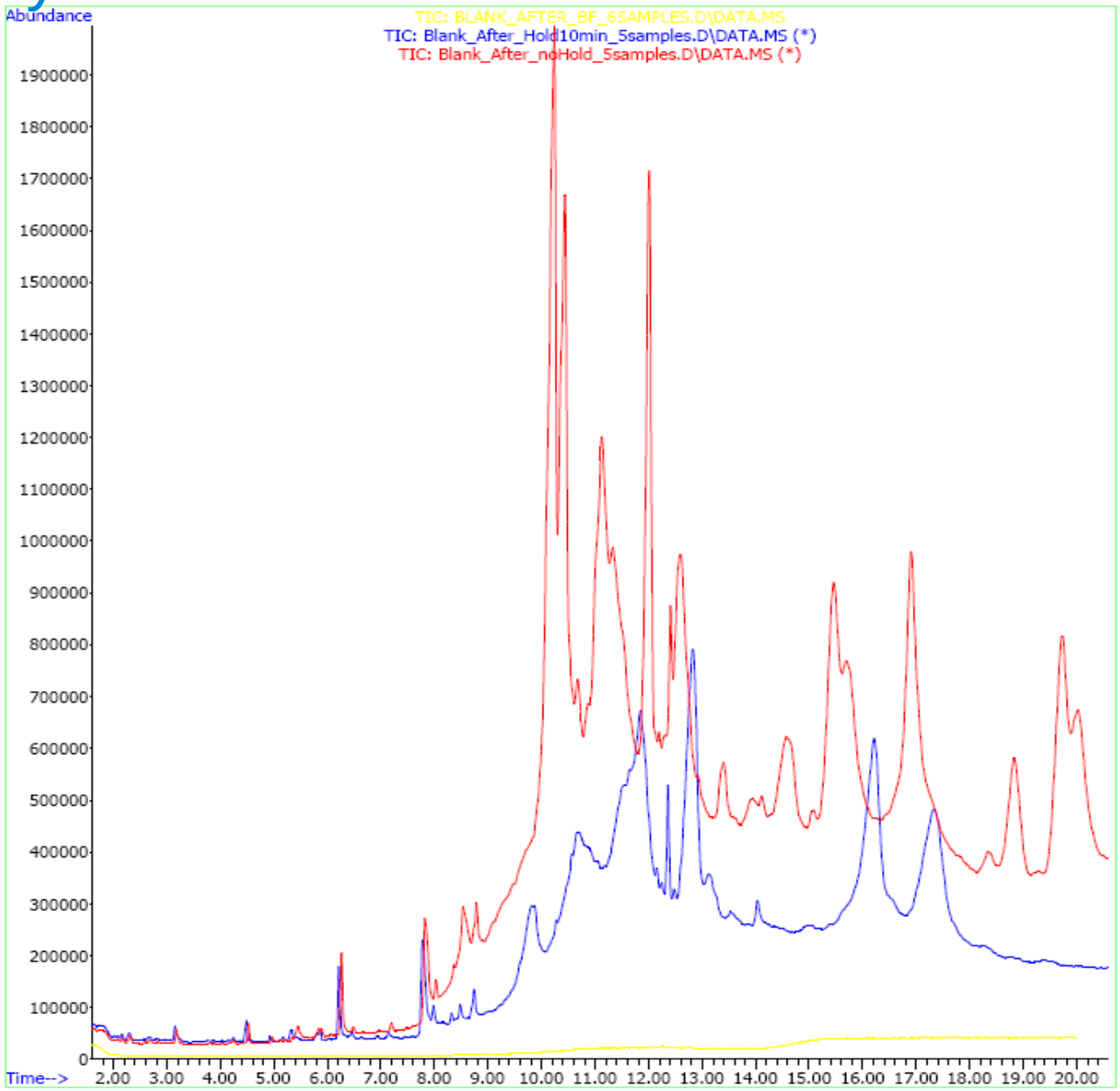


Why Use Backflush?

- Avoid unwanted sample components from entering analytical column
- Avoid heavy compounds from reaching detector
- Shorten run times and increase sample throughput



Why Use Backflush?

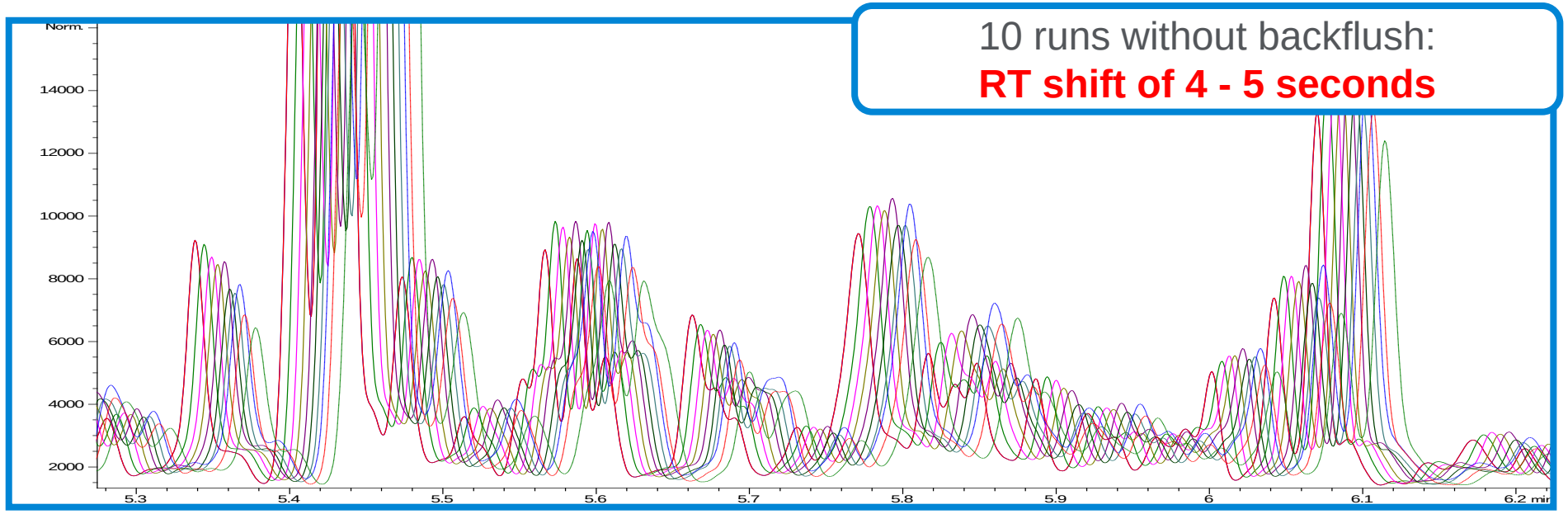


Blank after five injections (normal method)

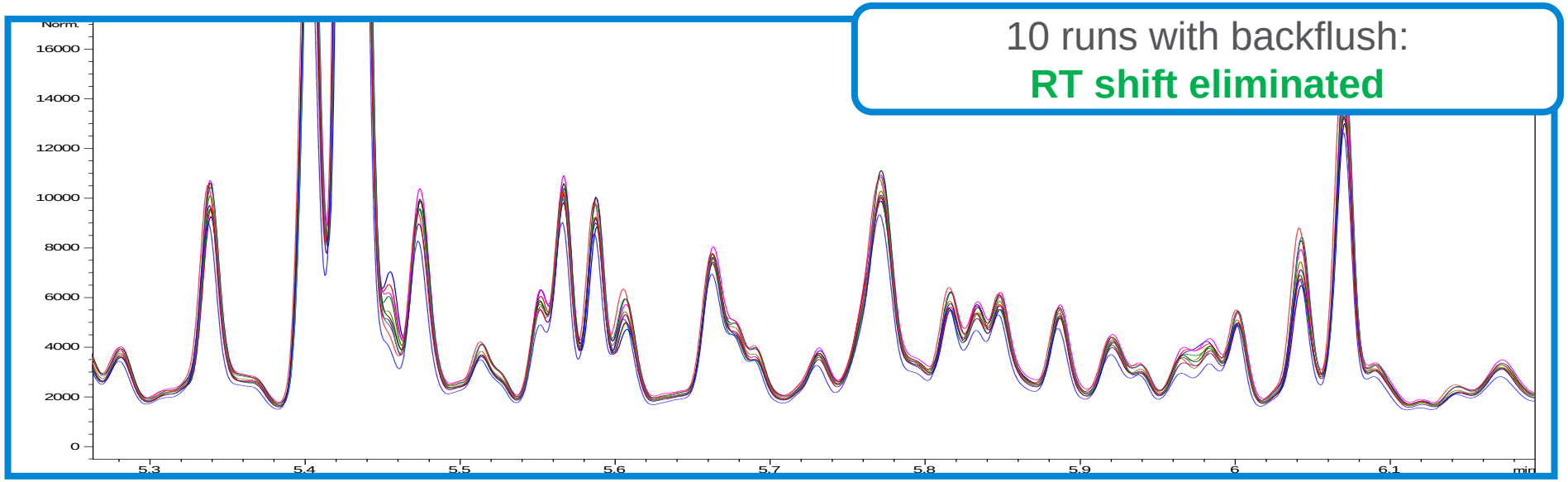
Blank after five injections (with 10 minute bake at 280 °C)

Blank after five injections (2 minute backflush, 50 PSI at 280 ° C)

Why Use Backflush?

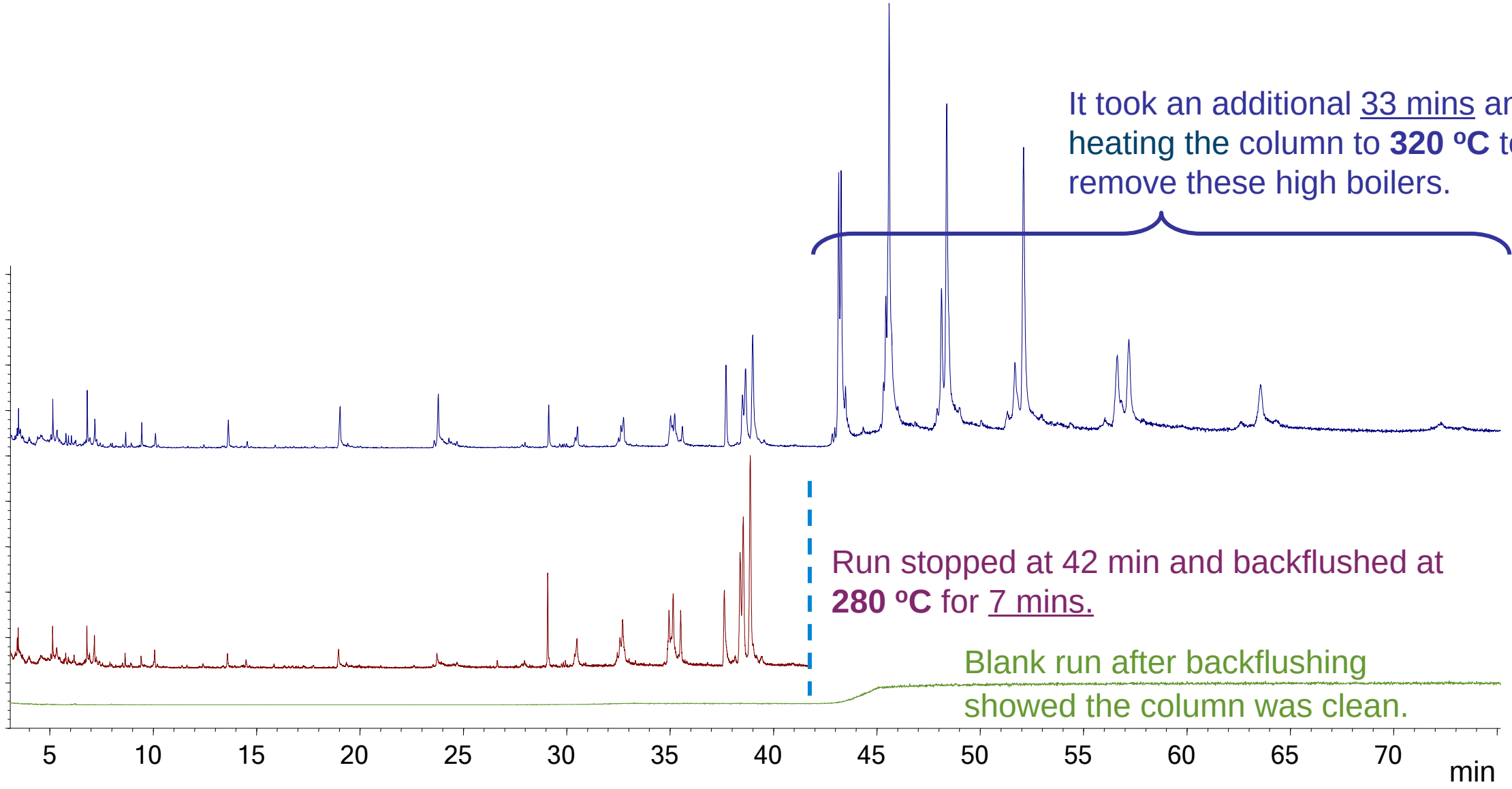


10 runs without backflush:
RT shift of 4 - 5 seconds

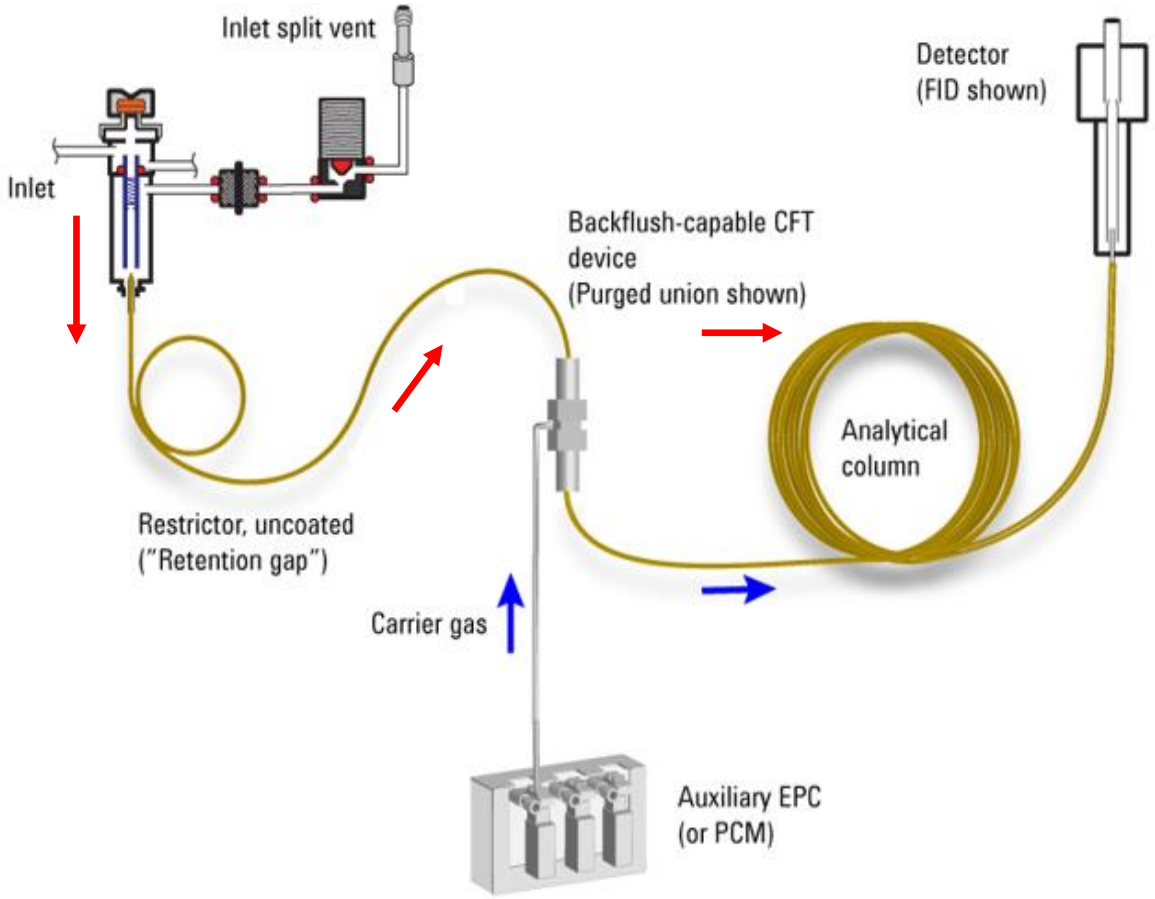


10 runs with backflush:
RT shift eliminated

Why Use Backflush?



Why Use Backflush?



Summary

- Stay within the temperature limits of your column and handle it carefully
- Be mindful of what you are injecting into your system
- Take notice of any chromatography or baseline changes from when the column was brand new
- Ultimately, sample preparation/cleanup is the most reliable way to address common chromatography data problems.
- Agilent offers a wide range of sample preparation products to support your analysis using established methods and protocols:
  - Filtration, protein and lipid removal
  - SLE
  - QuEChERS
  - SPE
- Backflush techniques can help extend the lifetime of your column if your instrument can be set up with it

Contact Agilent Chemistries and Supplies Technical Support

1-800-227-9770 option 3, option 3:

Option 1 for GC and GC/MS columns and supplies

Option 2 for LC and LC/MS columns and supplies

Option 3 for sample preparation, filtration, and QuEChERS

Option 4 for spectroscopy supplies

Option 5 for chemical standards

Available in the U.S. and Canada 8–5, all time zones.

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