Keeping Your Chromatography Alive: Tips and Tricks for Getting the Most out of Your GC Column

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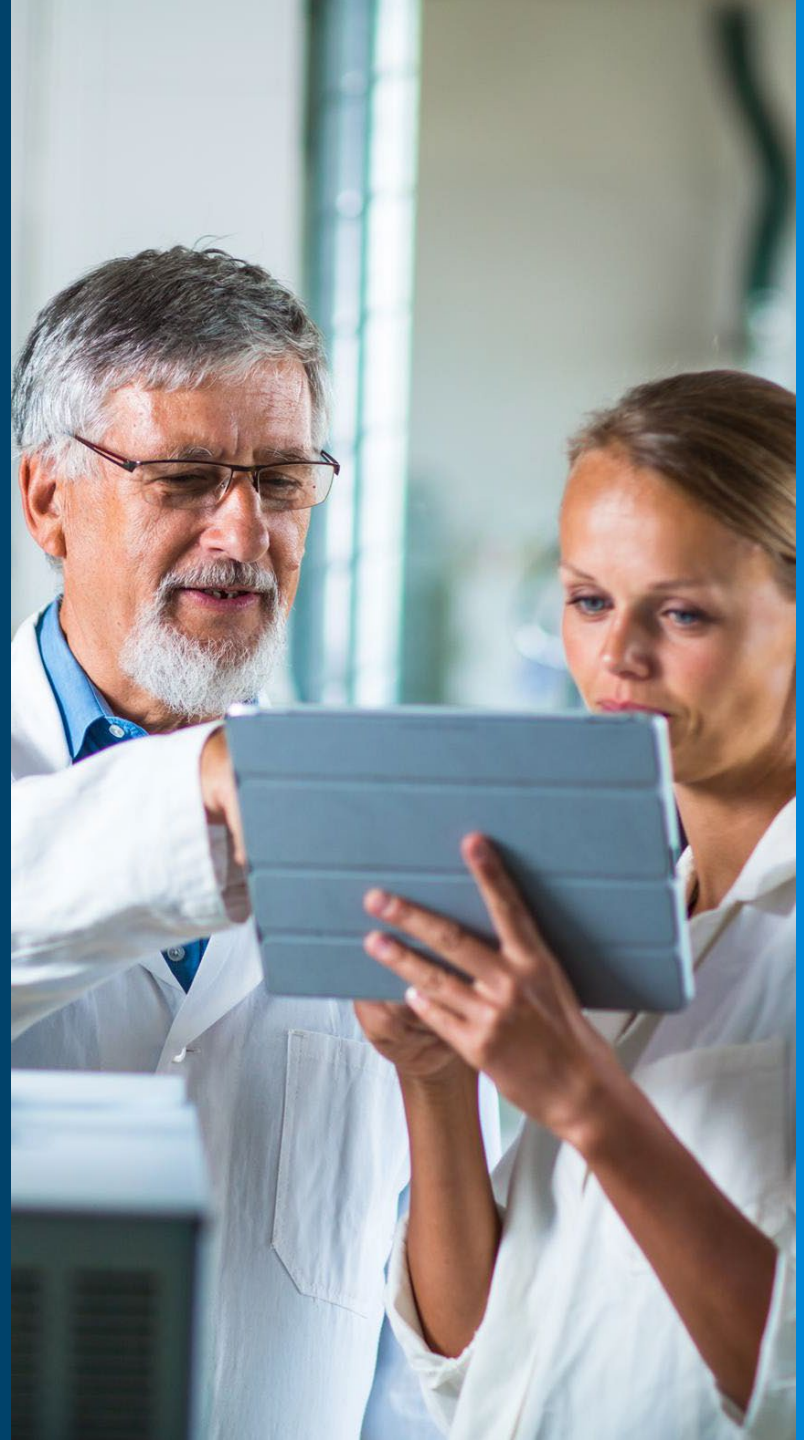
Online Application Engineer
September 24, 2020

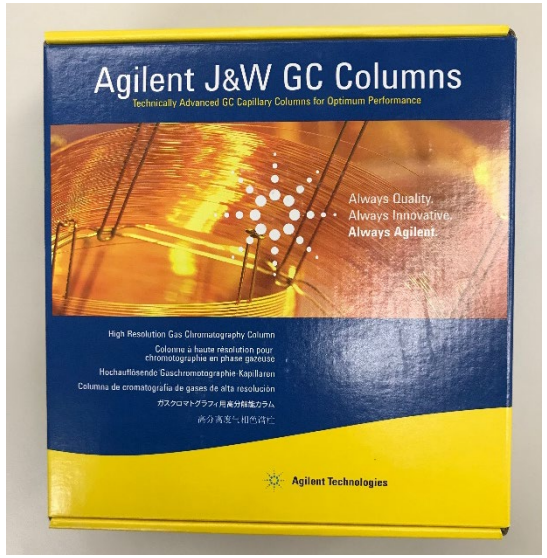


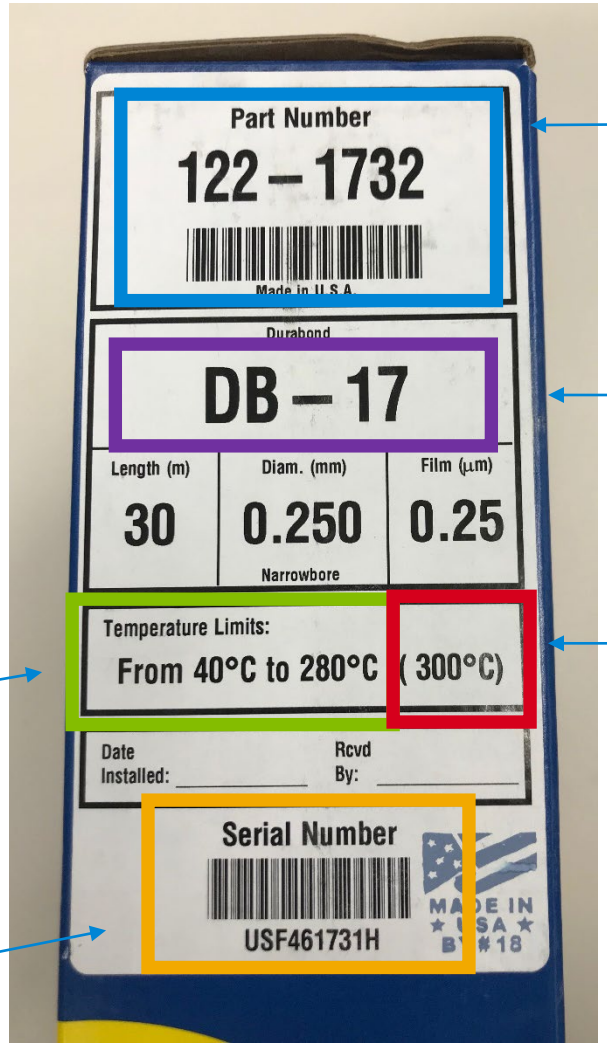
Agilent
Trusted Answers

Agenda

- Review: Get to know your column
- Signs and symptoms of an unhealthy column
- How to know if your column is healthy?
- What you can 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 reordering

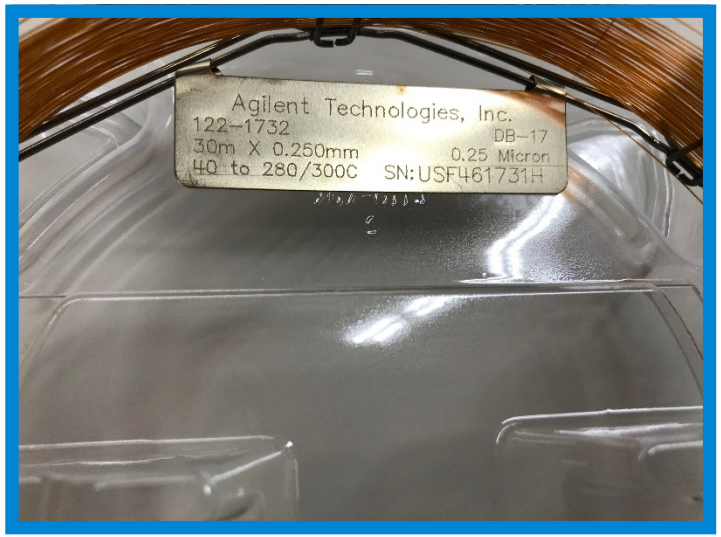
Column stationary phase

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 into storage, use this plug 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:

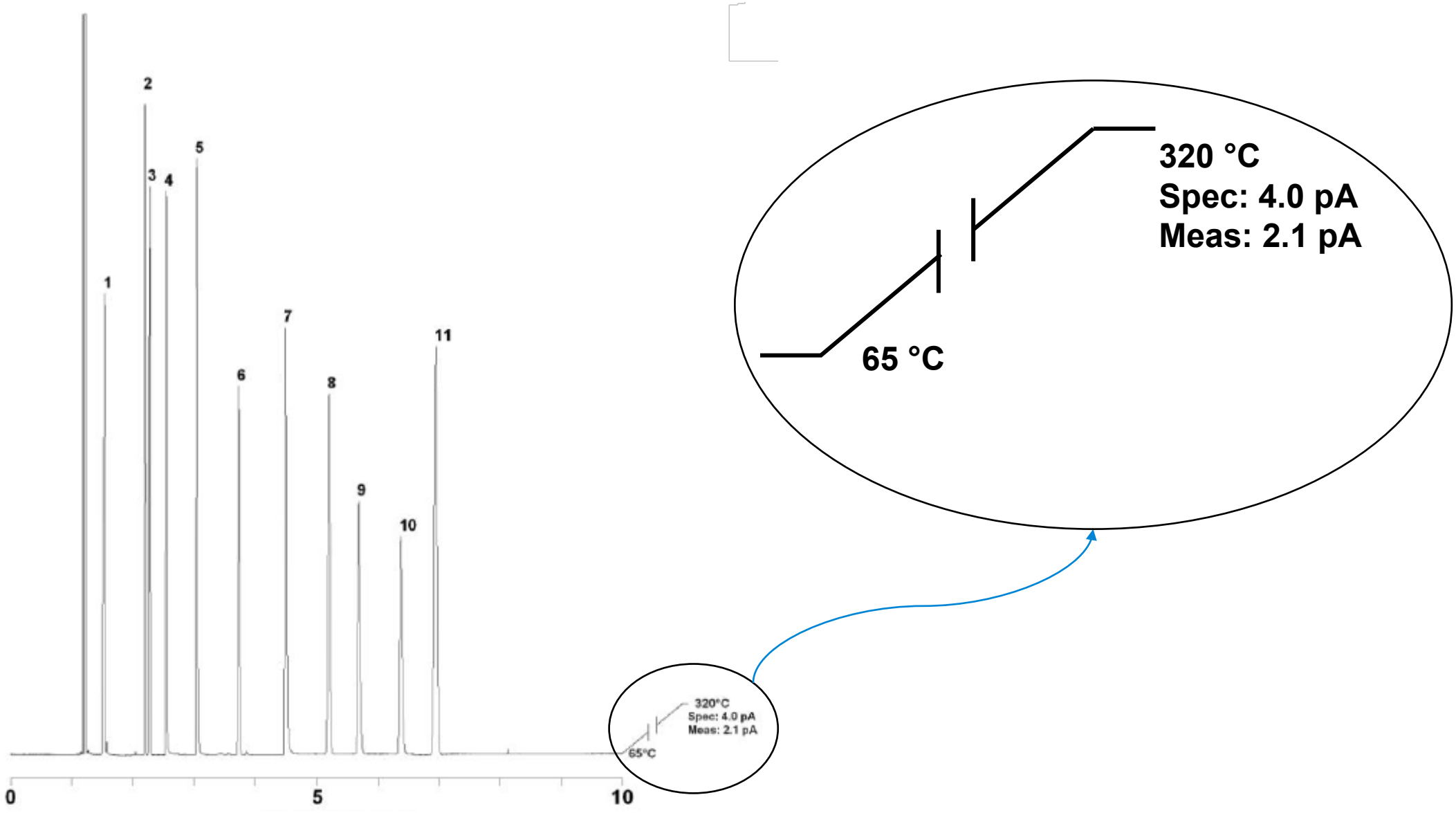
1-OCTENE, n-OCTANE	2.97

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

Common Peak Shape Issues

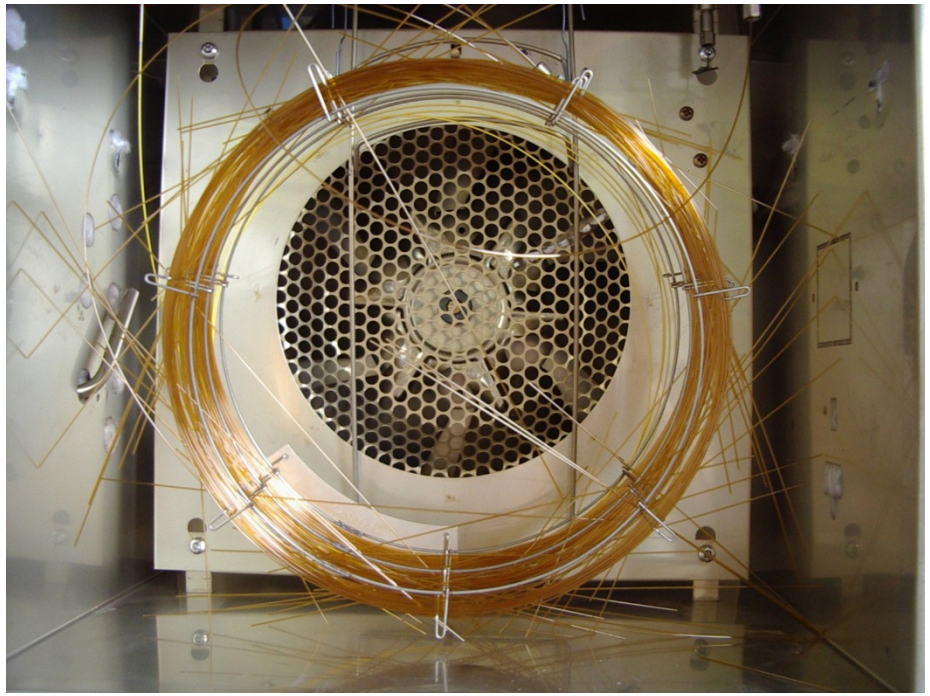
- **Peak tailing** – flow path or activity
- **Bonus peaks** – in sample or backflash (carryover)
- **Split peaks** – injector problems, mixed solvent
- **No peaks** – wasn't introduced, wasn't detected
- **Response changes** – activity, injector discrimination, detector problem
- **Peak fronting** – overload or solubility mismatch, injector problems
- **Shifting retention** – leaks, column ageing, 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



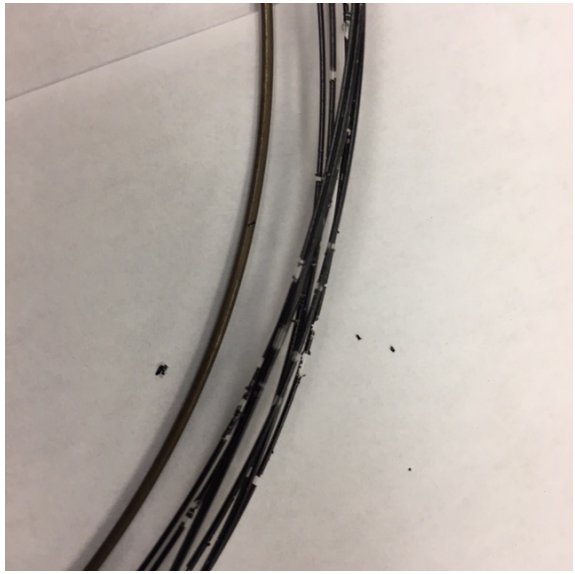
Thermal Damage

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

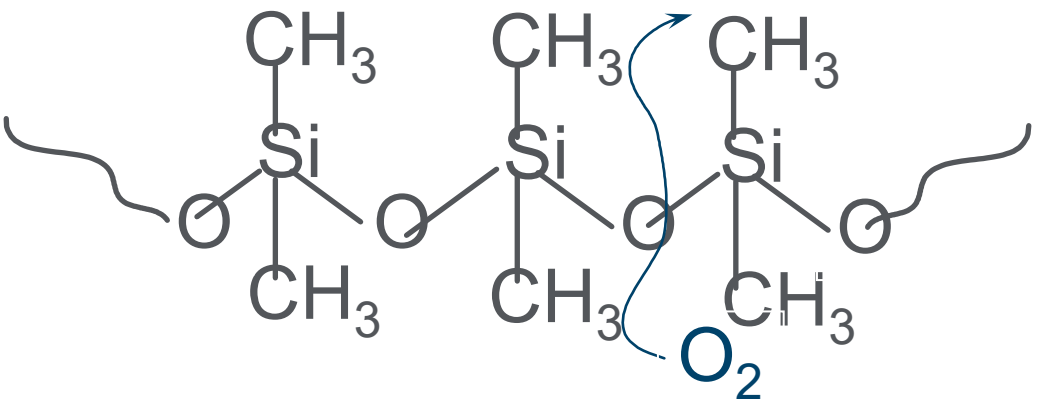
- Temporary "column failure" below lower temperature limit
- If this happens:
  - Disconnect column from detector
  - “Bake out” overnight at isothermal limit
  - Remove 10–15 cm from column end



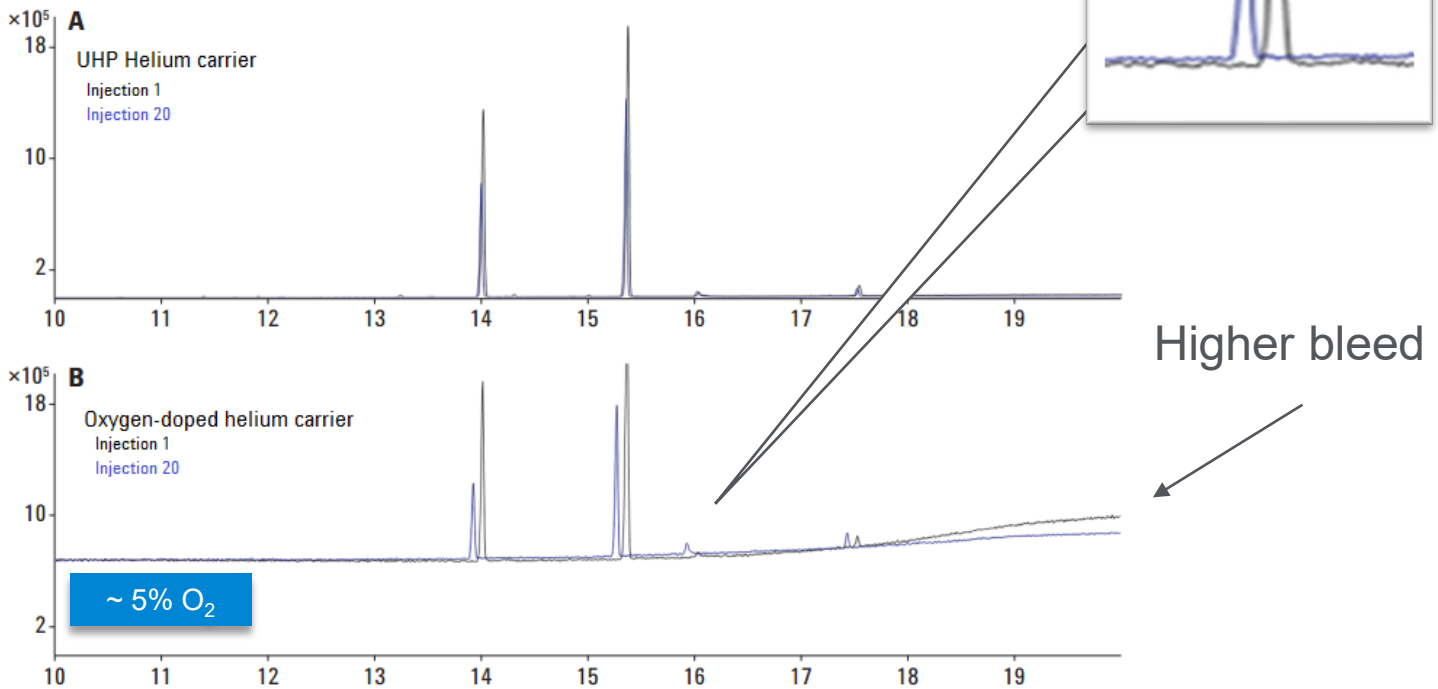
Column continuously exposed to temperatures above its temperature limit

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).

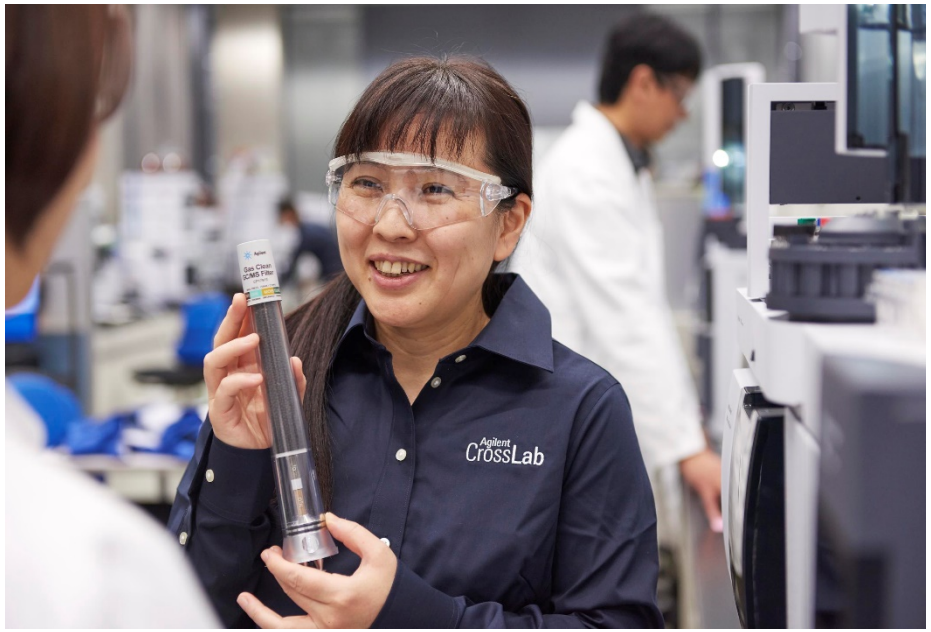


Dimethylpolysiloxane



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

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 columns
- Severe cases may require removal of up to 5 m

What is a Bleed Problem?

A bleed problem is:

- An abnormal elevated baseline at high temperature

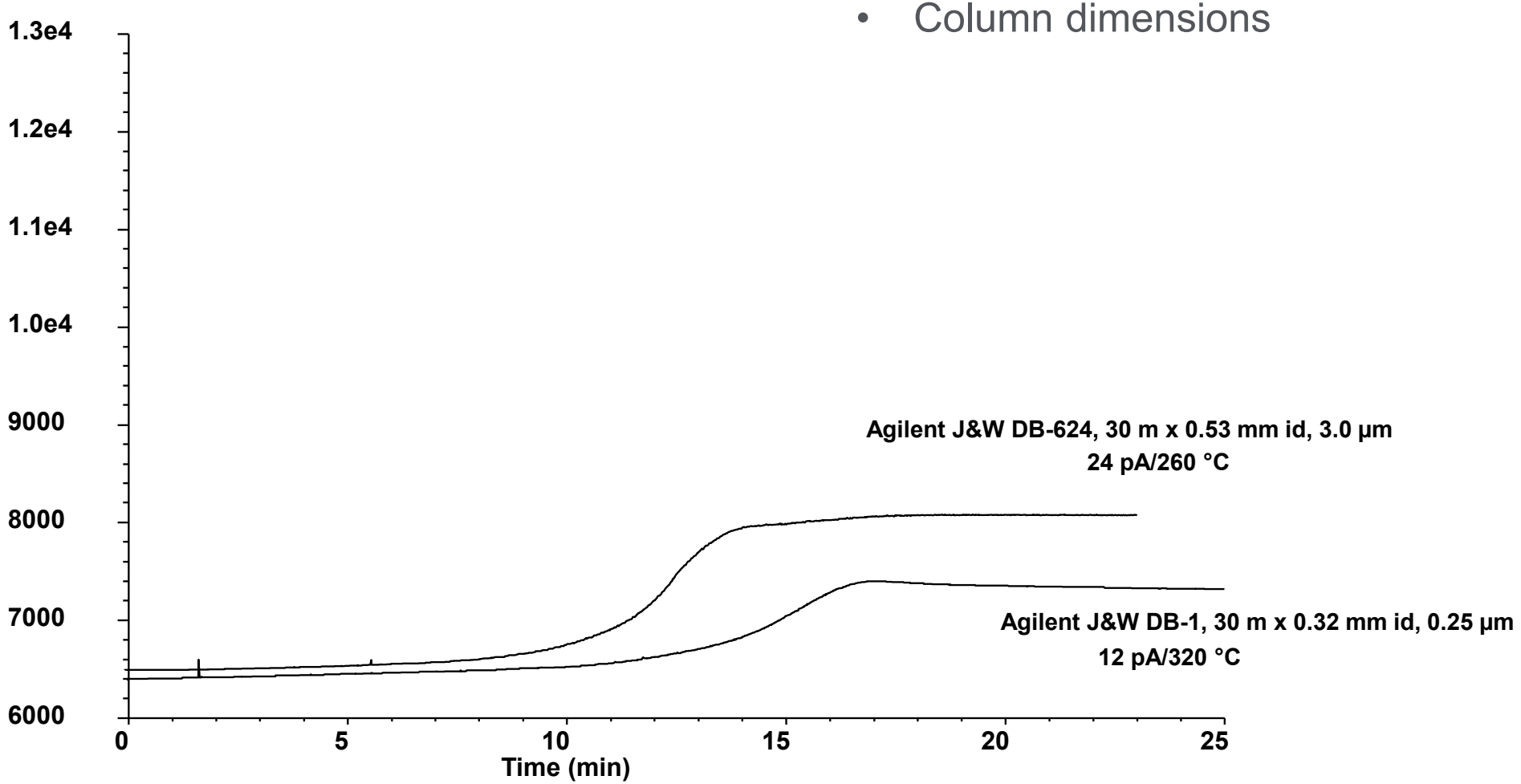
A bleed problem is **not**:

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

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



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)

Typical Samples That Contain a Large Amount of Residues

Biological

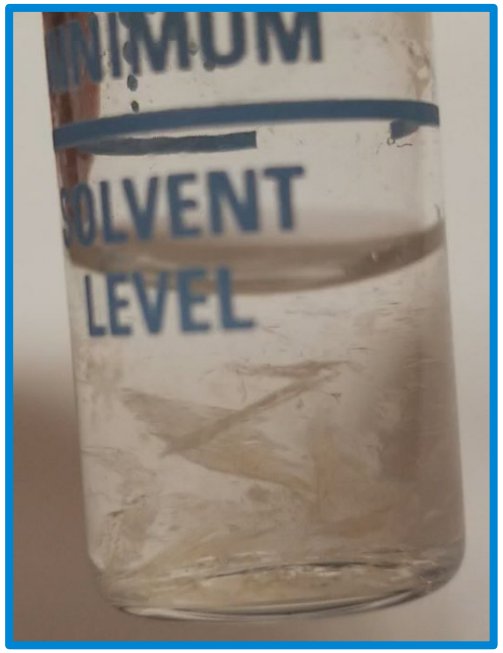
Soils Foods

Wastewater Sludges

All samples contain residues (even standards)

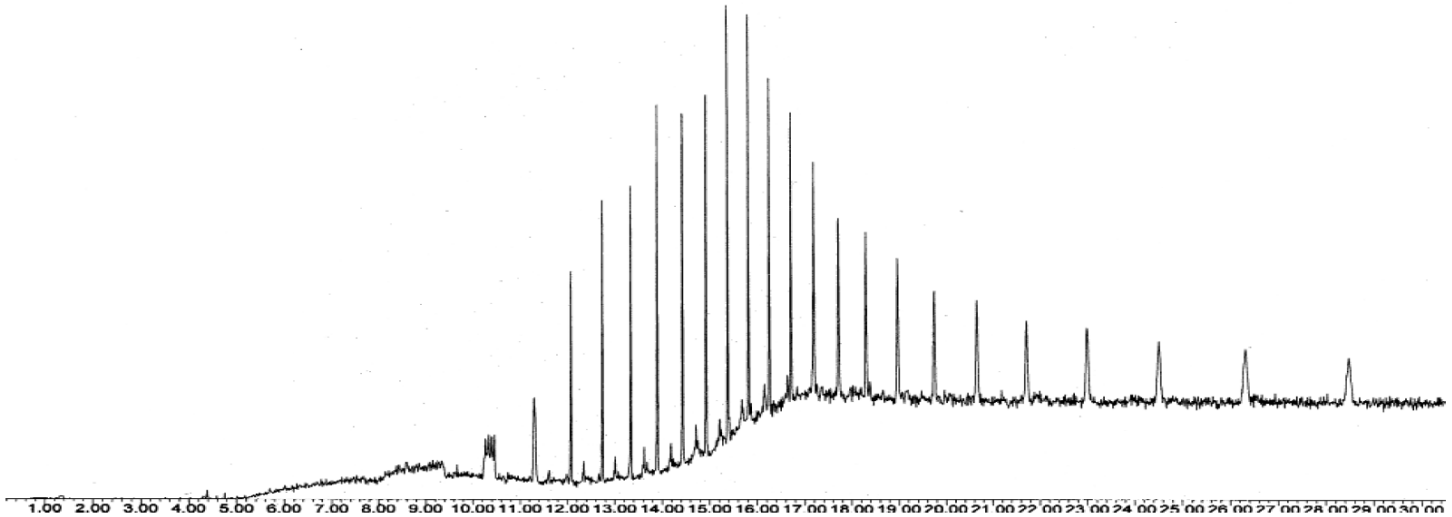
Other Sources of Contamination

- Septum and ferrule particles
- Gas and trap impurities
- Unknown sources (vials, syringes, etc.)



Contaminated wash solvent

Sample vial septum bleed profile:



Types of Residues

Nonvolatile residues:

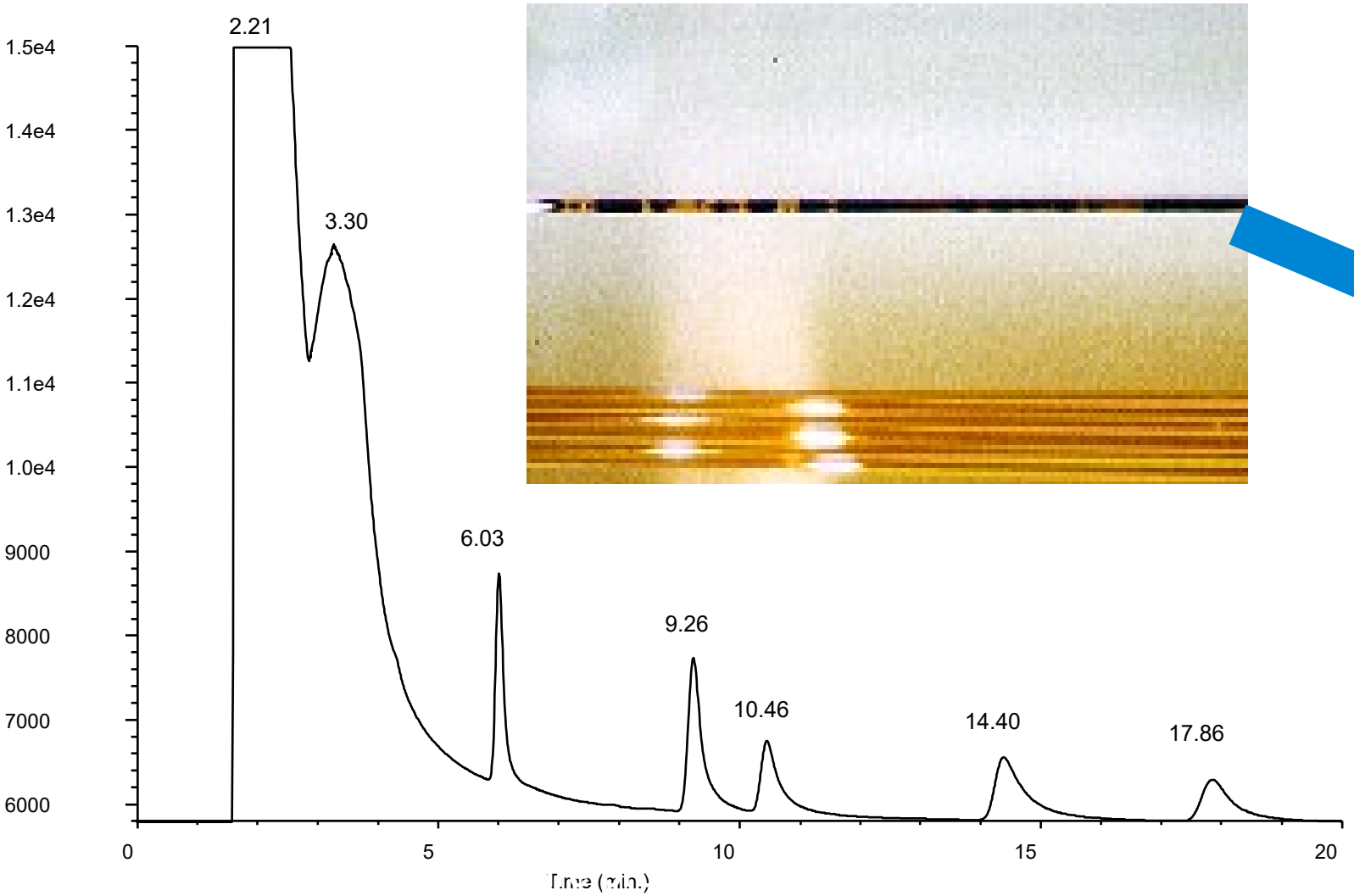
- Any portion of the sample that does not elute from the column or remains in the injector.

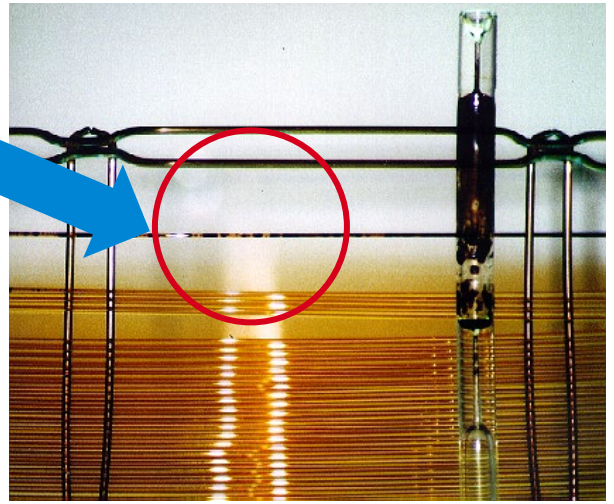
Semivolatile residues:

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

Example of Column Contamination

Agilent J&W DB-624 QC Test Mix*
After 75 injections of oily sample





Nonvolatile Contamination

What to do if it happens

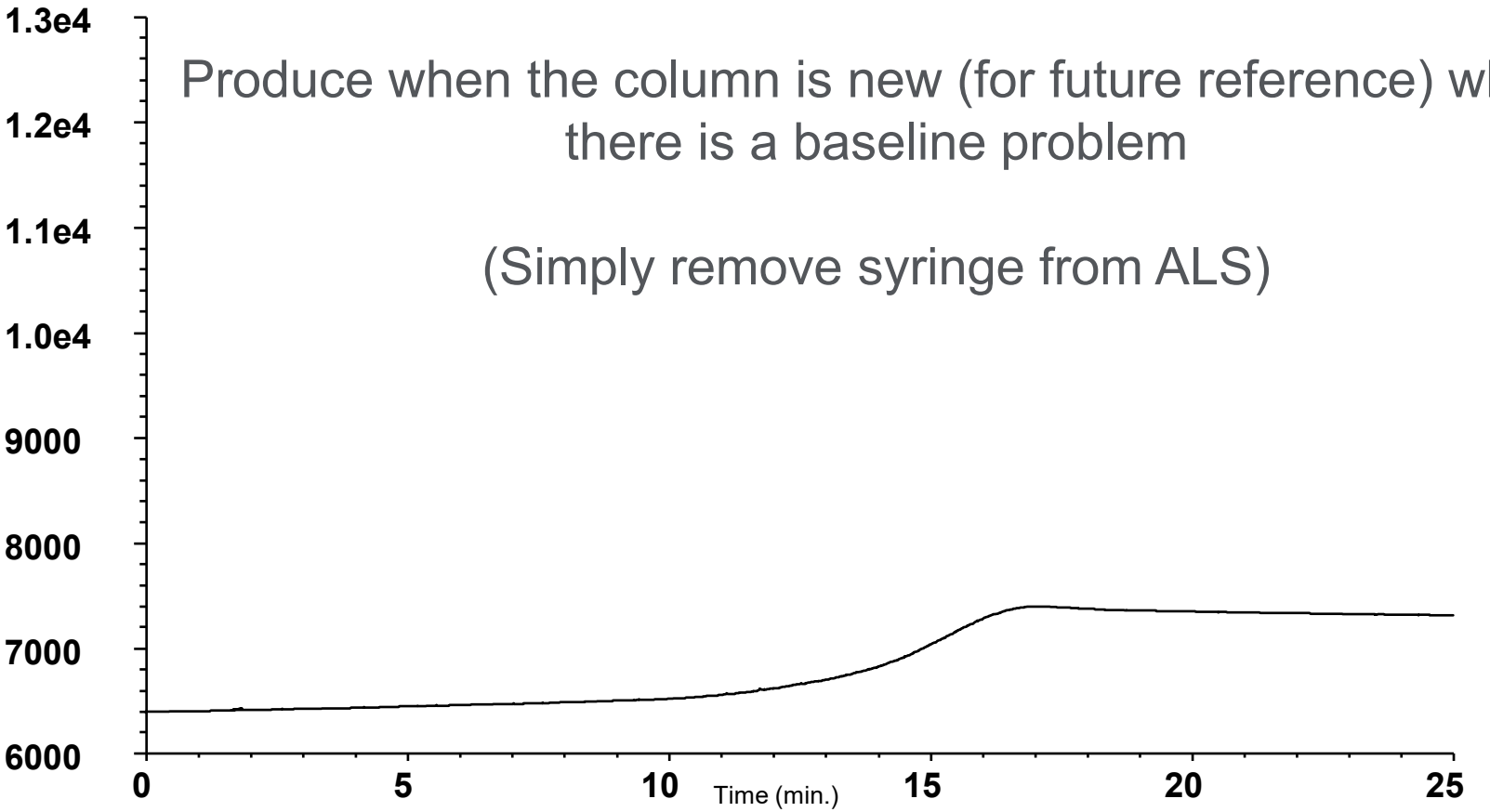
- Do not “bake out” the column
- Front-end maintenance
  - Clean or change the injector liner
  - Clean the injector
  - Cut off 0.5–1 m of the front of the column
- Turn the column around
- Cut the column in half

Semivolatile Contamination

What to do if it happens

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

Running an Instrument Blank



Produce when the column is new (for future reference) when there is a baseline problem

(Simply remove syringe from ALS)

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.

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

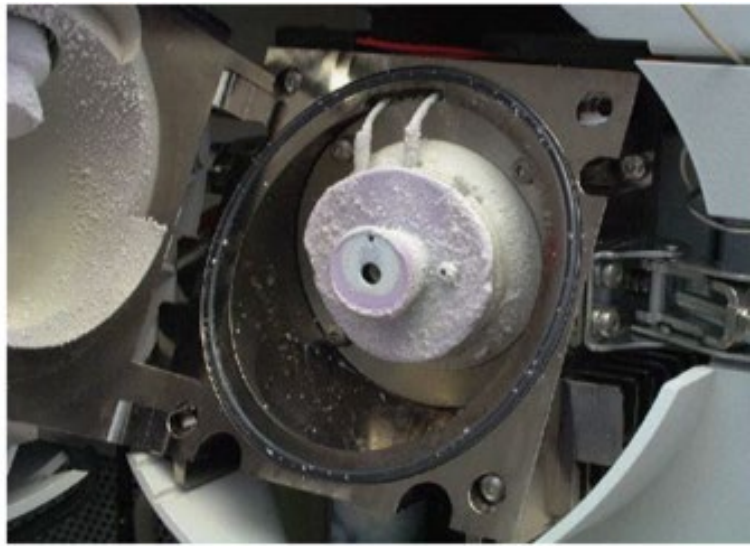


Image of salt buildup on an ESI-LC/MS inlet from unremoved salts.

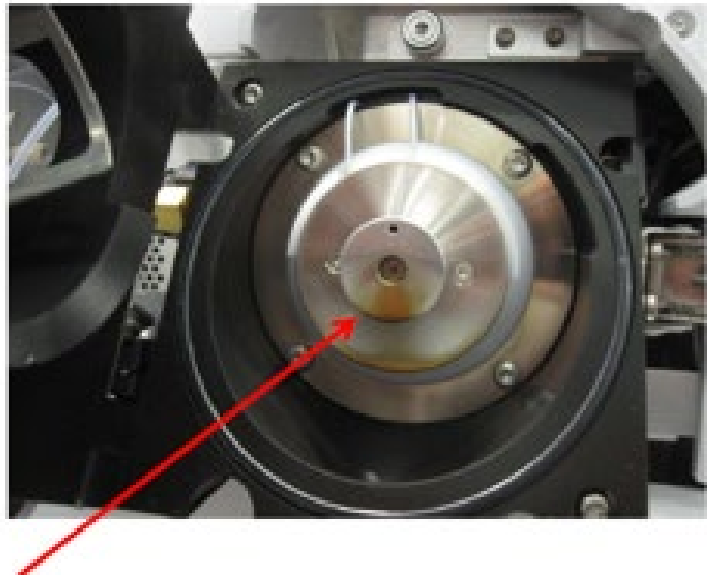


Image shows the build up on the ESI-MS inlet after 3000X urine dilute and shoot injections.

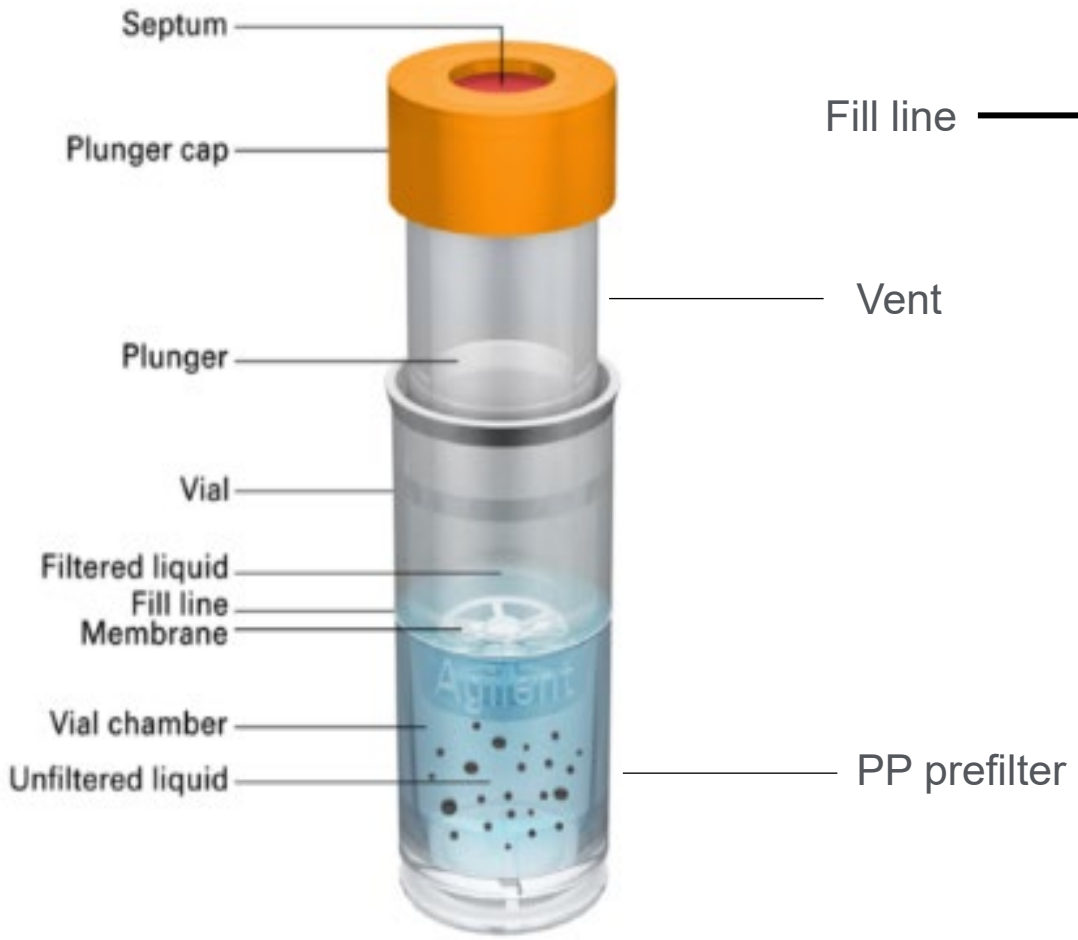
Filtration

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			♦	♦	♦	♦

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

See appendix for solvent compatibility poster request

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

Filtration – Targeted Filtration

Captiva EMR-Lipid

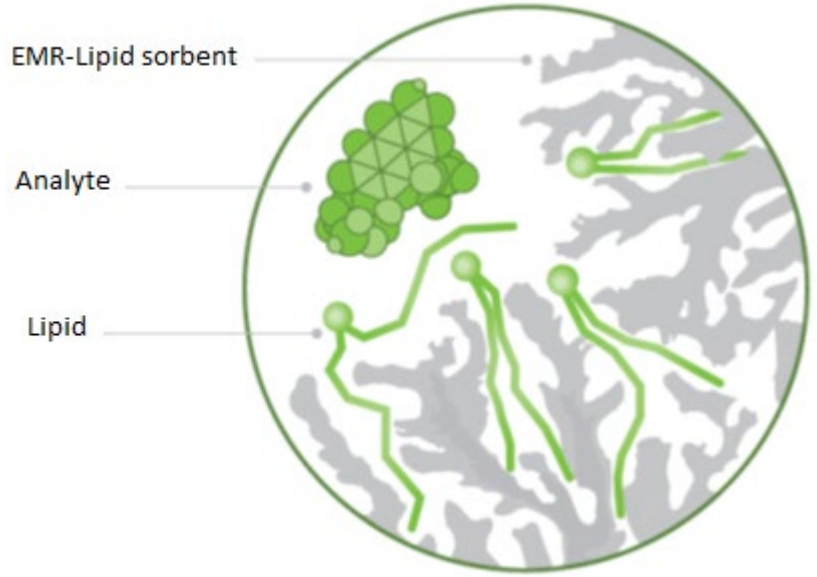
- One of the newest Agilent sample cleanup products with a 2-in-1 benefit of removing proteins and lipids.
- It reduces ion suppression, increases analyte sensitivity, improves peak shape, and extends the lifetime of your analytical column.
- Simple pass-through format, 96-well plate, 1 mL, 3 mL, and 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

Filtration – Targeted Filtration

Captiva EMR-Lipid

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

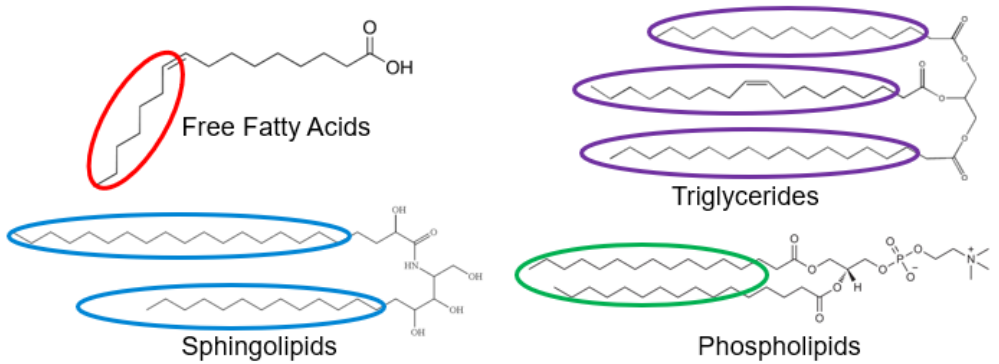
- Size exclusion – Unbranched hydrocarbon chains (lipids) enter the sorbent; bulky analytes do not
- Sorbent chemistry – Lipid chains that enter the sorbent are trapped by hydrophobic interactions



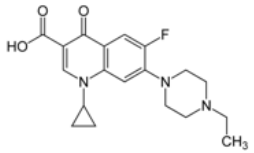
Captiva EMR-Lipid

Selective removal of lipids

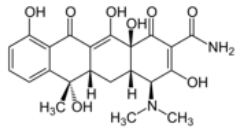
Removes lipids



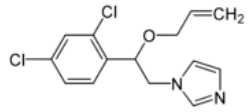
Does not remove target analytes



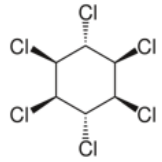
Fluoroquinolones



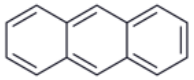
Tetracyclines



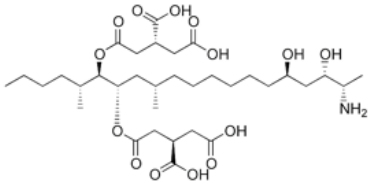
Imidazole pesticides



Organochlorine Pesticides



PAHs

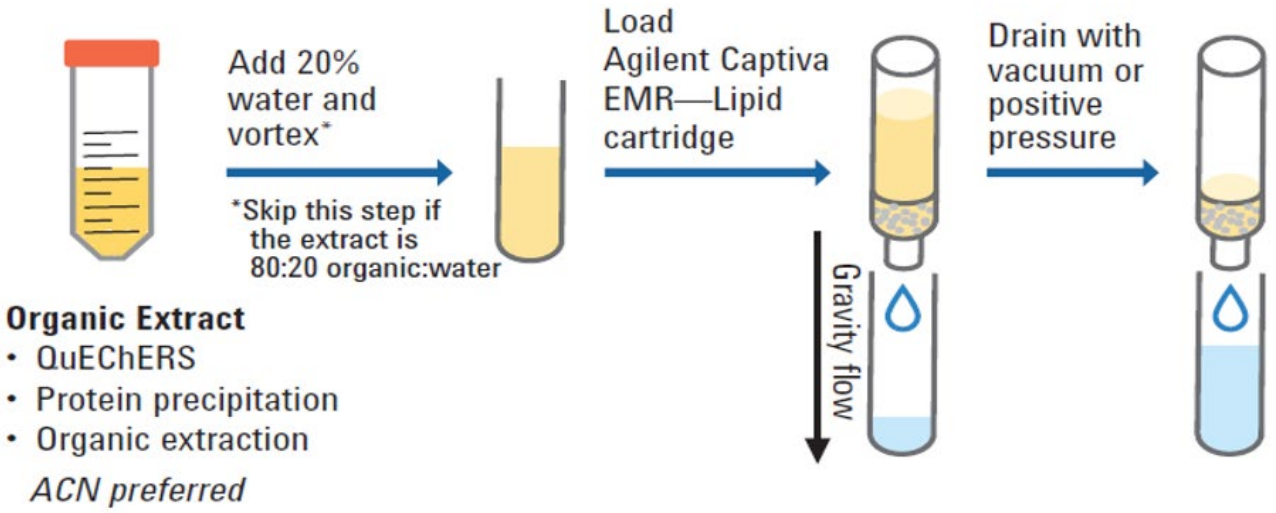


Fumonisin B2

Captiva EMR-Lipid

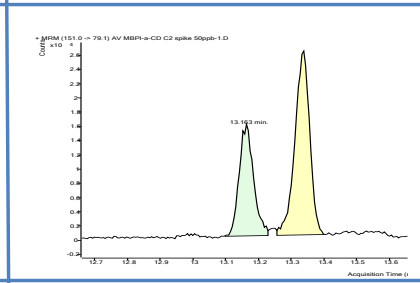
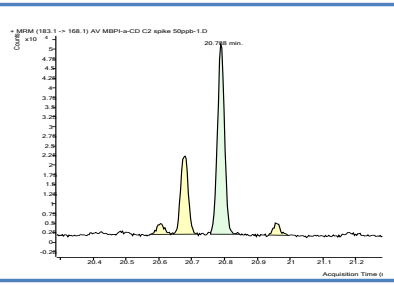
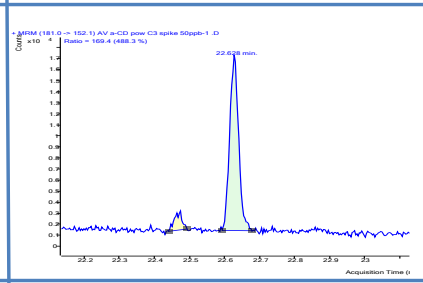
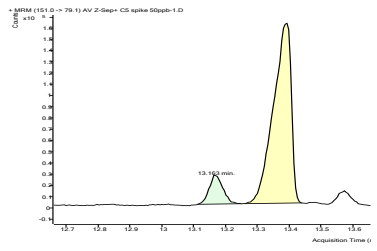
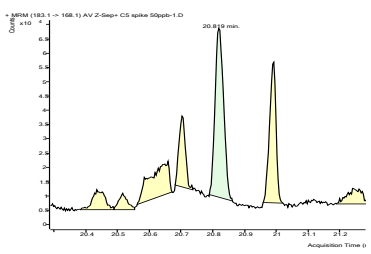
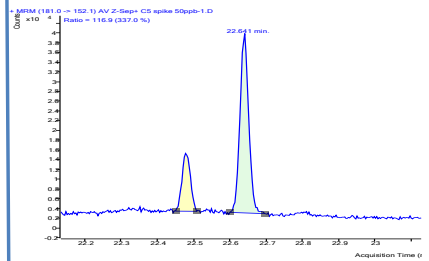
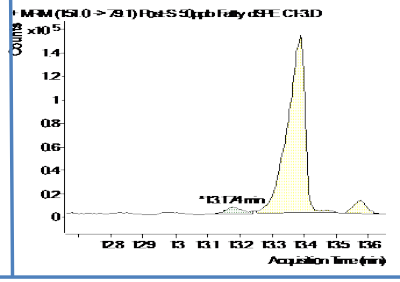
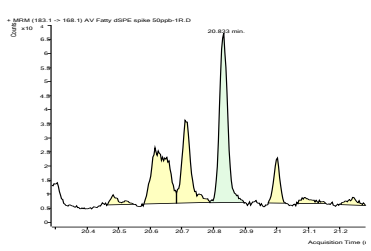
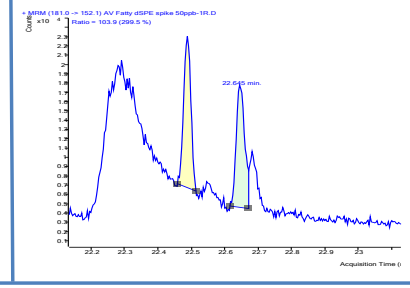
General protocol for food and food products using 3 mL and 6 mL cartridges

Operating instructions



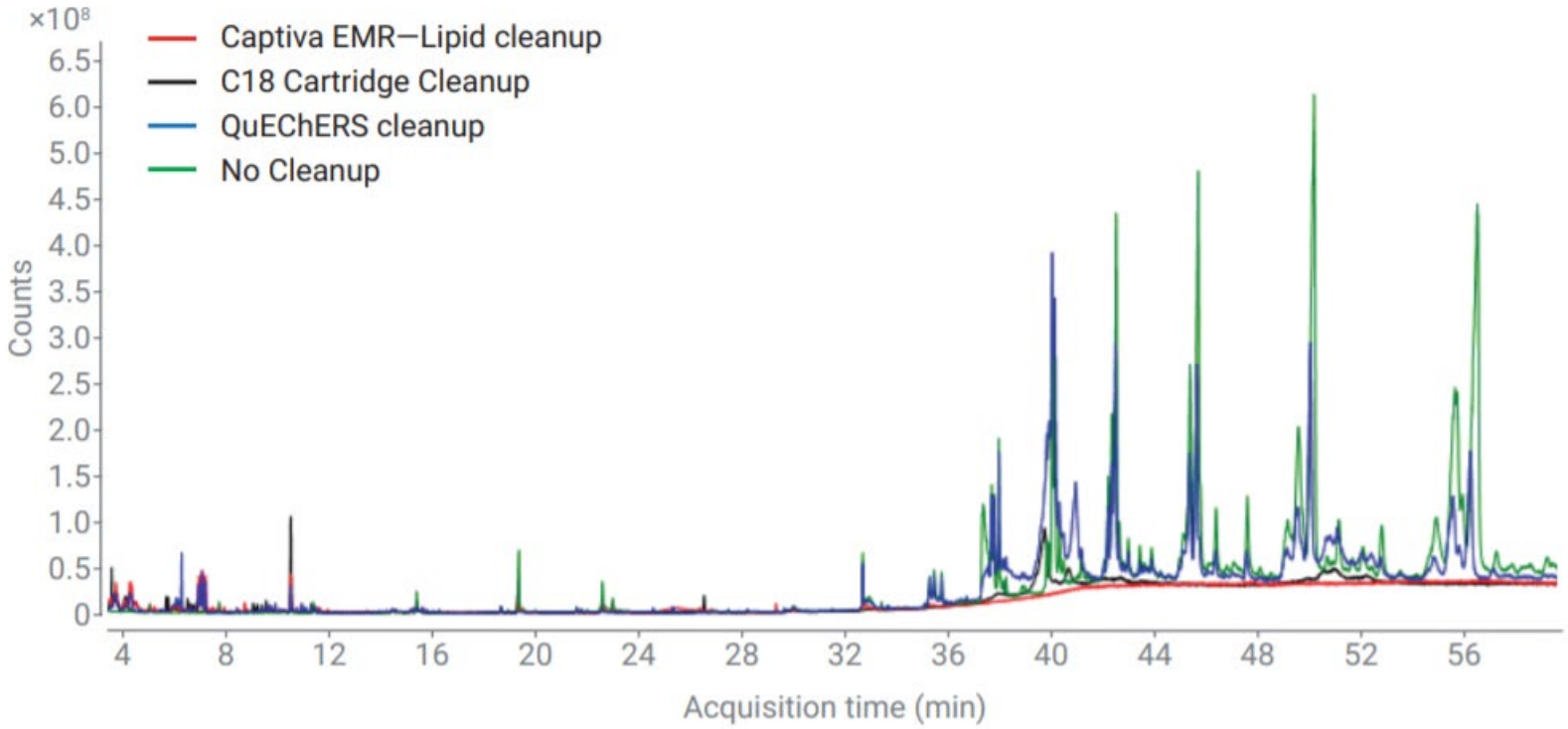
[Captiva EMR-Lipid method guide for 3 mL and 6 mL cartridges]

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

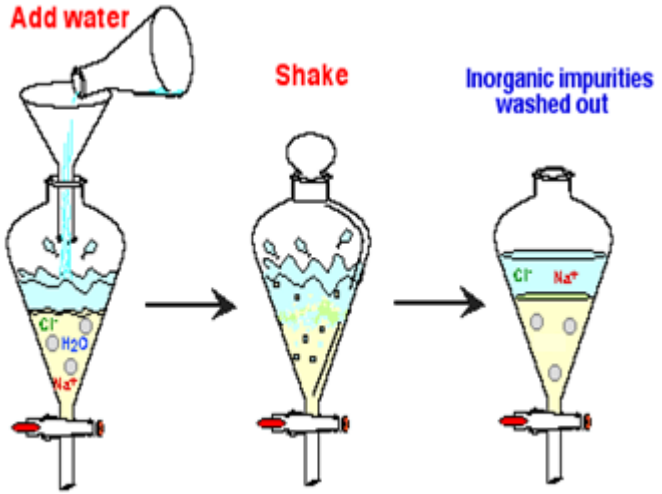


Application Note Examples

- Determination of 14 Polycyclic Aromatic Hydrocarbon Compounds in Edible Oil (5994-1483EN)
- Determination of UV Filters in Sunscreens Using Agilent Captiva EMR-Lipid Cleanup by HPLC (5994-1611EN)
- A Fast Sample Preparation Workflow for Veterinary Drugs Analysis in Salmon (5994-1124EN)
- Analysis of Nitroimidazoles in Egg Using Agilent Captiva EMR-Lipid and LC/MS/MS (5994-0641EN)
- Mycotoxin Analysis in Peanut Butter Using Captiva EMR-Lipid Cleanup and LC/MS/MS (5994-0366EN)

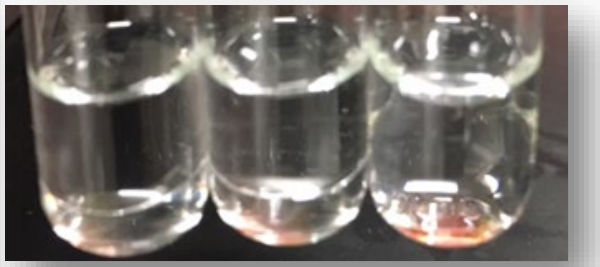
Liquid/Liquid Extraction (LLE)

- LLE has been successfully used as a method of sample preparation for many years.
- It separates the more organic solvent soluble compounds from the more water-soluble compounds using water immiscible organic solvents.
- It can remove many interfering substances like salts.
- Modulating pH can selectively extract or eliminate specific compound types.



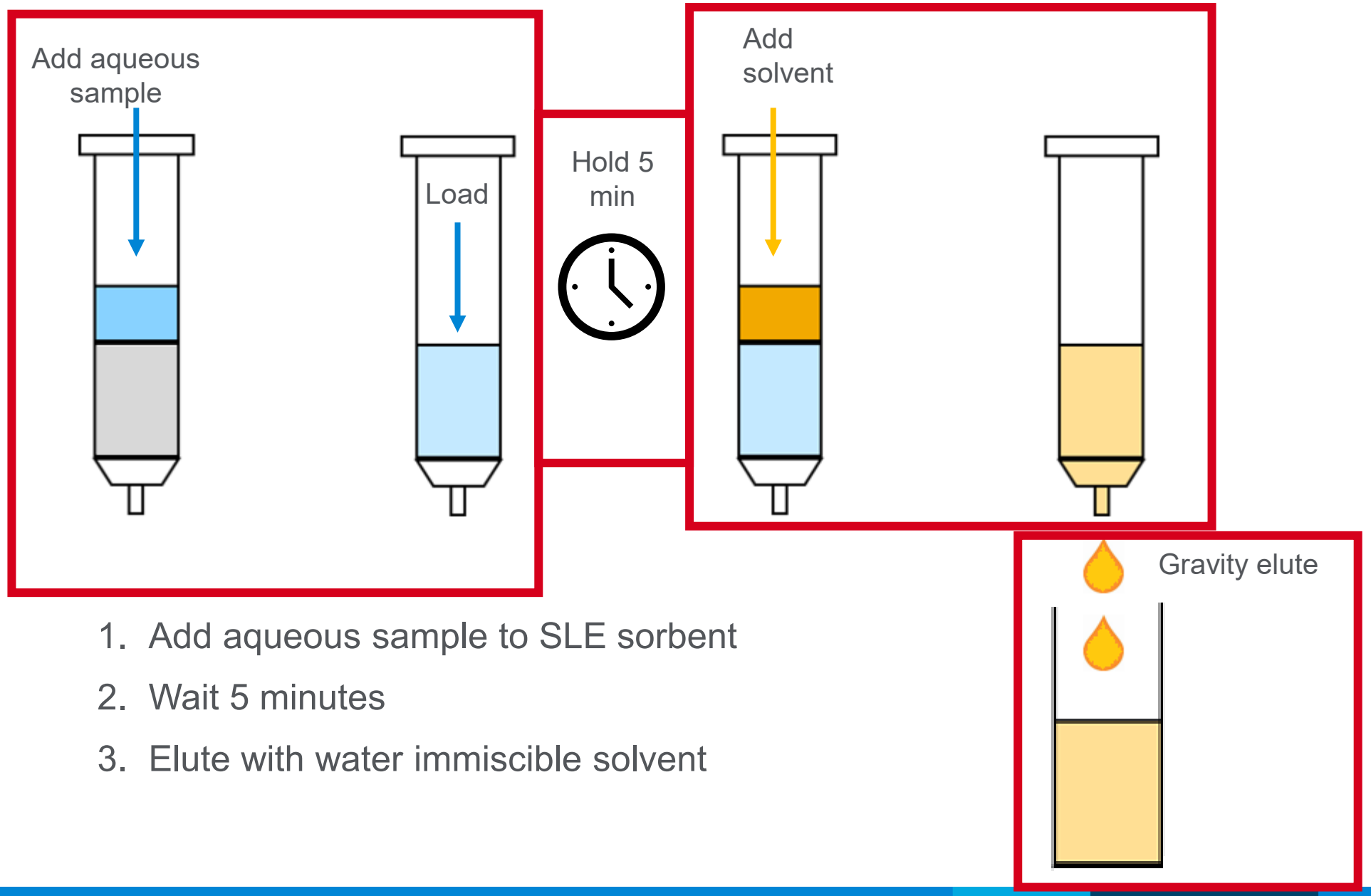
Drawbacks of Liquid-Liquid Extraction

- 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



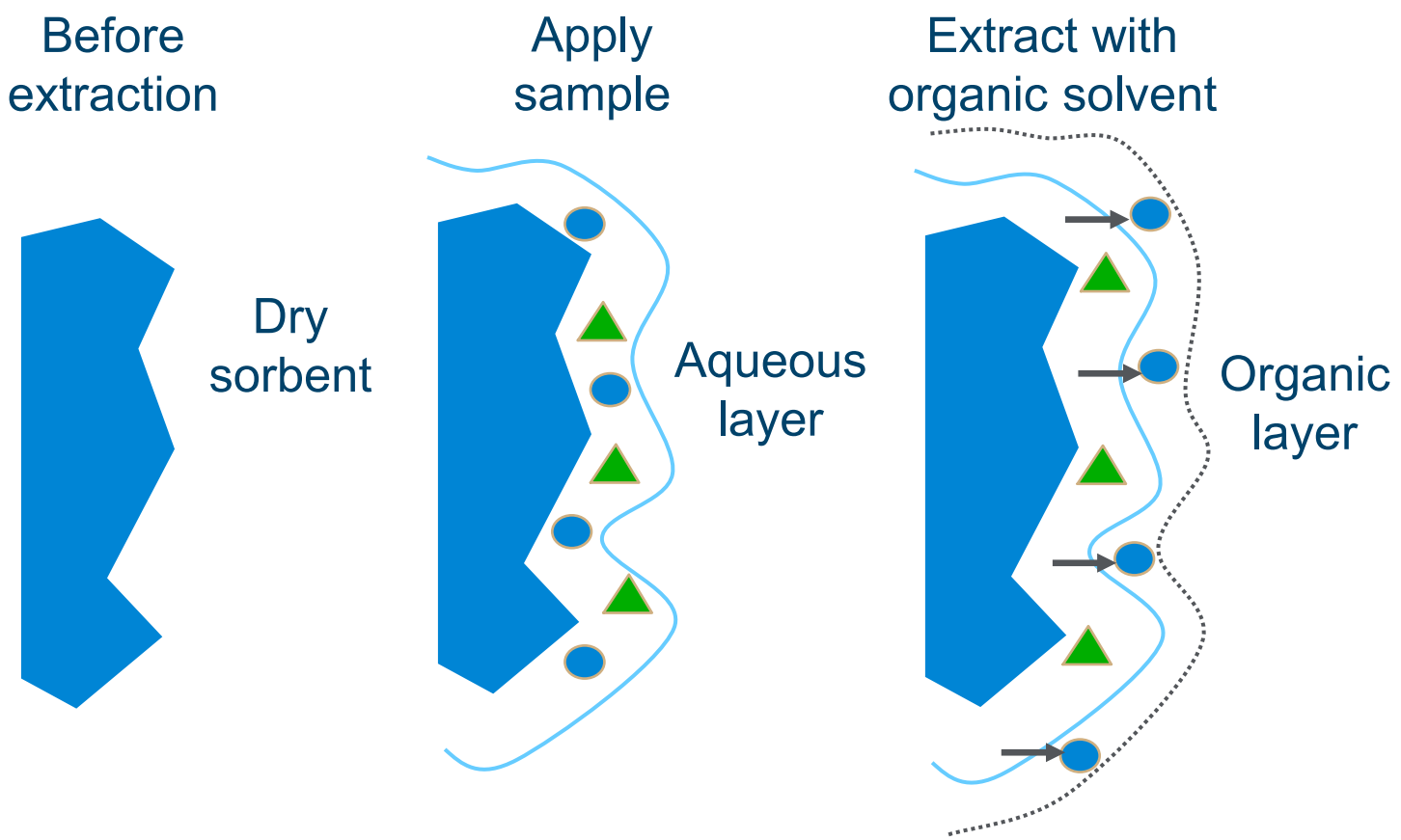
How many of these problems can be fixed with Solid Supported Liquid Extraction?

How Does SLE Work?



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

Supported Liquid Extraction (SLE)



- A thin layer of aqueous sample forms on the surface of SLE sorbent.
- When the organic solvent passes through the SLE bed, analytes are extracted under the same principles as LLE.
- Increased contact area between the two phases allows efficient extraction without mixing.

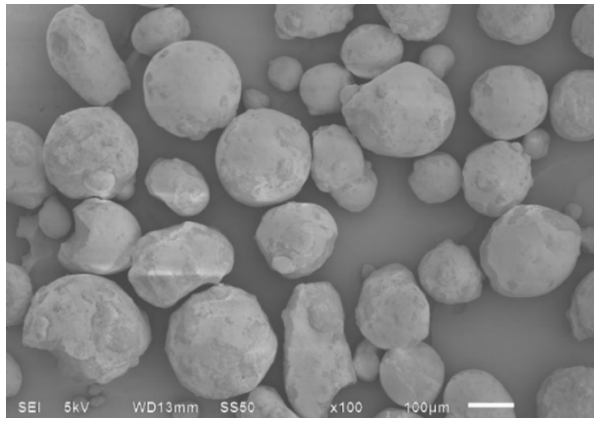
What is SLE Sorbent?

- 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 in 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

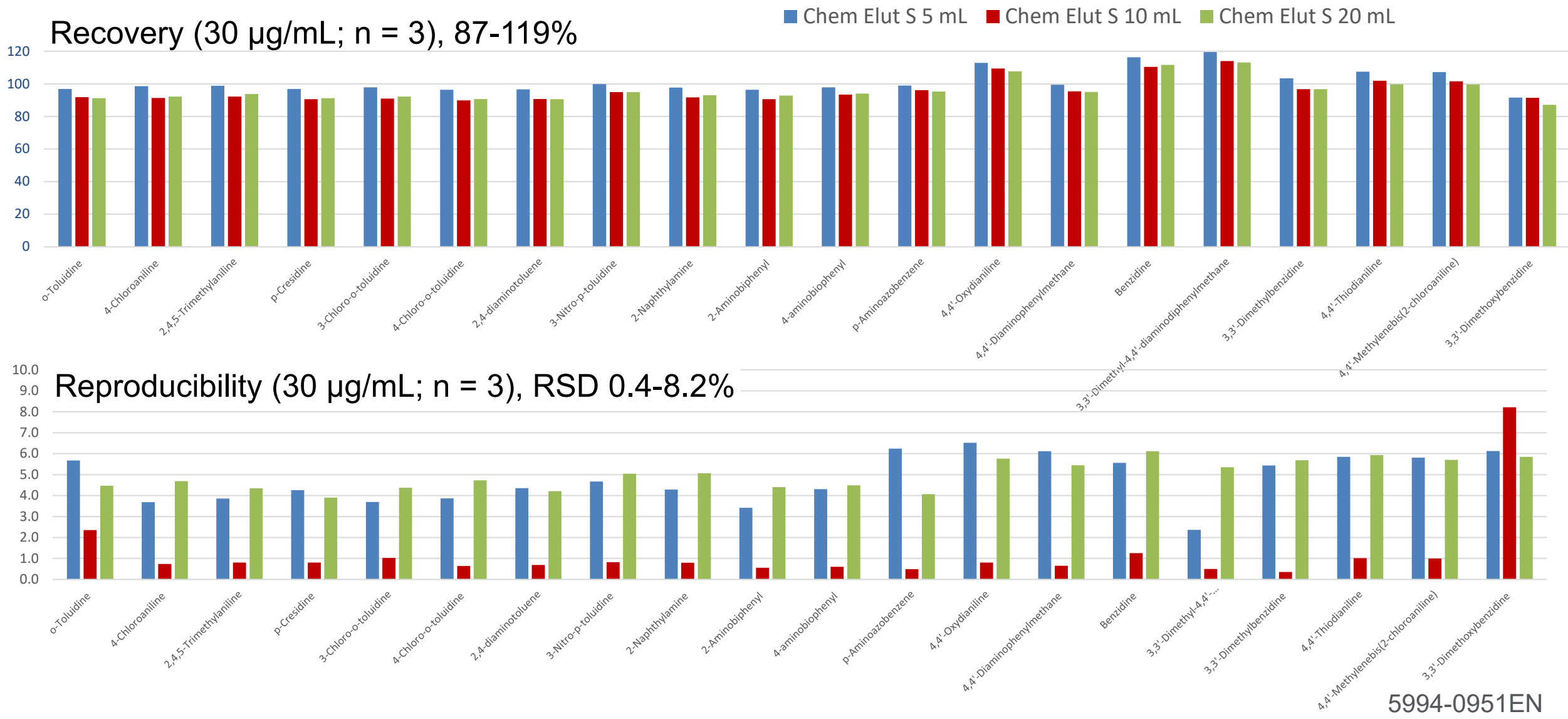
Cartridges for sample volumes 0.2 – 20 mL

Bulk Chem Elut S
1 kg and 4 kg

96-well plate for sample volume 200 µL and 400 µL

Chem Elut S – 15 Minute Hold Time

Large scale format comparison with aromatic amines using GC

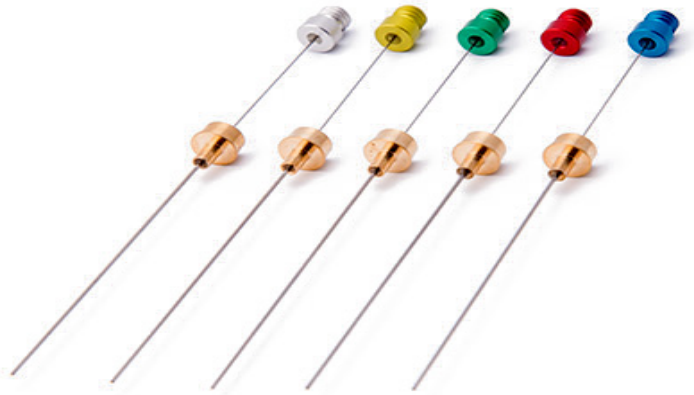


5994-0951EN

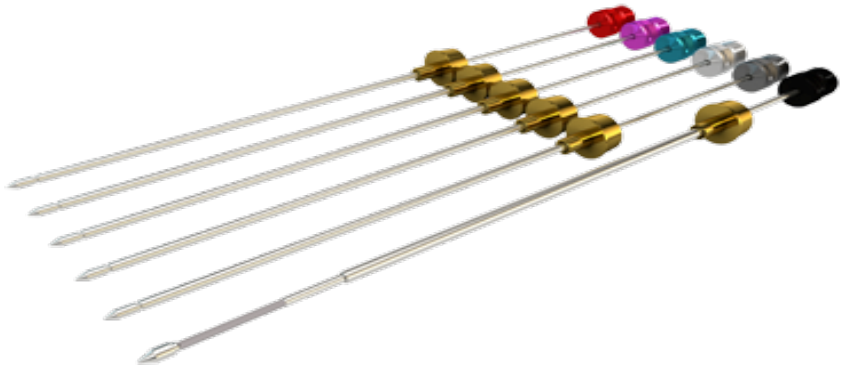
SPME Fibers and SPME Arrows from Agilent

Solid Phase Microextraction (SPME)

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



SPME fibers

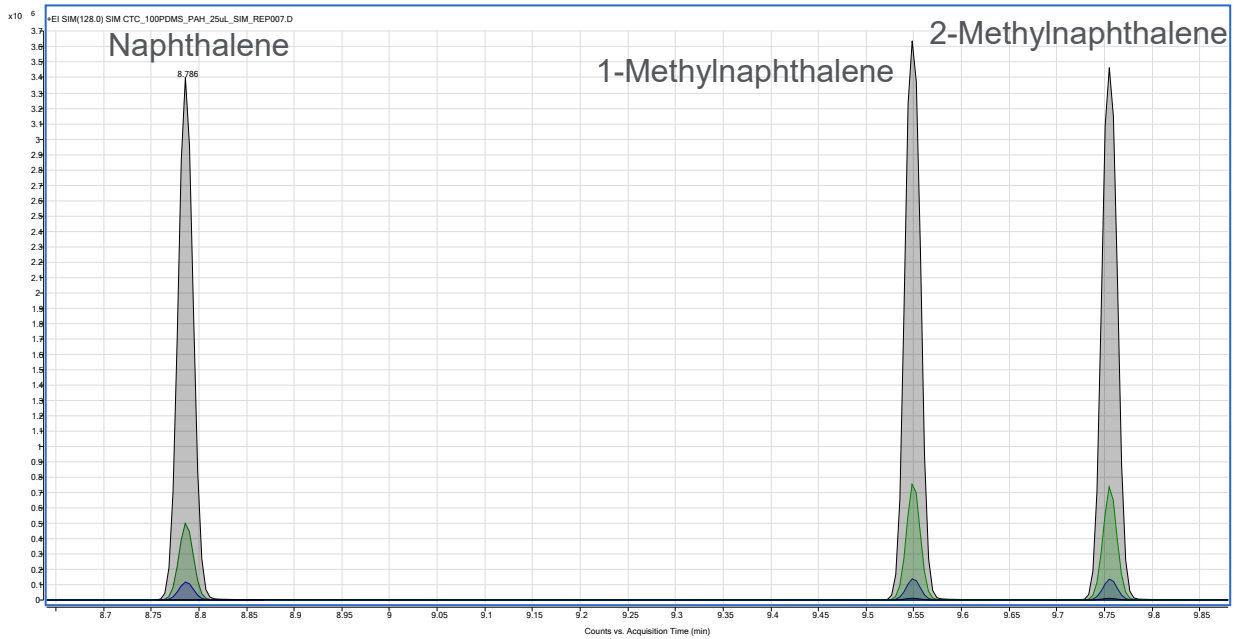


SPME Arrows

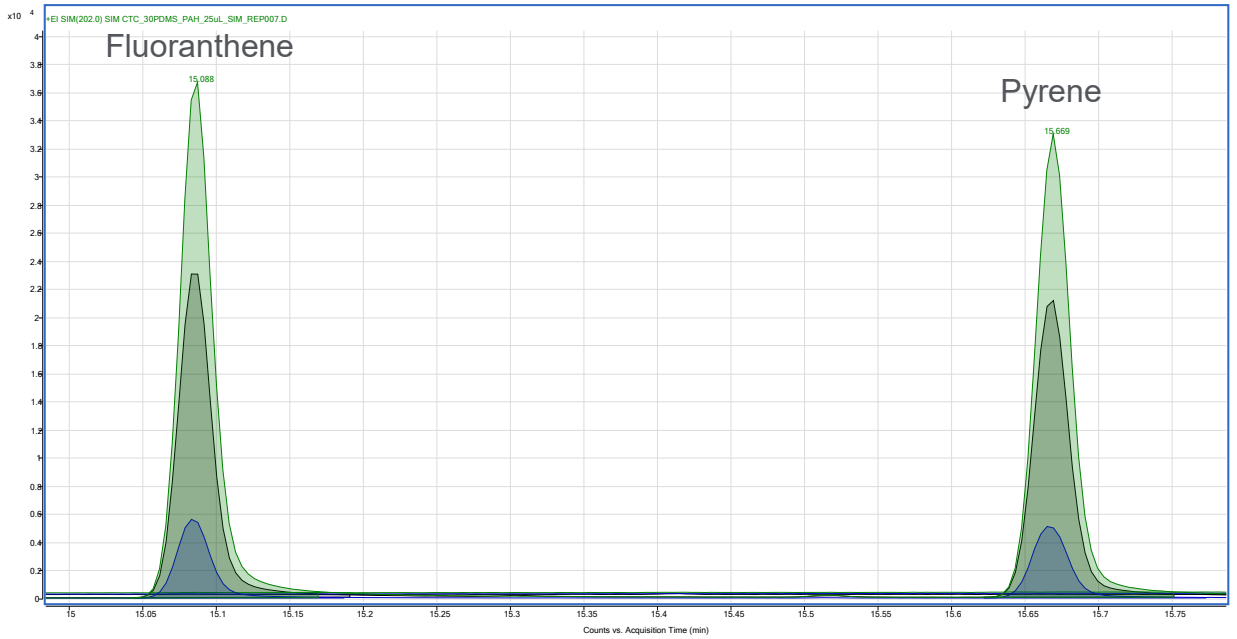
Examination of Lower Molecular Weight PAHs in Drinking Water Using Agilent PDMS SPME Fibers

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)

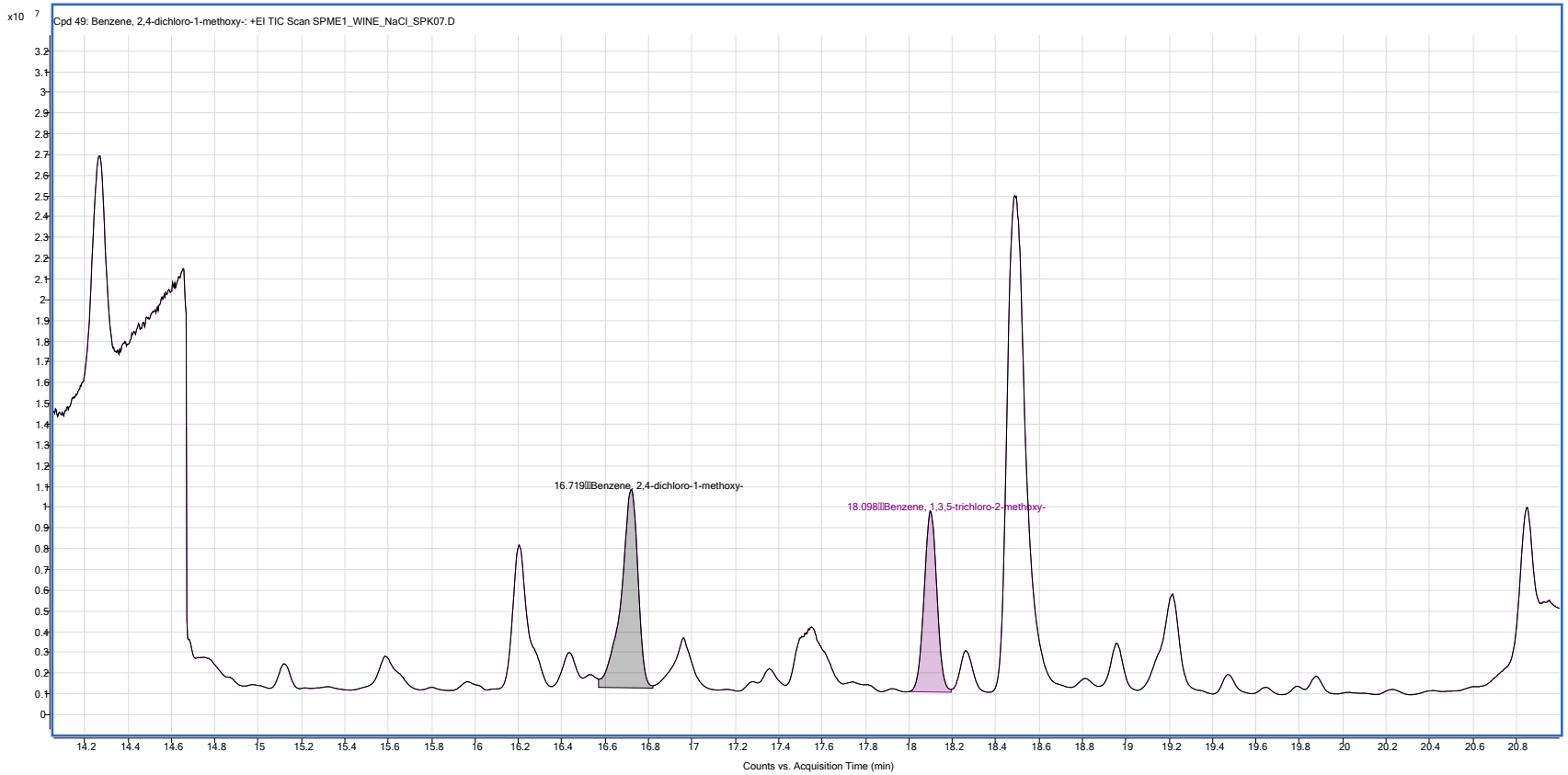


SIM chromatogram of fluoranthene and pyrene with PDMS fibers (black trace = 100 µm; green trace = 30 µm; blue trace = 7 µm)

SPME-GC/MS of 2,4,6-trichloroanisole Using Agilent DVB/PDMS SPME Fiber

To learn more, search 5994-1564EN at www.agilent.com

The main compound responsible for cork taint is 2,4,6-trichloroanisole. It is one of the most odor intense compounds known and has a distinct musty, moldy aroma.



2,4-Dichloroanisole and 2,4,6-Trichloroanisole at 25 ppb in red wine

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
- GCB: 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).

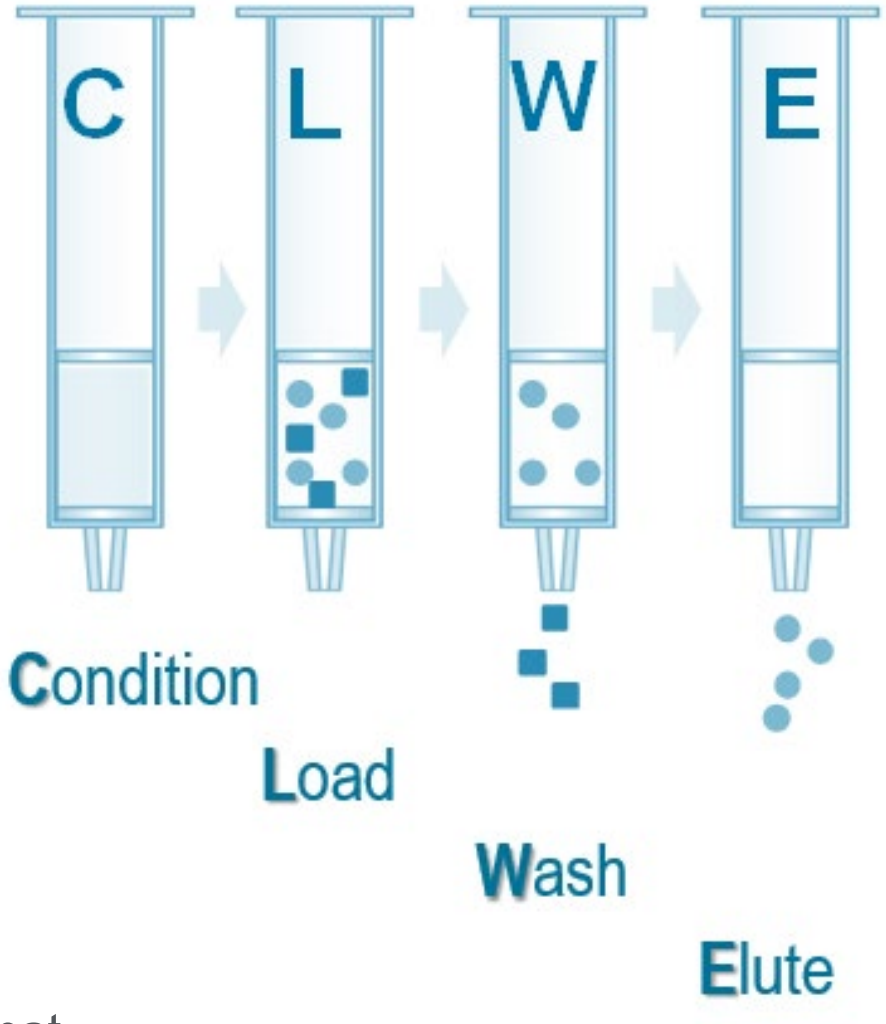
QuEChERS is a nonselective technique and does not remove **all** the matrix, just enough.

Dispersive sorbents are also available as bulk material.

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

SampliQ SPE

Multiple phases

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

Manifolds for Processing Cartridges and 96-Well Plates

Captiva vacuum collar

Vac Elut 20 vacuum manifold

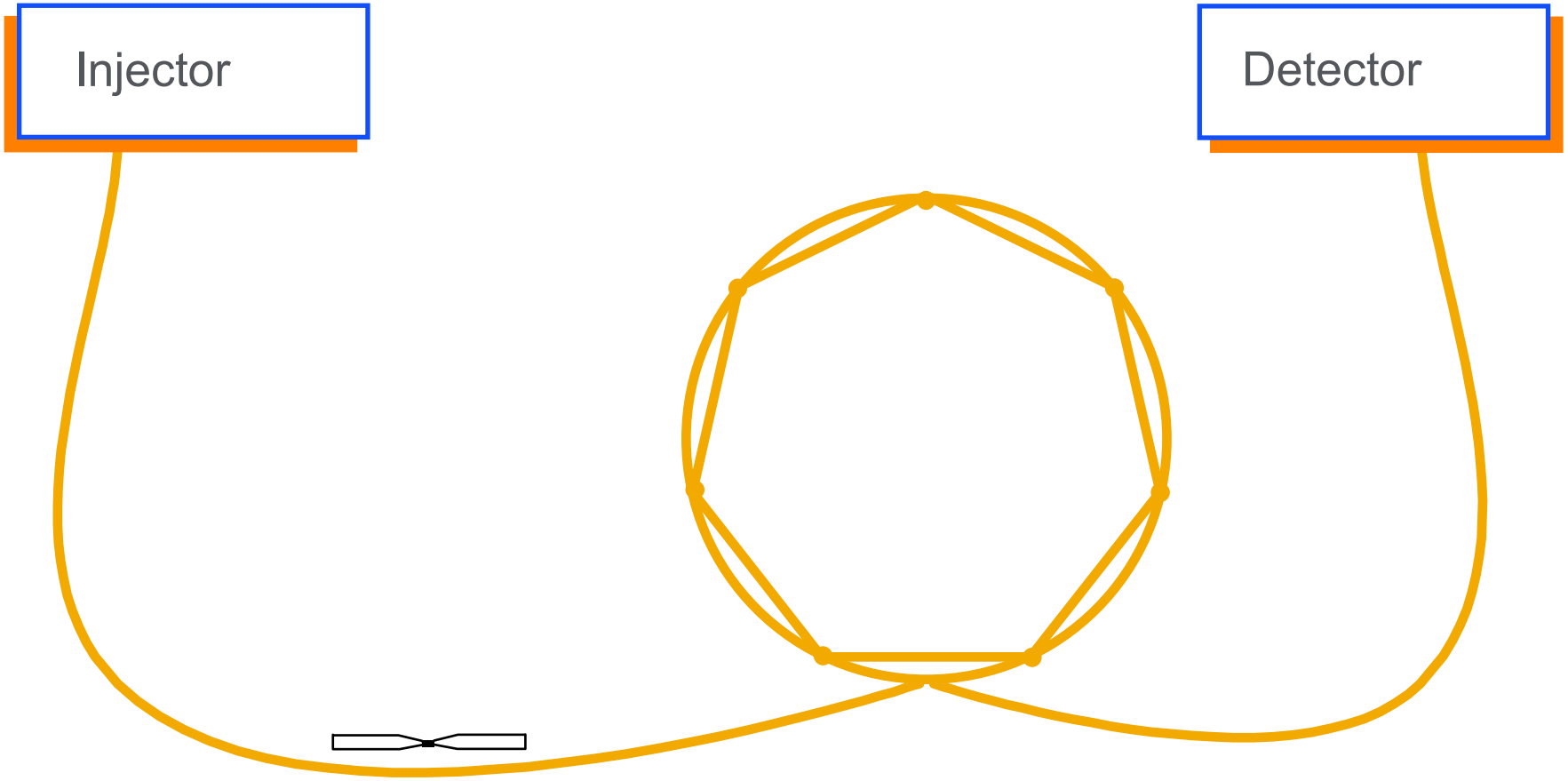
Vac Elut 12 vacuum manifold

SPS 24 vacuum manifold

96 well plate vacuum manifold

Positive Pressure Manifolds

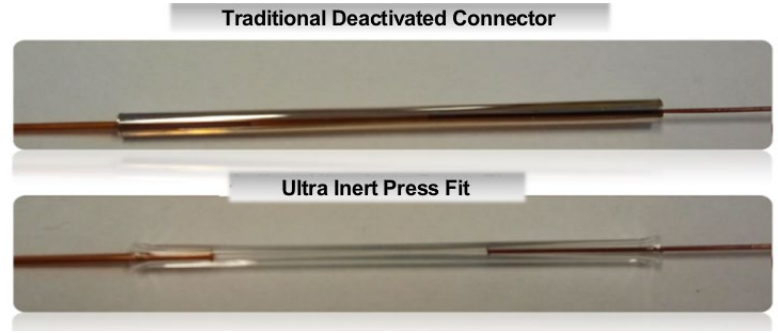
Guard Column or Retention Gap



The guard column is 3–5 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 column connectors:
  - 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



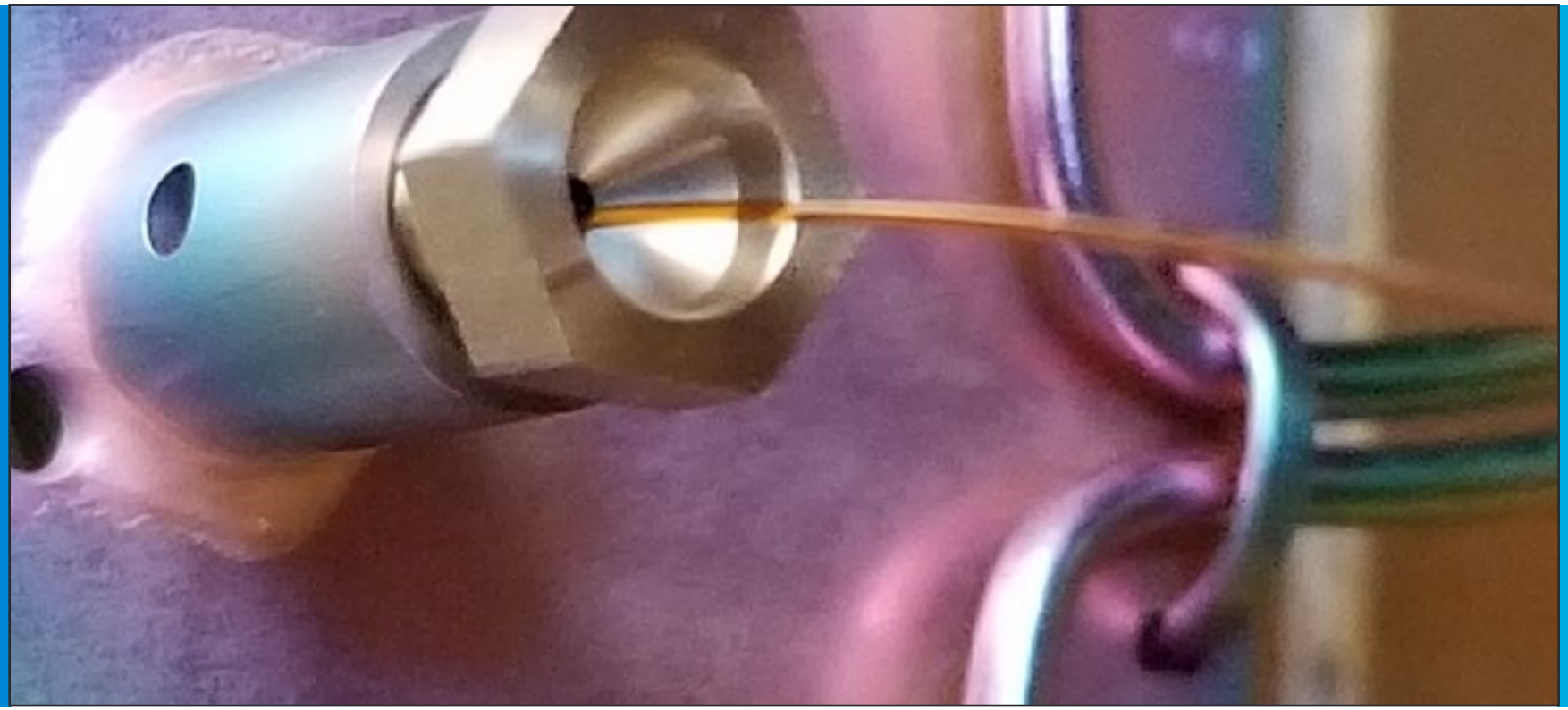
Integrated Guards - DuraGuard

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

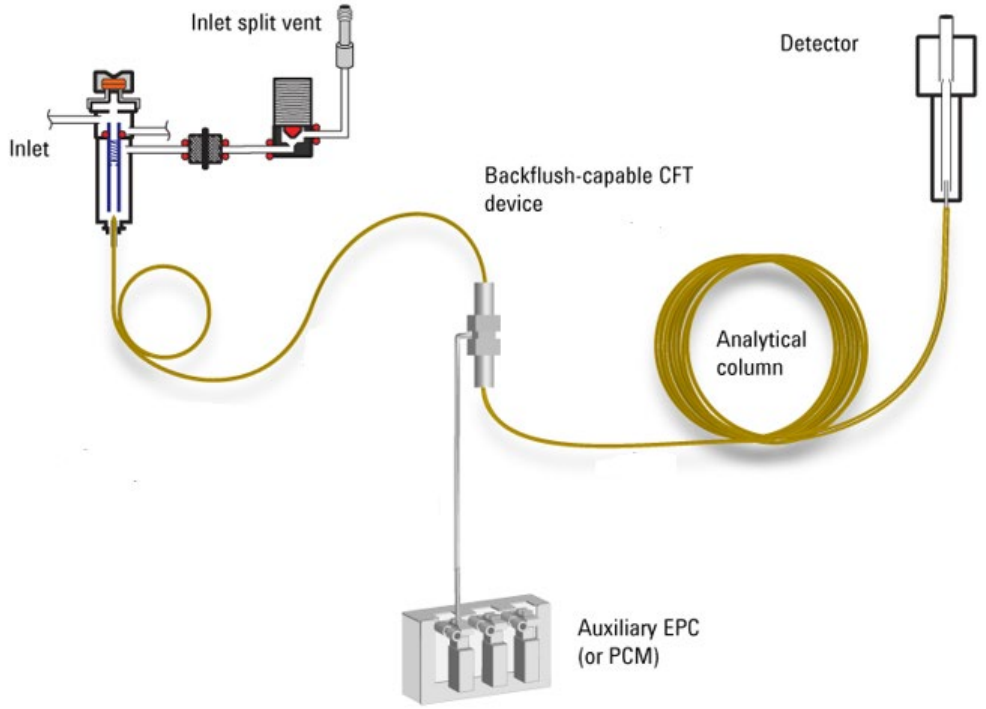
DuraGuard

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



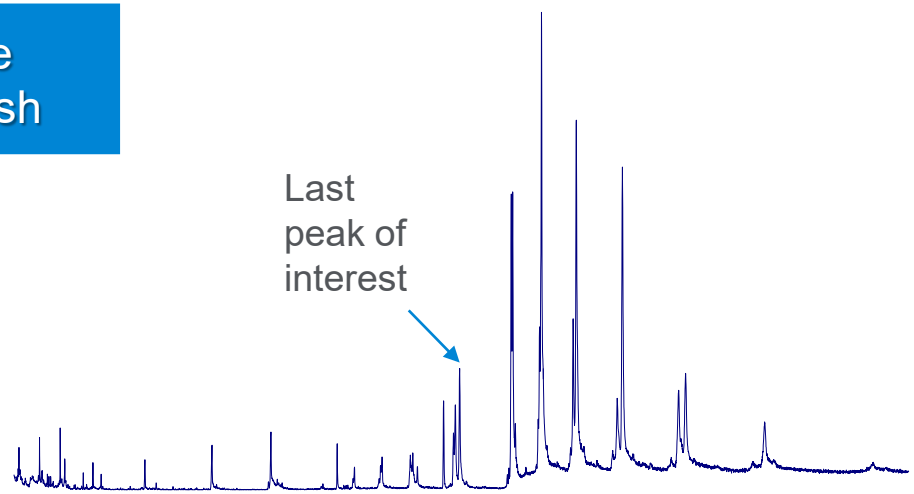
What is GC Capillary Column Backflush?



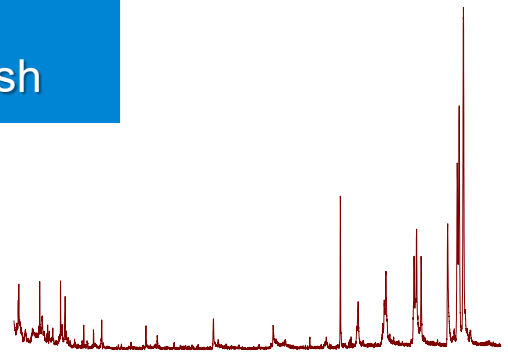
Backflush is a technique that is used to chromatographically remove mainly high boiling compounds from samples.

Backflush is performed either at the end of, or during, an analytical run.

Before backflush

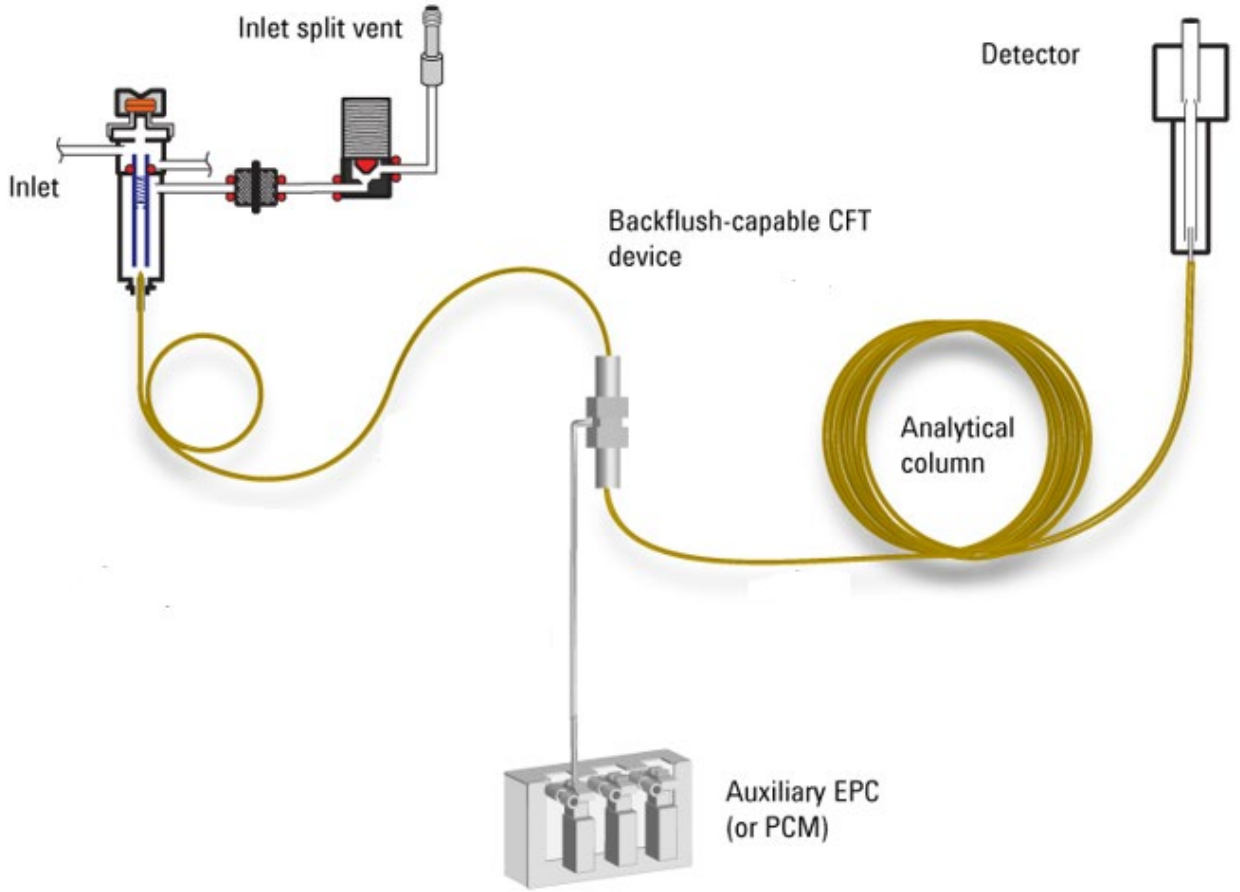


After backflush

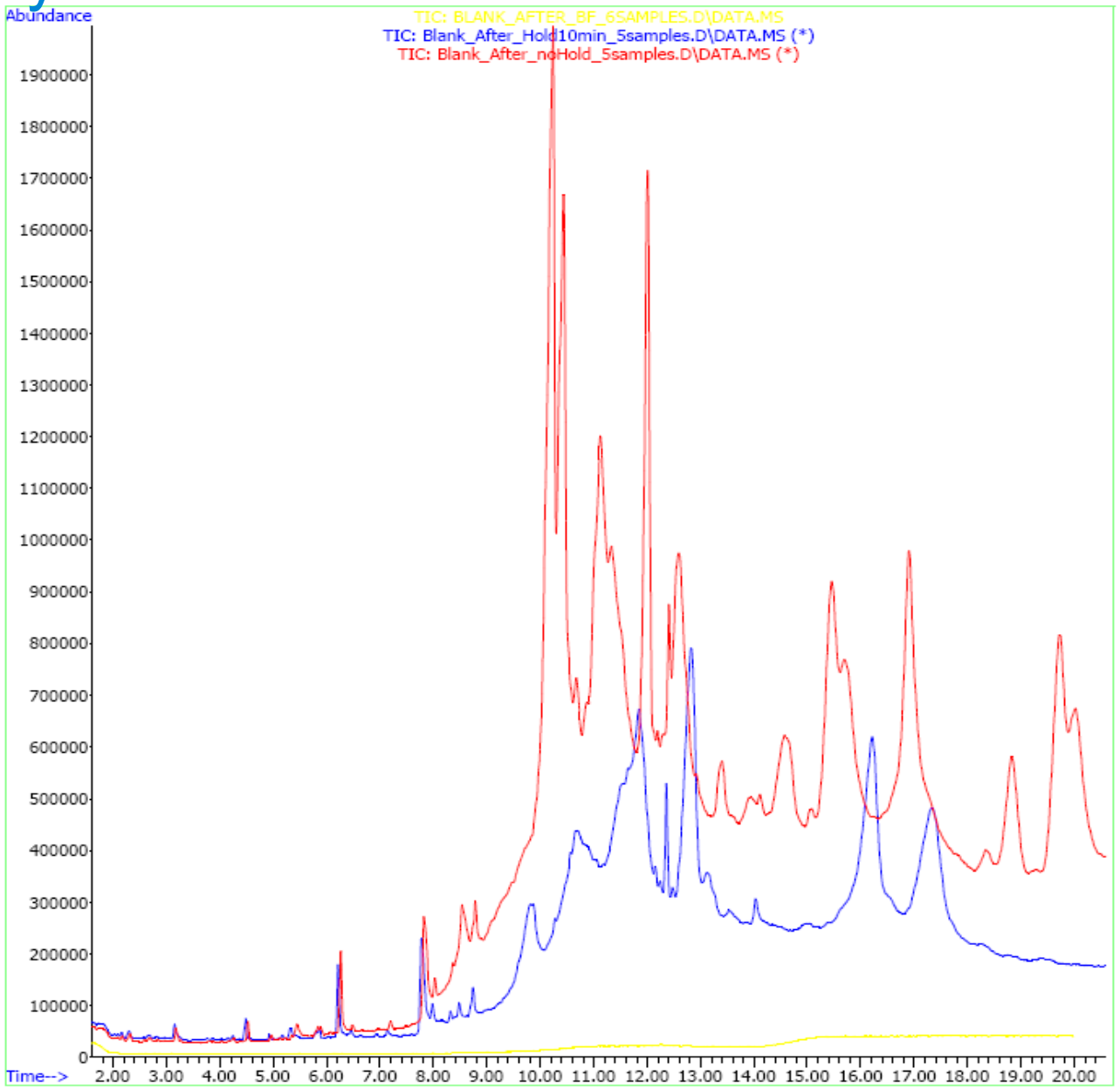


Why Use Backflush?

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



Why Use Backflush?

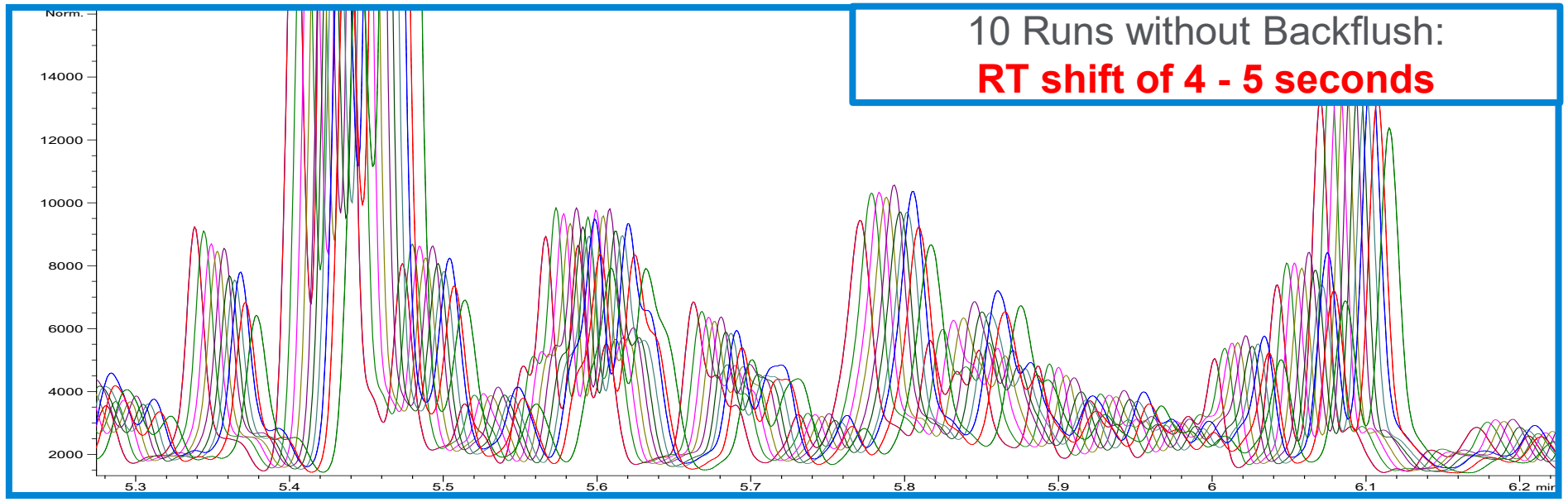


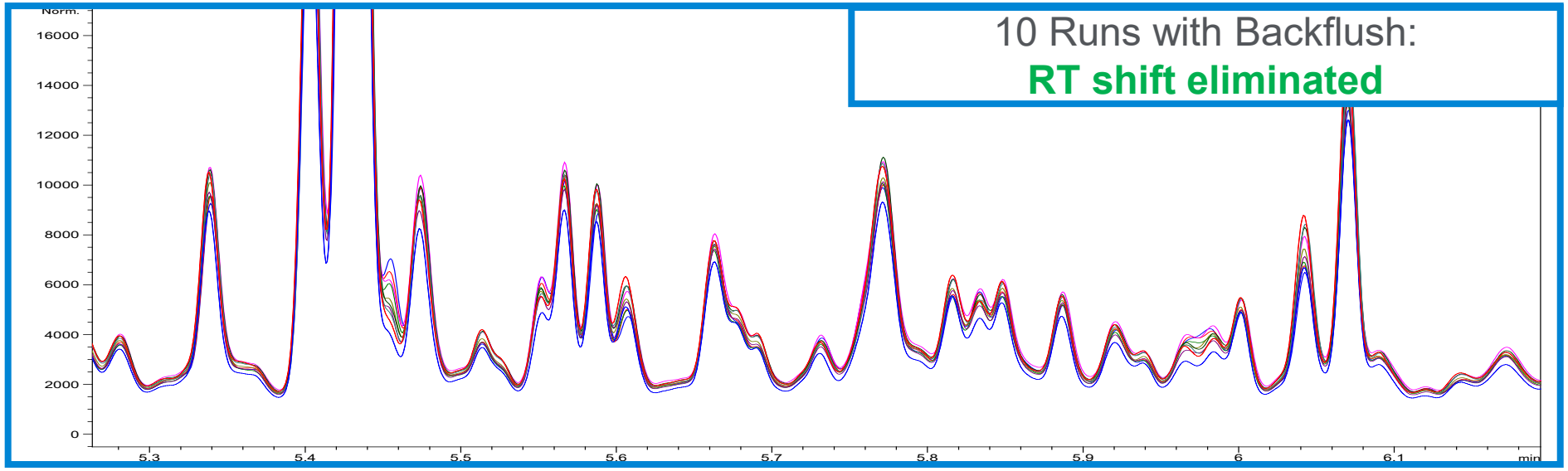
Blank after 5 injections
(normal method)

Blank after 5 injections
(with 10 minute bake at 280 C)

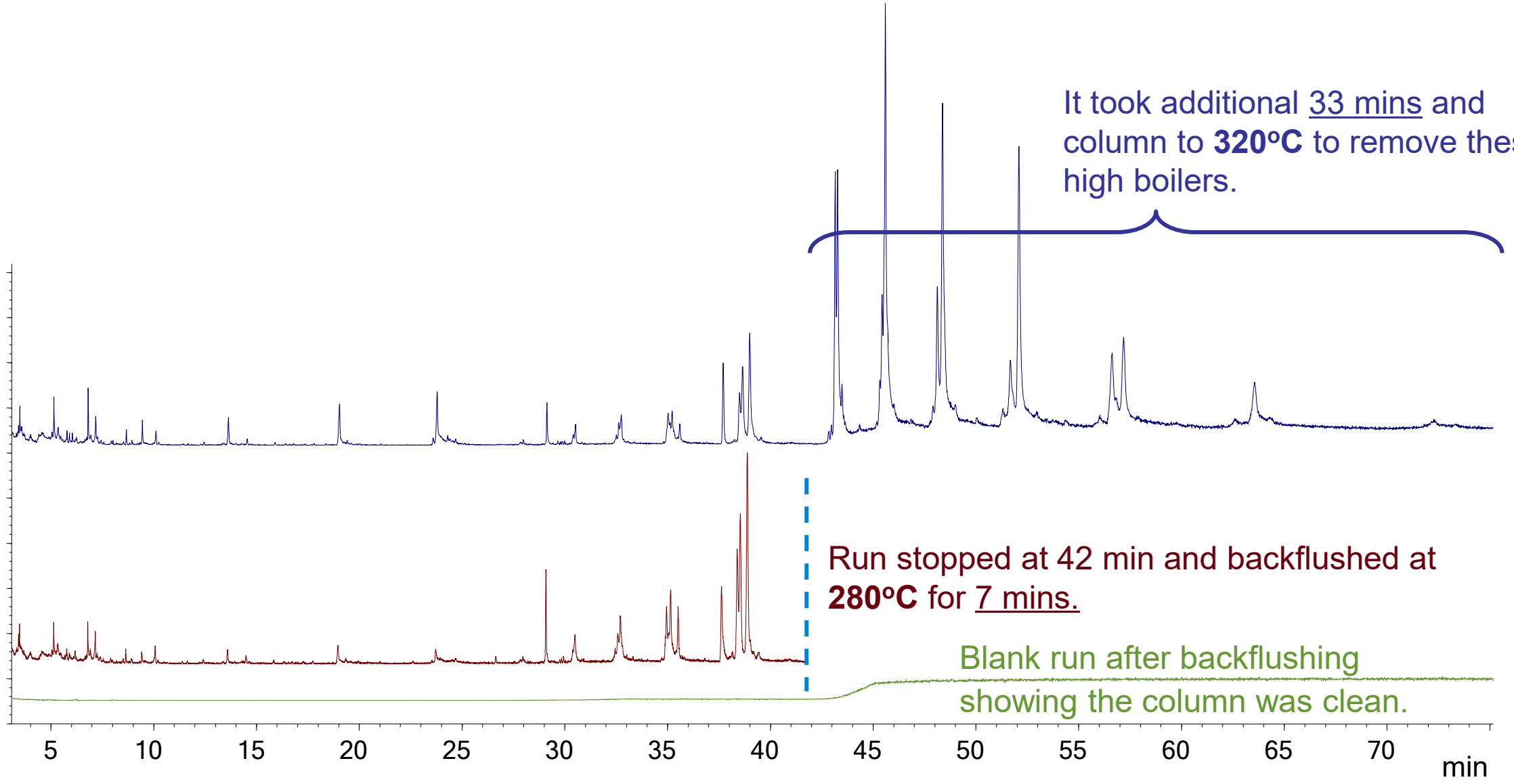
Blank after 5 injections
(2 minute backflush, 50 PSI @ 280 C)

Why Use Backflush?





Why Use Backflush?



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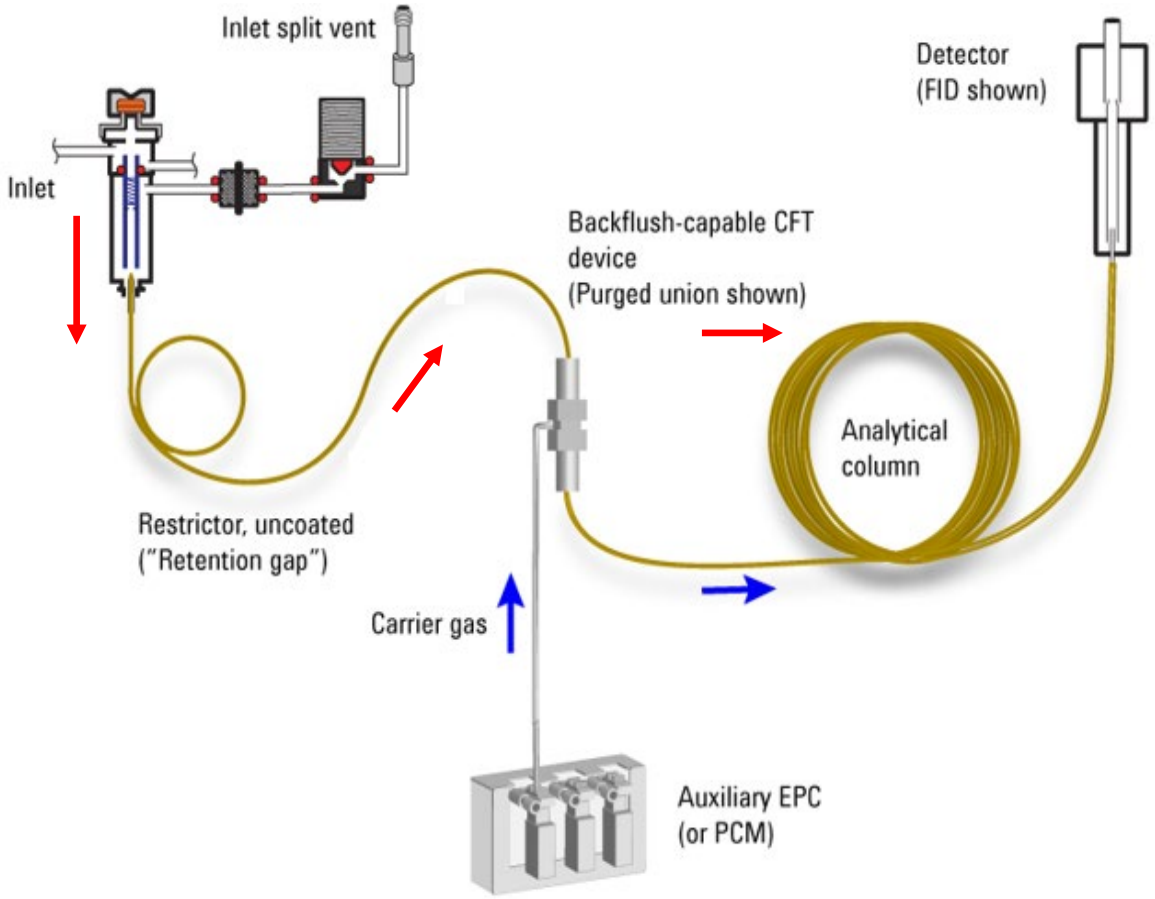
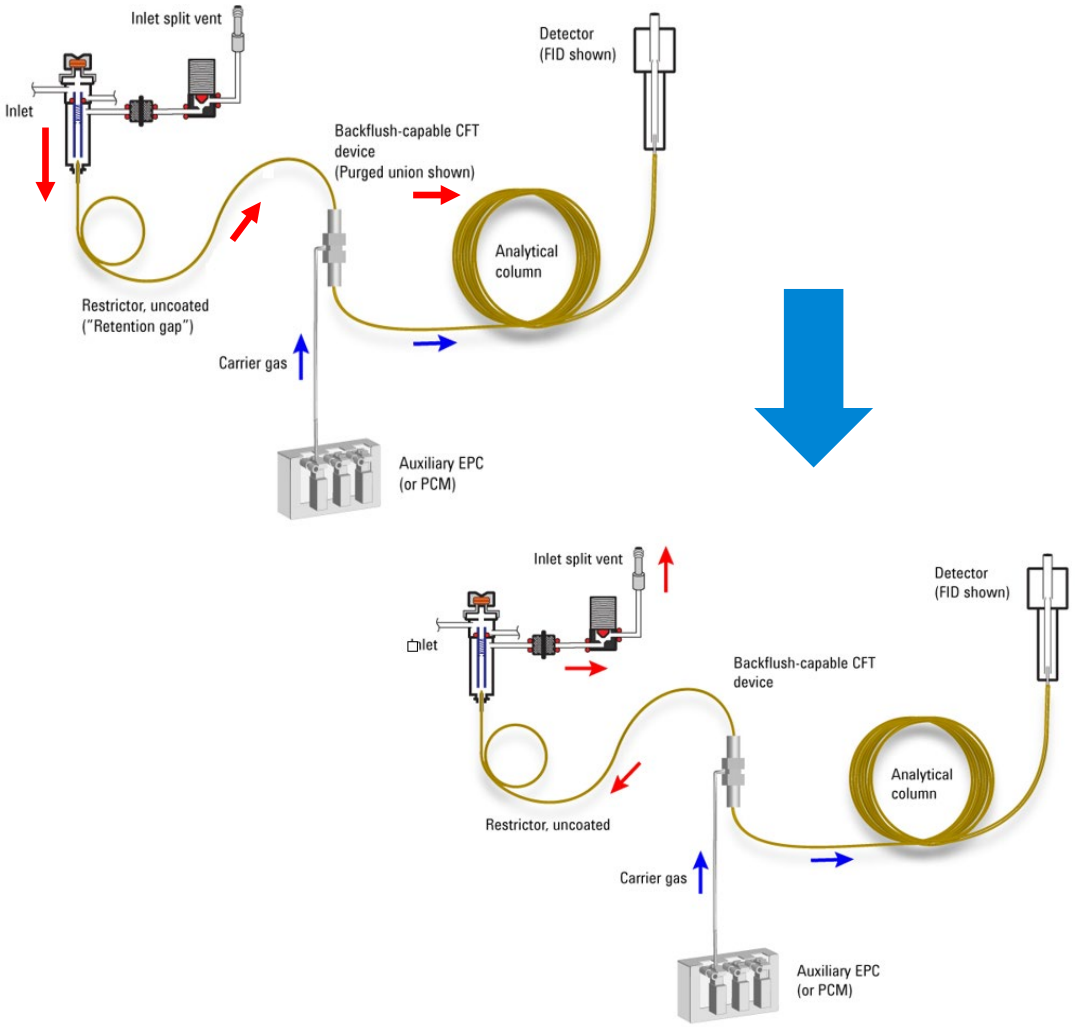
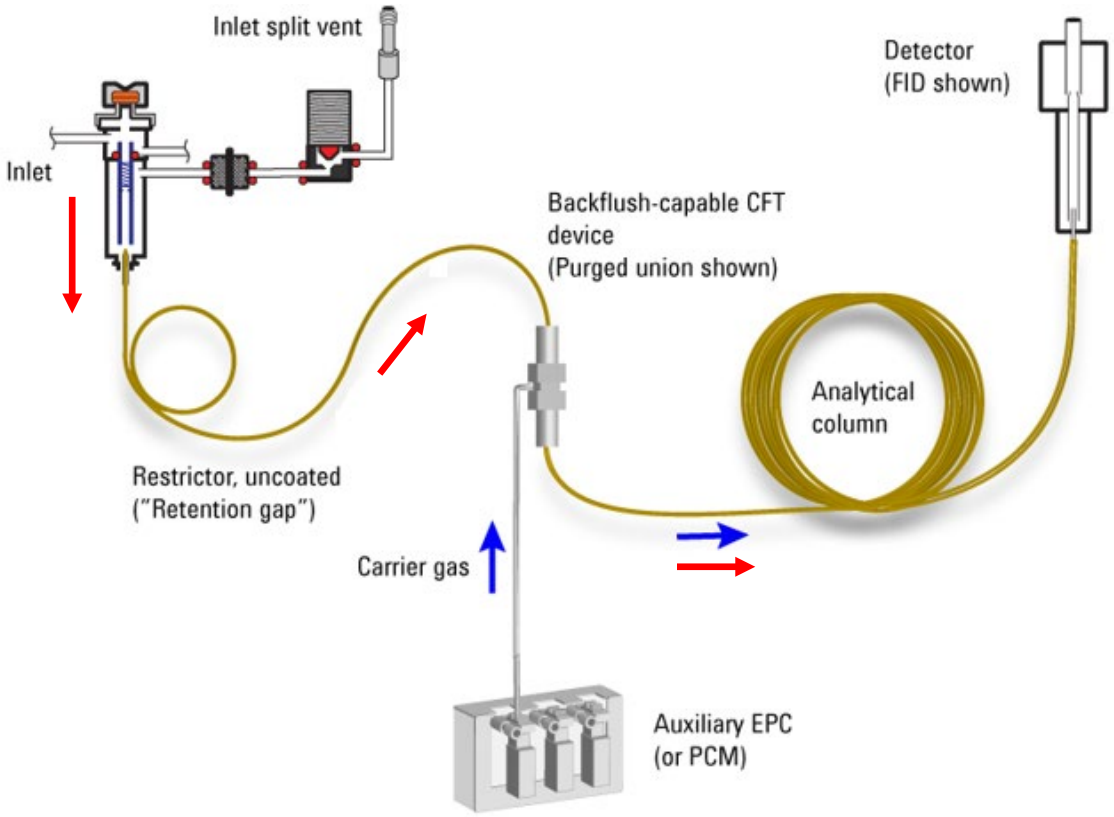


Backflush Techniques – Uncoated Precolumn: How Does it Work?



- Configure the analytical column from the EPC module to the detector. Flow rate set to that needed for peak separation.
- Configure restrictor/precolumn from inlet to EPC module. Flow must be set at least 10% lower than that of the analytical column.
- Injection performed
- Compounds travel through restrictor/precolumn
- When all compounds of interest are eluted out of the first column onto the analytical column, inlet pressure is dropped, reversing the flow through the first column. The "unwanted" compounds are backflushed out of the split vent.
- Flow is maintained through the analytical column by the AUX EPC or PCM.

Backflush Techniques – Uncoated Precolumn: How Does it Work?



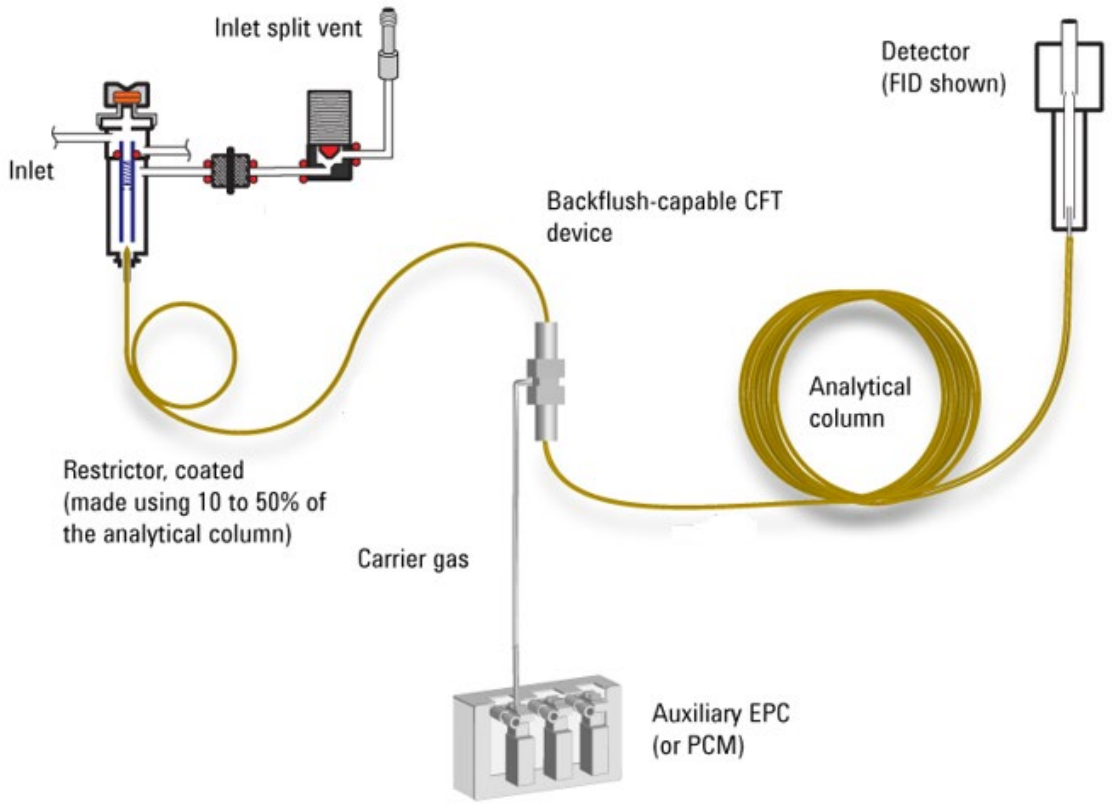
Pros:

- Contaminants do not reach column.
- Contaminants do not reach detector.
- Shorter Run times (backflush during run)
- Inexpensive retention gap.

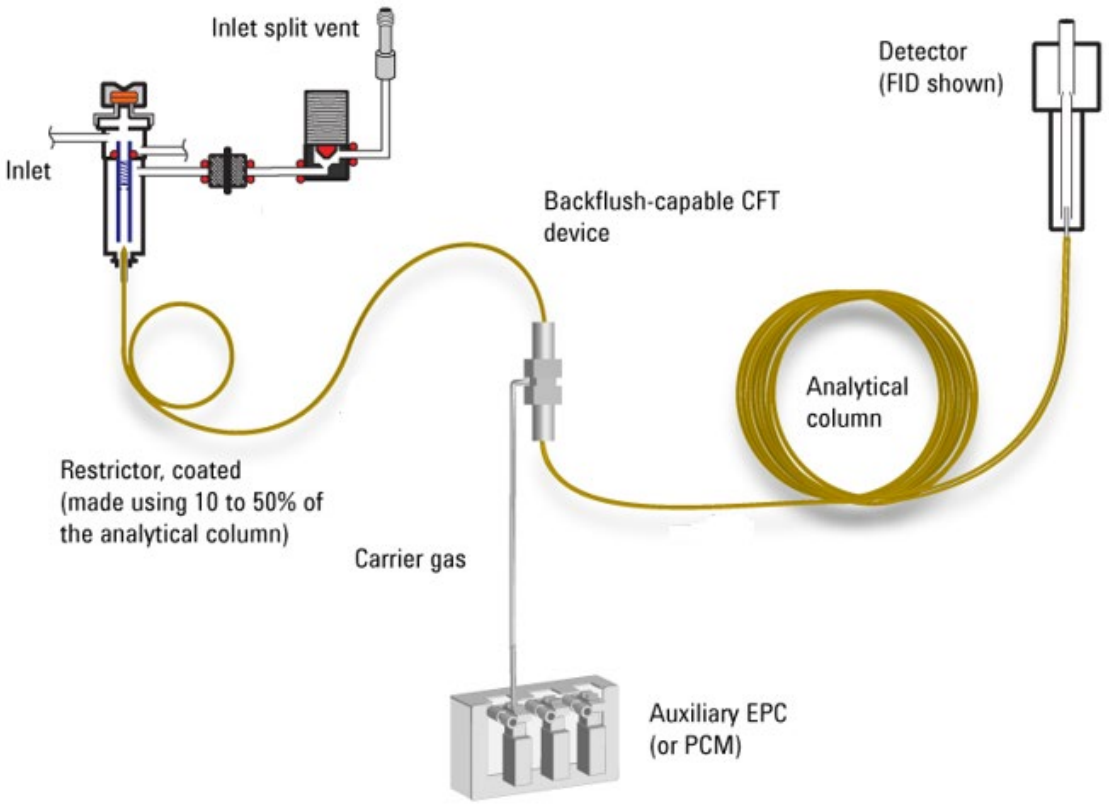
Cons:

- Requires extra hardware (EPC module/CFT device).
- Requires setup to determine when peaks elute from retention gap onto the analytical column. Use backflush wizard or trial and error injections.
- Retention gap has very little retention.

Backflush Techniques – Coated Precolumn



Backflush Techniques – Coated Precolumn



Pros:

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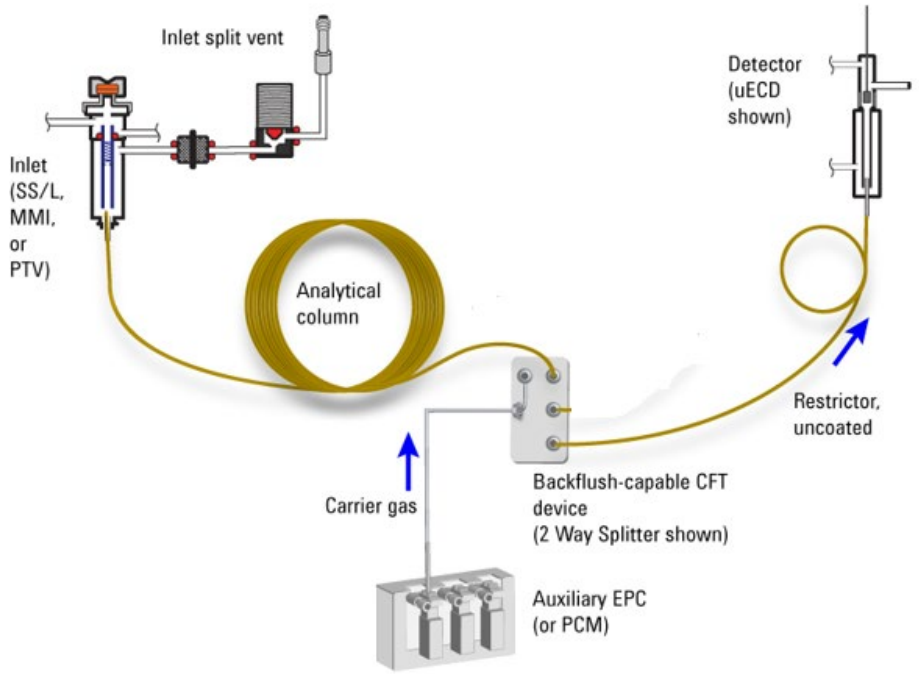
- Contaminants do not reach detector.
- Shorter Run times (backflush during run)

Cons:

- Requires extra hardware (EPC module/CFT device).
- Requires setup to determine when peaks elute from retention gap onto the analytical column. Use backflush wizard or trial and error injections.

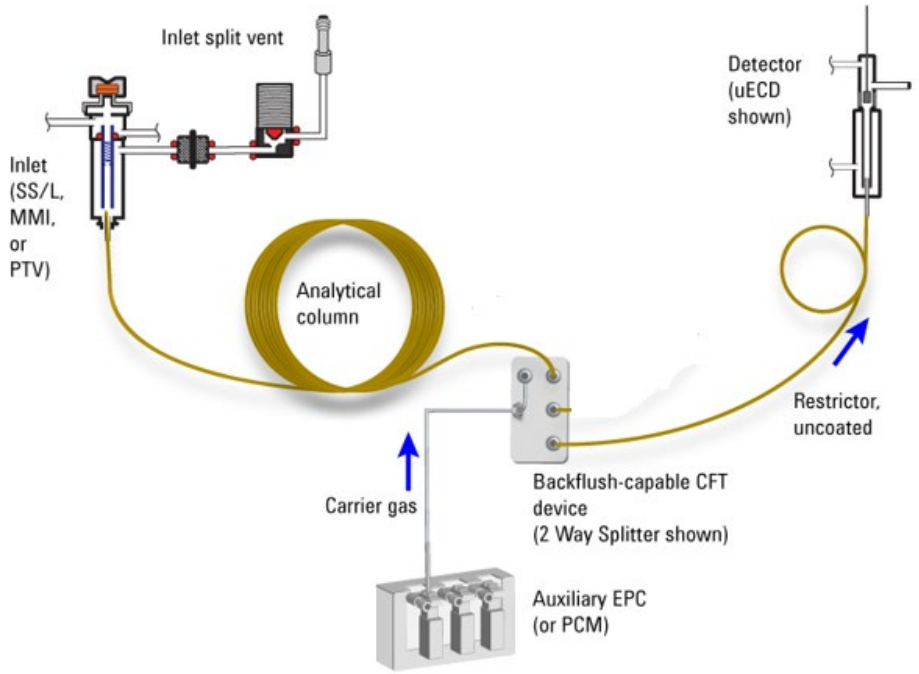
Backflush Techniques – Post Column Backflush

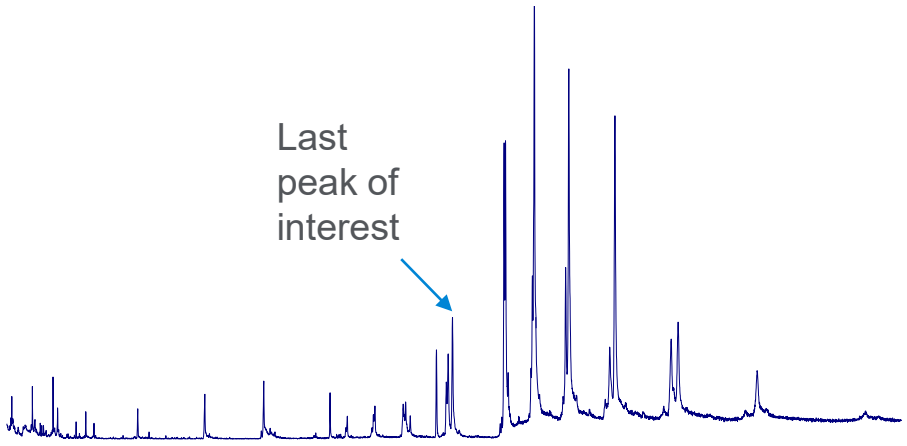


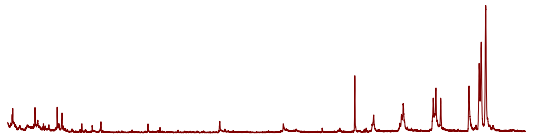
Post column backflush

- Configure the analytical column from the inlet to the EPC module. Flow rate set to that needed for peak separation.
- Configure Restrictor from EPC module to detector. Flow must be set at least 10% higher than that of the analytical column.
- Injection performed.
- Compounds travel through analytical column.
- When all compounds of interest are eluted out of the analytical column the run is stopped and the post run starts.
- The EPC module pressure is increased and the inlet pressure is dropped, reversing the flow through the analytical column. The “unwanted” compounds are backflushed out of the split vent.

Backflush Techniques – Post Column Backflush







Pros:

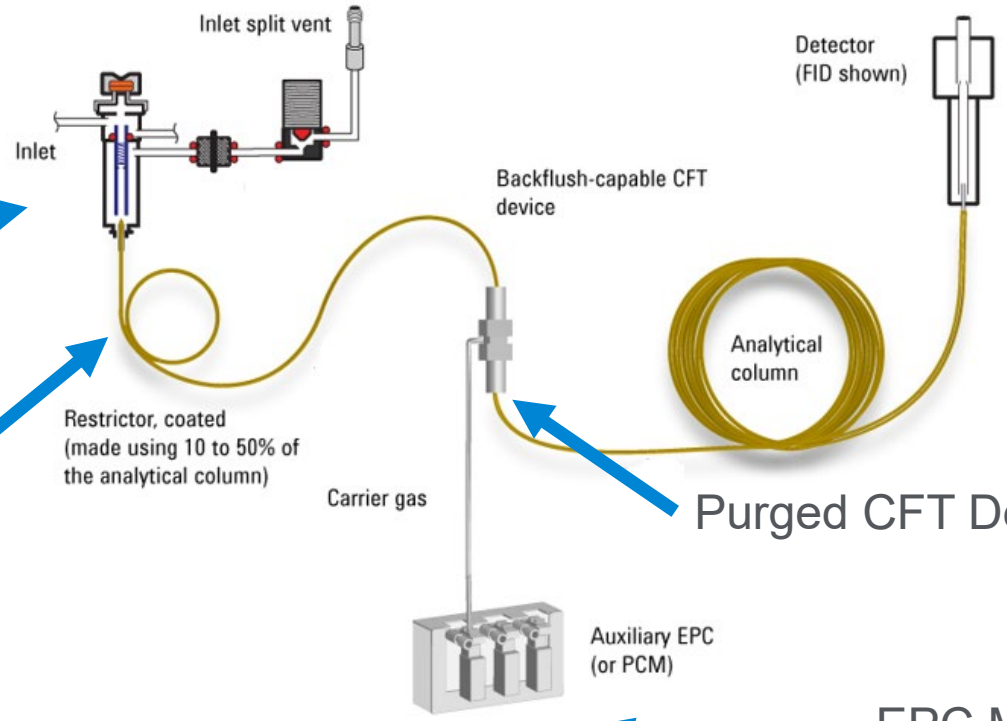
- Contaminants do not reach detector.
- Shorter cycle times (No bakeout required)
- Easy to set up – backflush occurs during post run.

Cons:

- Requires extra hardware (EPC module/CFT device).
- Occurs after the analytical run and therefore the GC cycle time is longer than that of precolumn backflush.
- Cannot be performed with some detectors

Backflush Techniques – Post Column Backflush

Inlet must have a split vent (SSI/MMI or PTV)

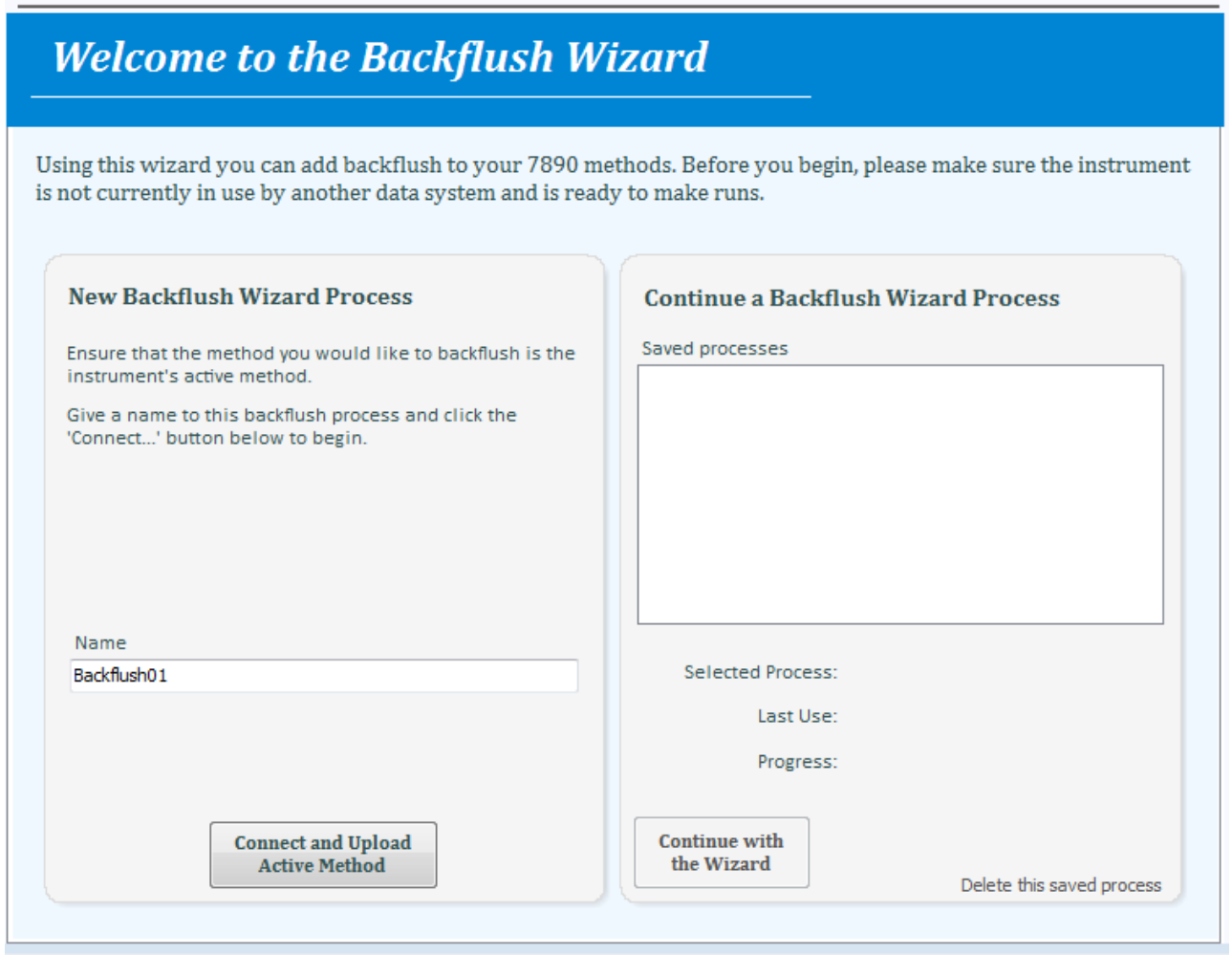


Purged CFT Device

Restrictor

EPC Module can be either Aux EPC or PCM.

Backflush Techniques – Tips and Tricks



Backflush Wizard:

- Program used to convert a non backflush method to one which uses backflush.
- Stored on GC/MS User Manuals and Tools Version <B.01.14.
- Can be used with Open Lab CDS Chemstation, MassHunter GCMS Acquisition, or standalone.
- Takes the user through the process of installing the backflush hardware, running the samples, setting up backflush times and running blanks.

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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