Things You Can Do to Make GC Systems Stay Up Longer to Analyze More POPs Samples

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The Main Problems with GC-MS

- GC inlet
  - Liner
  - Seal

- GC column
  - Guard column
  - Analytical column

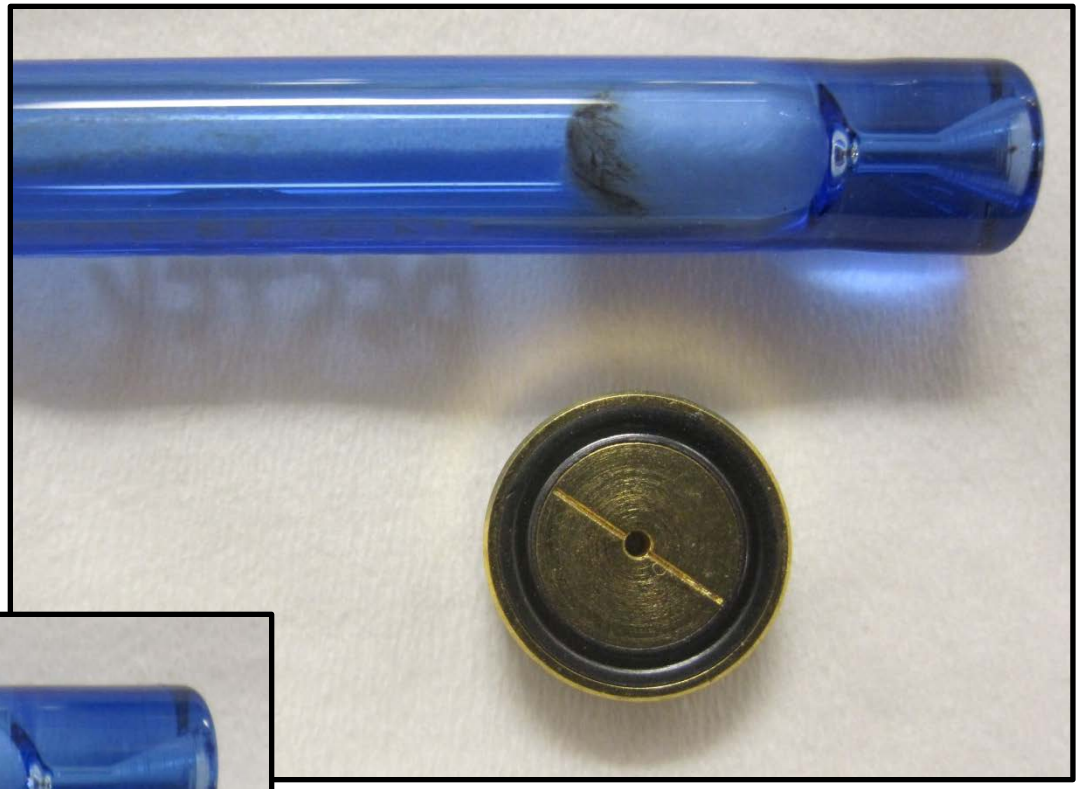
- Dirty samples contaminate liner wool and bottom seal
  - Poor compound transfer
  - Compound degradation

- Dirty samples contaminate front of column system
  - Poor compound transfer
  - Compound degradation

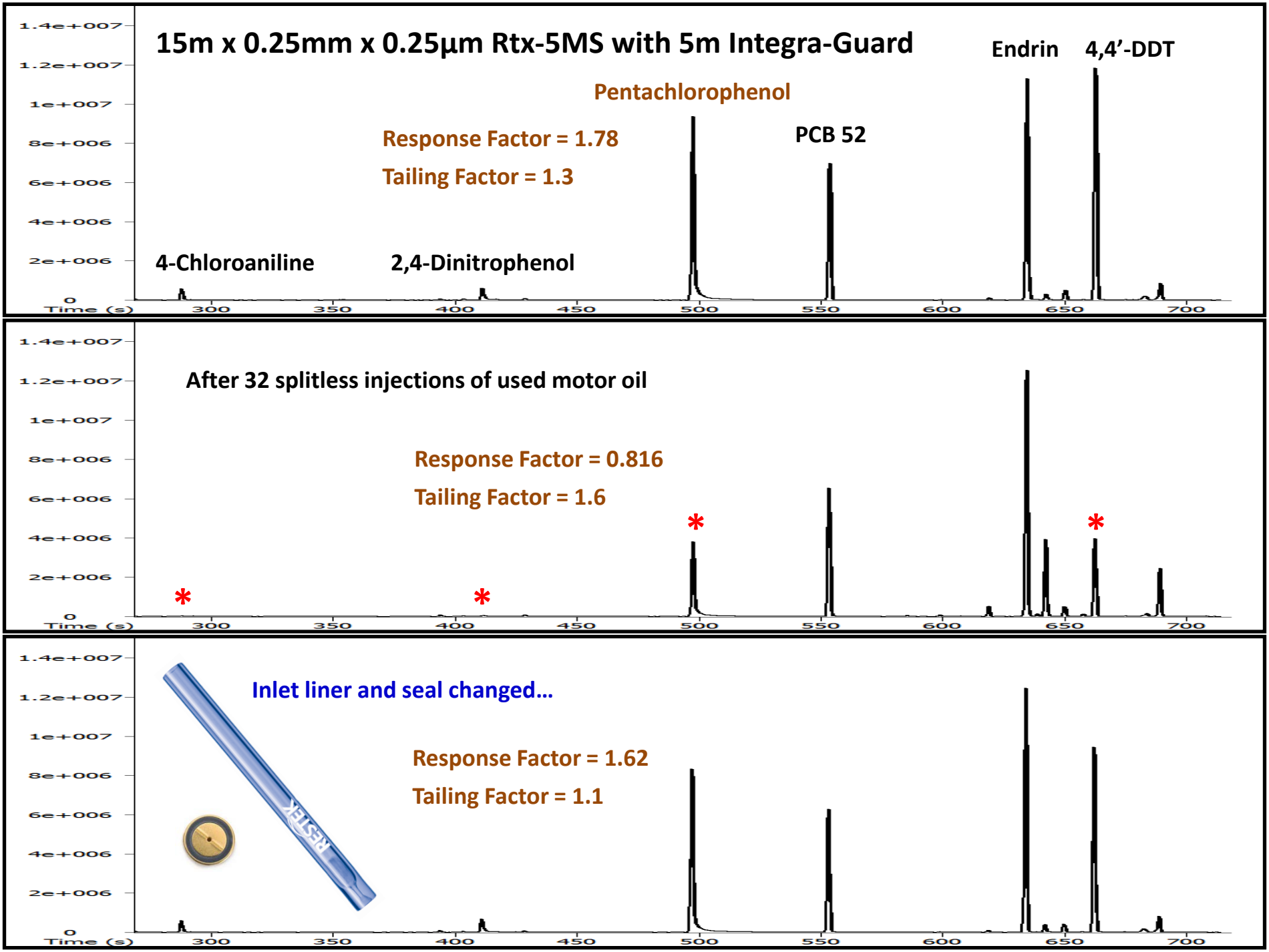
Both issues lead to poor data quality and downtime for maintenance…

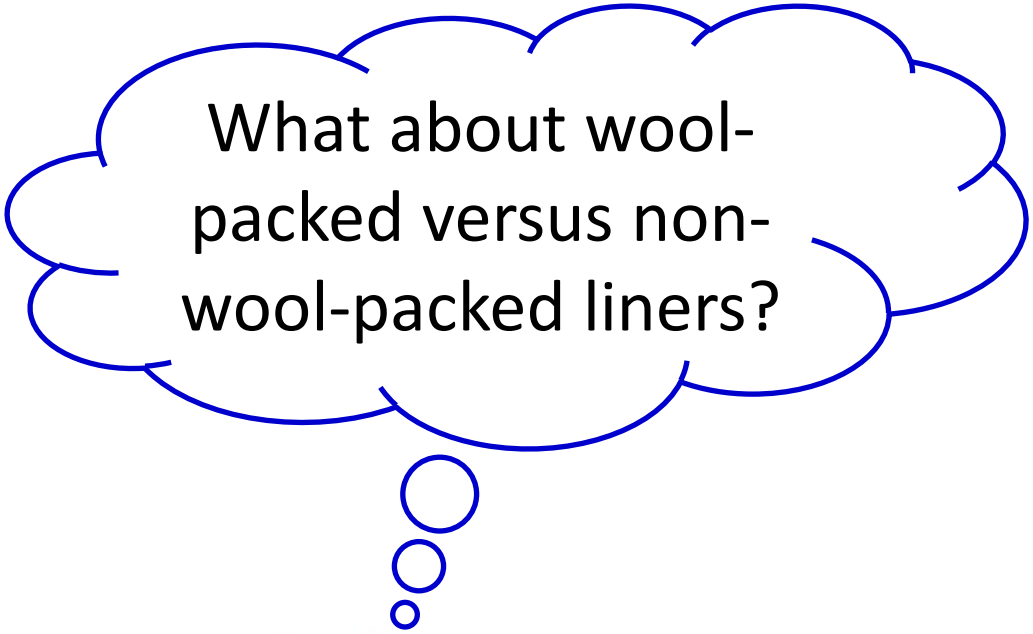
Splitless injection

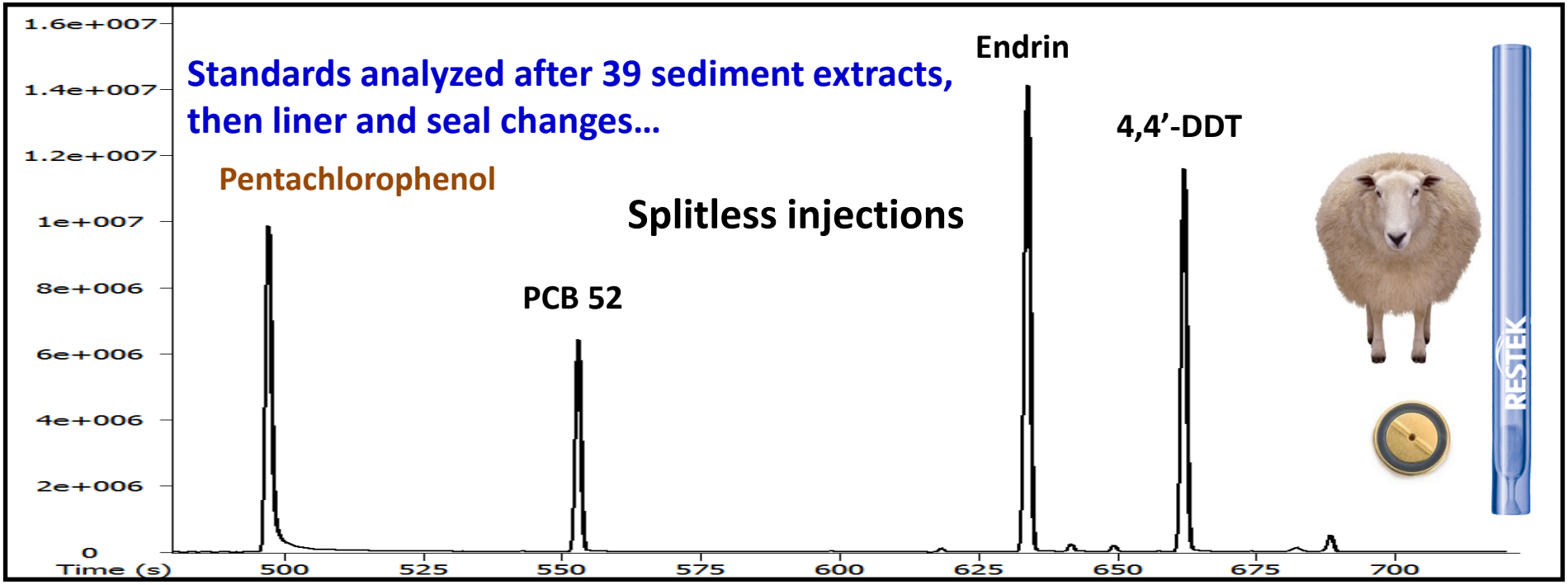
27 × Used Motor Oil
50,000 µg/mL

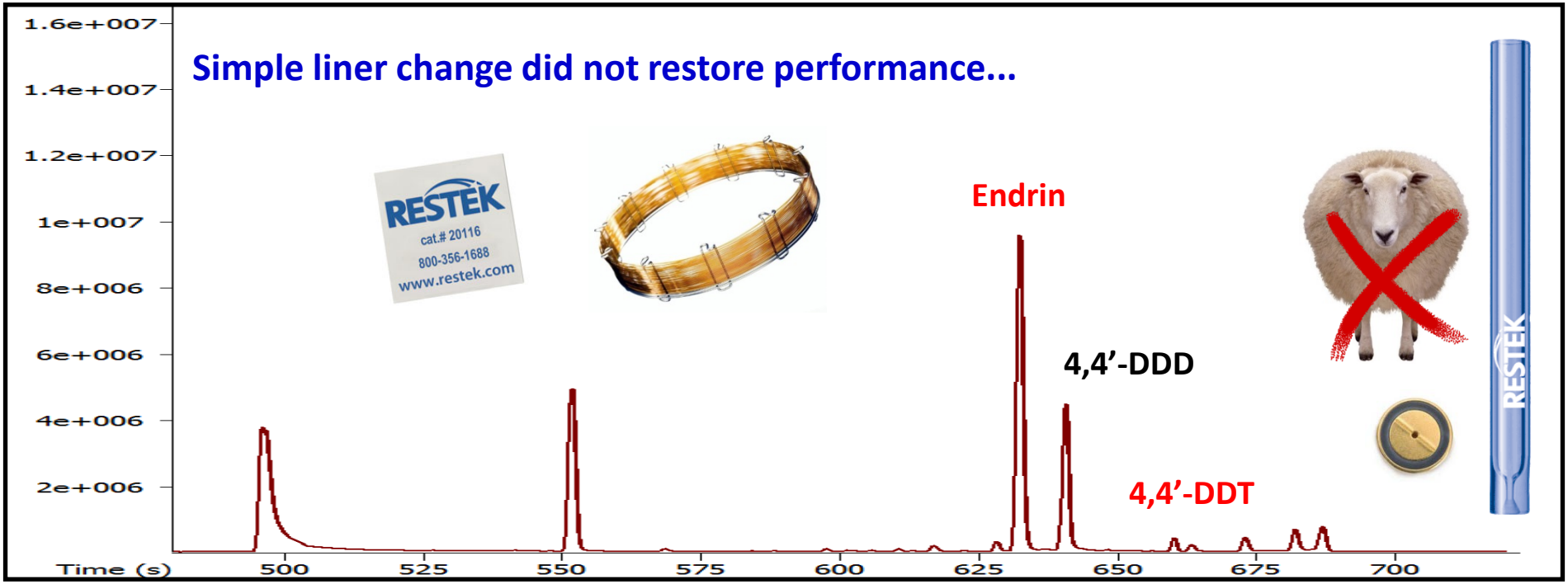


39 × Sediment Extract
(no dilution)


15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard
Endrin
4,4'-DDT
Pentachlorophenol
Response Factor = 1.78
Tailing Factor = 1.3
PCB 52
4-Chloroaniline
2,4-Dinitrophenol
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
300
350
400
450
500
550
600
650
700
After 32 splitless injections of used motor oil
Response Factor = 0.816
Tailing Factor = 1.6
Inlet liner and seal changed…
Response Factor = 1.62
Tailing Factor = 1.1

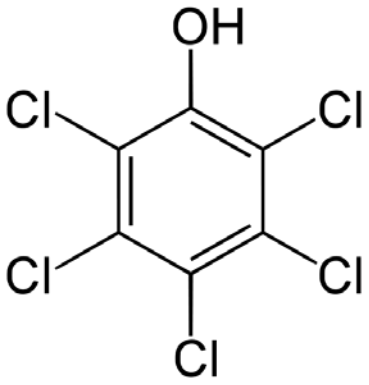

What about wool-packed versus non-wool-packed liners?


Standards analyzed after 39 sediment extracts, then liner and seal changes…
Pentachlorophenol
Splitless injections
PCB 52
Endrin
4,4’-DDT
RESTEK
1.6e+007
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
500
525
550
575
600
625
650
675
700


Simple liner change did not restore performance...
RESTEK
cat.# 20116
800-356-1688
www.restek.com
Endrin
4,4’-DDD
4,4’-DDT
RESTEK
1.6e+007
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
Time (s)
500
525
550
575
600
625
650
675
700

A Better Way?

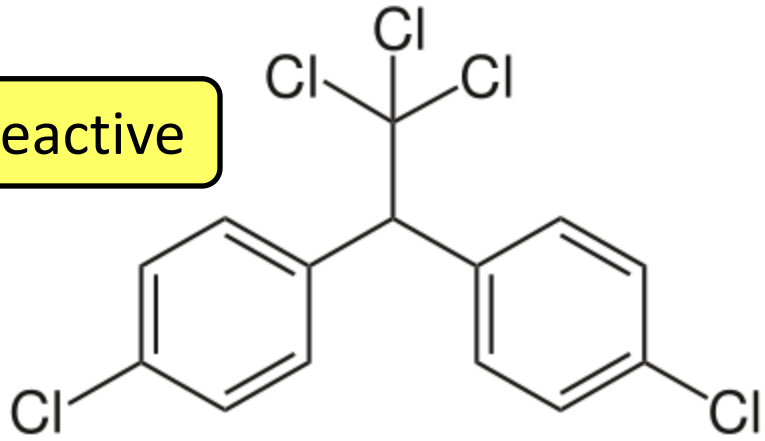
- Analyte Protectants
  - GC system active site masking compounds
  - Improve compound transfer from GC inlet
  - Allow better peak shape from GC column
- Split injection (shoot-and-dilute)
  - High GC inlet flow improves compound transfer
  - Less “dirt” on GC column improves peak shapes
  - System stays up longer



Active

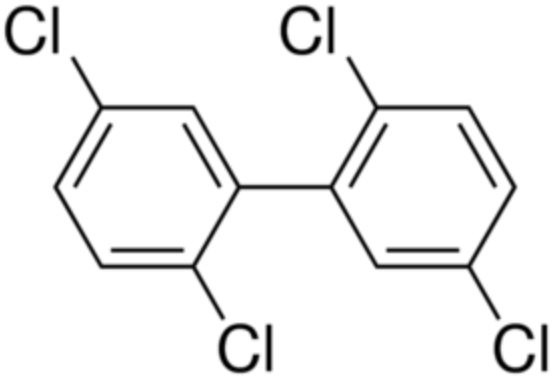
Pentachlorophenol

Reactive

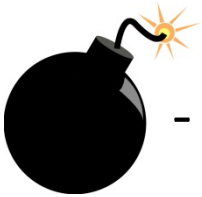


4,4’-DDT

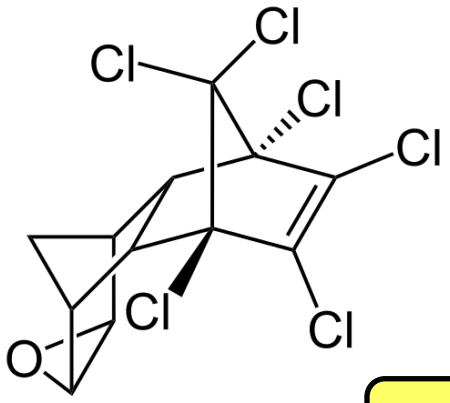
Internal standard



PCB 52

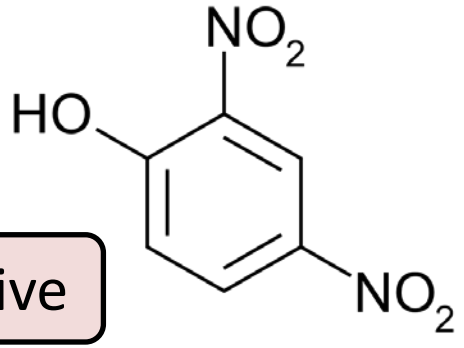


- Proof



Endrin

Reactive



Active

2,4-Dinitrophenol

UNITED NATIONS

UNEP

SC

UNEP/POPS/POPRC.10/10/Add.1

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Stockholm Convention on Persistent Organic Pollutants

Persistent Organic Pollutants Review Committee
Tenth meeting
Rome, 27–30 October 2014

Report of the Persistent Organic Pollutants Review Committee on the work of its tenth meeting

Addendum

Risk management evaluation on pentachlorophenol and its salts and esters

By its decision POPRC-10/1, the Persistent Organic Pollutants Review Committee adopted a risk management evaluation for pentachlorophenol and its salts and esters on the basis of the draft contained in the note by the secretariat (UNEP/POPS/POPRC.10/2). The text of the risk management evaluation, as amended, is set out in the annex to the present addendum. It has not been formally edited.

Executive Summary

1. Pentachlorophenol (PCP) and its salts and esters (sodium pentachlorophenate (Na-PCP), a PCP salt and; pentachlorophenyl laurate (PCP-L), a PCP ester) were proposed as a POPs candidate by the European Commission in 2011. At its eighth meeting, the POPs Review Committee concluded that while the PCP molecule does not meet all the screening criteria specified in Annex D, taking into account its transformation product pentachloroanisole (PCA), PCP and its salts and esters do meet the screening criteria. This led to the POPs Review Committee at its ninth meeting to decide that PCP, its salts and esters, are likely, including consideration of the transformation product, PCA, as a result of their long-range environmental transport, to lead to significant adverse human health and environmental effects such that global action is warranted (Decision POPRC-9/3).

2. PCP has had multiple uses in the past (biocide, insecticide, fungicide, disinfectant, defoliant, anti-sapstain agent, anti-microbial agent and wood preservative) which have now been phased out with the remaining key use being wood preservation, particularly for use in utility poles and cross-arms, with minor uses for railway ties (cross ties or 'sleepers') and outdoor construction materials (UNECE 2010). PCP has also been used to produce the ester PCP-L, which was used in textiles, but there is no evidence of continued use. Its salt, Na-PCP, is used for similar purposes to PCP and readily dissociates to PCP. PCA is not used as a commercial chemical or pesticide and is not intentionally released directly into the environment.

8. The risk profile concluded that PCP and its related compounds are likely to lead to significant adverse human health and environmental effects. In addition, the manufacturing and use of PCP-treated wood is a source of dioxins and furans. Therefore, the implementation of further control measures would reduce potential risks from exposure to humans and the environment from PCP and PCA. In addition it will reduce the potential exposure to dioxins and furans present as impurities from in-service PCP-treated wood, which is not covered by the listing of dioxins in Annex C (UNEP/POPS/POPRC.9/13/Add.3).

Executive Summary

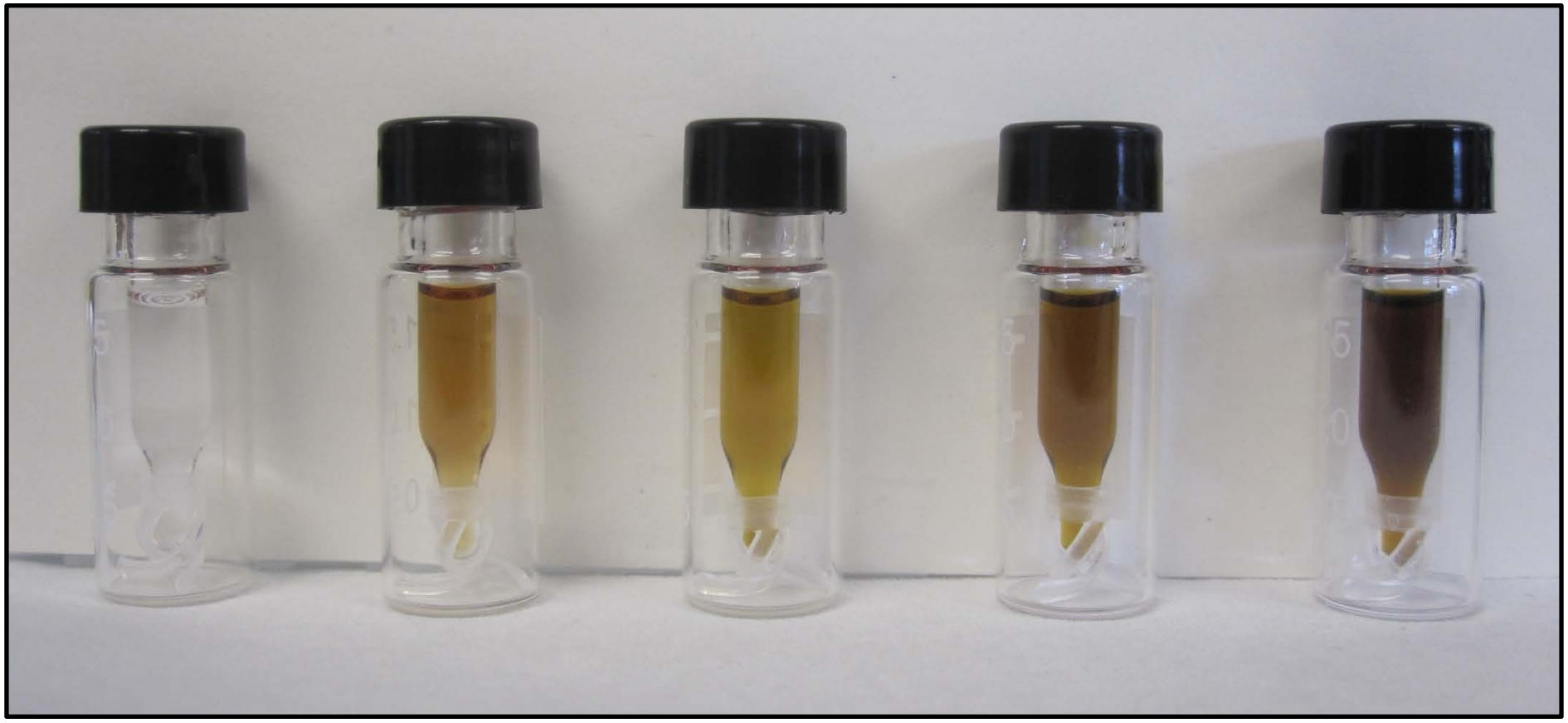
1.

Pentachlorophenol by itself did not meet all screening criteria for a POP (persistence, bio-accumulation, potential for long-range environmental transport, adverse effects), but significant adverse human health and environmental effects, and its transformation product, pentachloroanisole, get it in the club…

GC-ECD Method

- Sky 4mm single taper liner
  - With quartz wool
  - 1 µL, 250°C, splitless valve time 1.5 min
- 15m x 0.25mm x 0.25µm Rtx-5MS
  - 5m Integra-Guard
  - Helium carrier at 1.4 mL/min constant flow
  - 70°C (1.5 min), 20°C/min to 330°C (1.5 min)
- Electron capture detector
  - 350°C, 50 mL/min N_2 makeup, 10 Hz

Toluene	Used Motor Oil 50,000 µg/mL	Sediment Extract 1 to 4	Sediment Extract 1 to 2	Sediment Extract 1 to 1



Used Motor Oil is an excellent “dirty sample” surrogate.

The Sediment Extract, which contained priority pollutants such as PAHs, was analyzed dilute and straight to create stress on the GC inlet liner and GC column.

Used Motor Oil Composite Standard

Prepared from an equal-volume blend from five gasoline-powered vehicles (belonging to Restek employees). After blending, a precisely weighed amount of the composite is added to a volumetric flask to produce the standard.

Used Motor Oil Composite Standard

Prepared from an equal-volume blend from five gasoline-powered vehicles (belonging to Restek employees). After blending, a precisely weighed amount of the composite is added to a volumetric flask to produce the standard.

Selected Product

Compound:	**used motor oil composite**
CAS #:	**64742-65-0**
Conc. In Solvent and Volume:	**50,000 µg/mL in methylene chloride, 1 mL/ampul**

Cat.#: **31465**

List Price: **$34.00**

Unit: ea.

Qty: 1

METHOD 8270D

SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

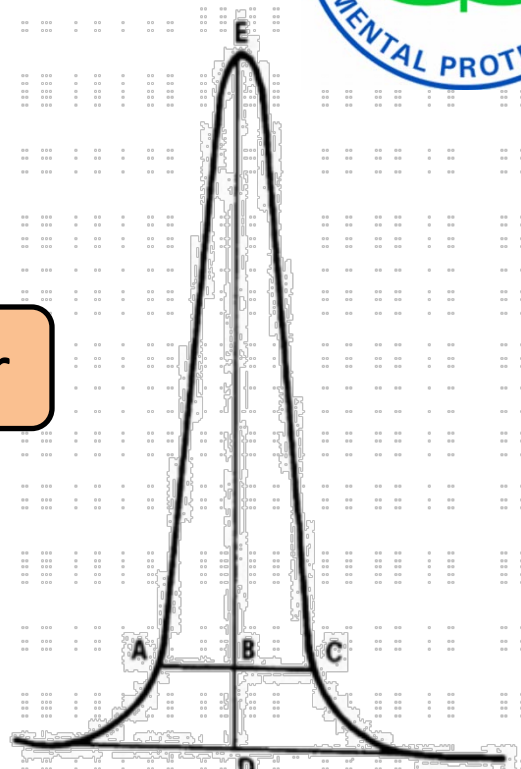
11.3.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. (See Method 8081 for the percent breakdown calculation.) Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{TailingFactor} = \frac{BC}{AB}$$

Tailing Factor

Where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

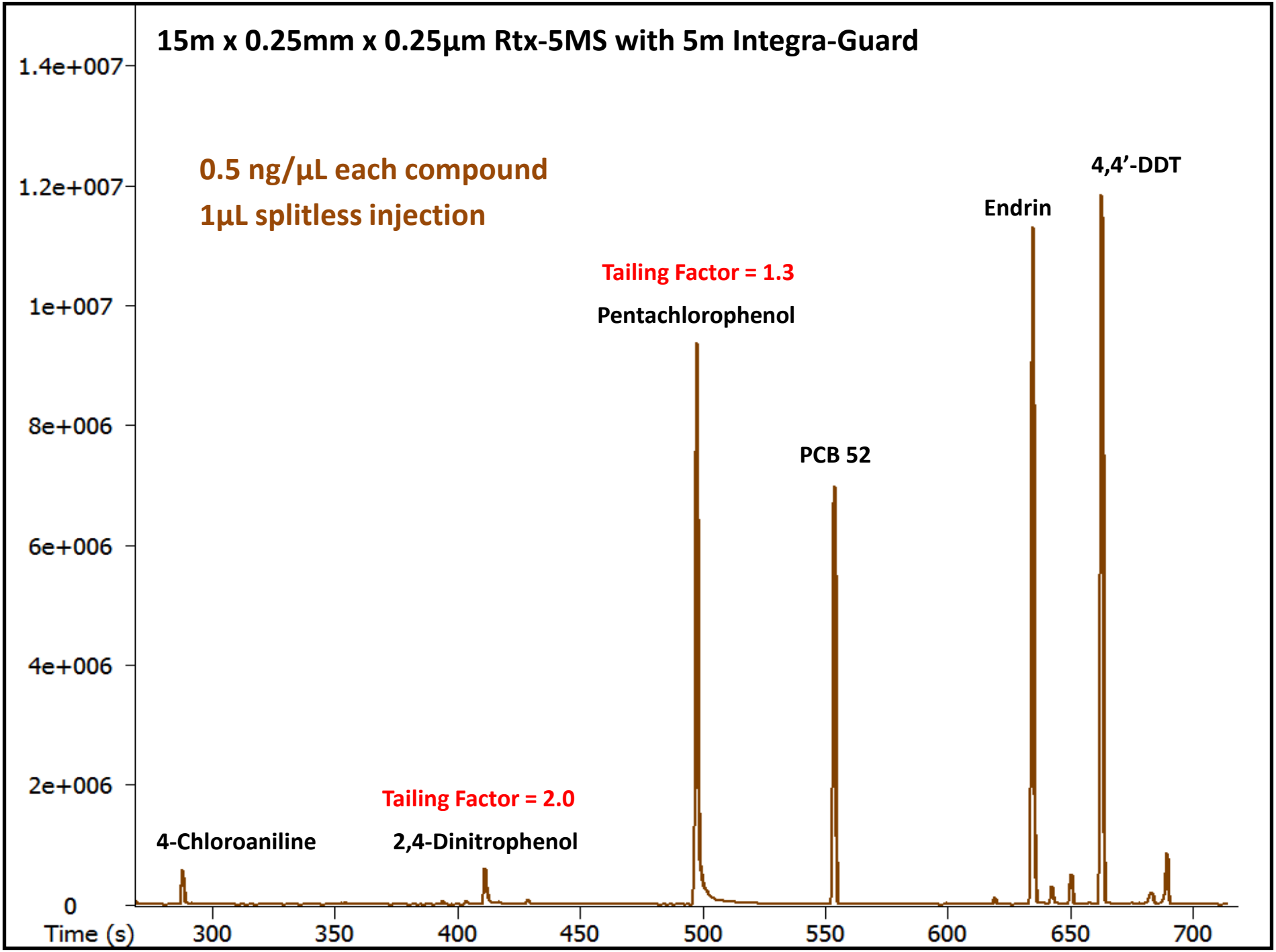
11.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6 to12 in. of the capillary column. The use of a guard column (Sec. 6.1.6) between the injection port and the analytical column may help prolong analytical column performance life.



Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm
Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

GC inlet inertness and GC column performance…

Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2…


15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard
0.5 ng/µL each compound
1µL splitless injection
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
300
350
400
450
500
550
600
650
700
4-Chloroaniline
Tailing Factor = 2.0
2,4-Dinitrophenol
Pentachlorophenol
Tailing Factor = 1.3
PCB 52
Endrin
4,4'-DDT

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JOURNAL OF CHROMATOGRAPHY A

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Evaluation of analyte protectants to improve gas chromatographic analysis of pesticides☆

Michelangelo Anastassiades[1], Kateřina Maštovská, Steven J. Lehotay*

US Department of Agriculture, Agricultural Research Service, Eastern Regional Research Center, 600 East Mermaid Lane; Wyndmoor, PA 19038, USA

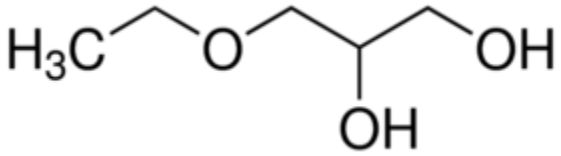
Received 9 August 2002; received in revised form 3 February 2003; accepted 4 February 2003

Significant peak quality improvements obtained when matrix components are present to fill active sites and reduce analyte interactions.

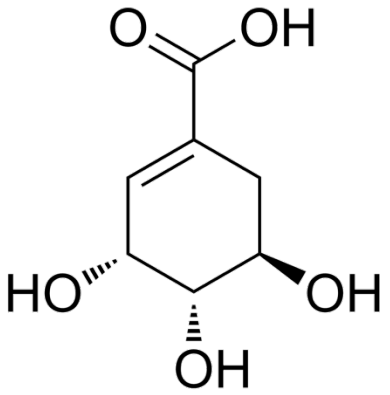
"Matrix-induced chromatographic response enhancement".

Addition of "analyte protectants" (e.g. sugars, acids, etc.) to standards and samples.

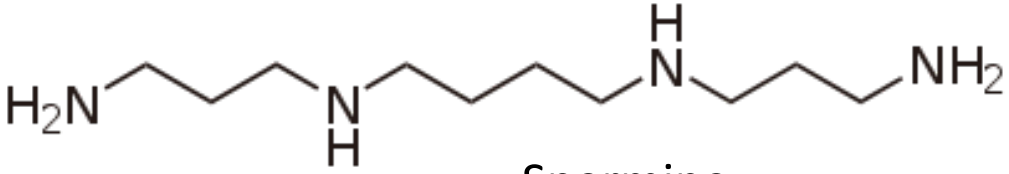
Provides chromatographic enhancement effect for analytes in a very dirty GC system.



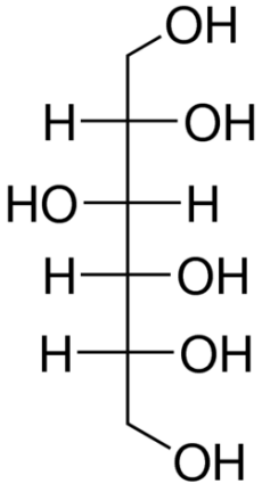
3-Ethoxy-1,2-propanediol



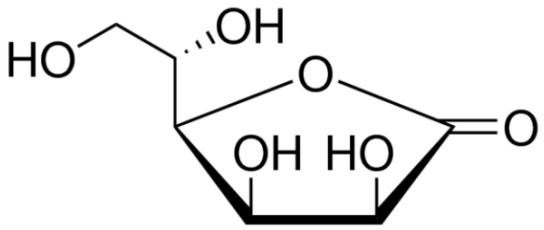
Shikimic acid



Spermine

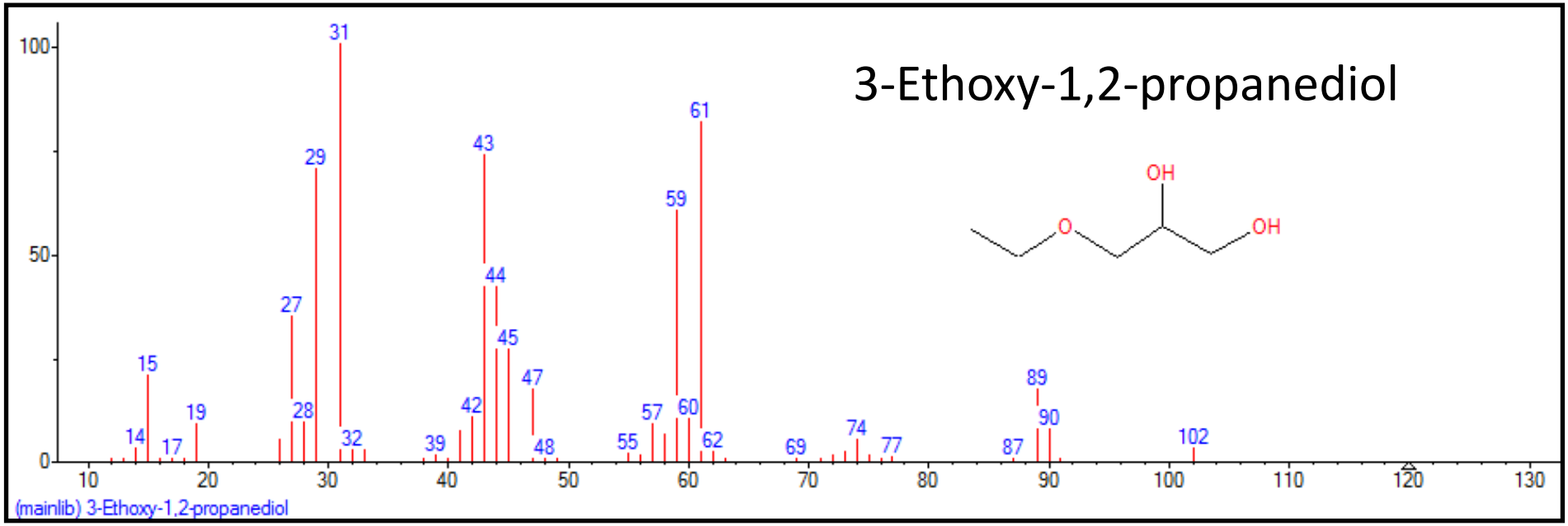


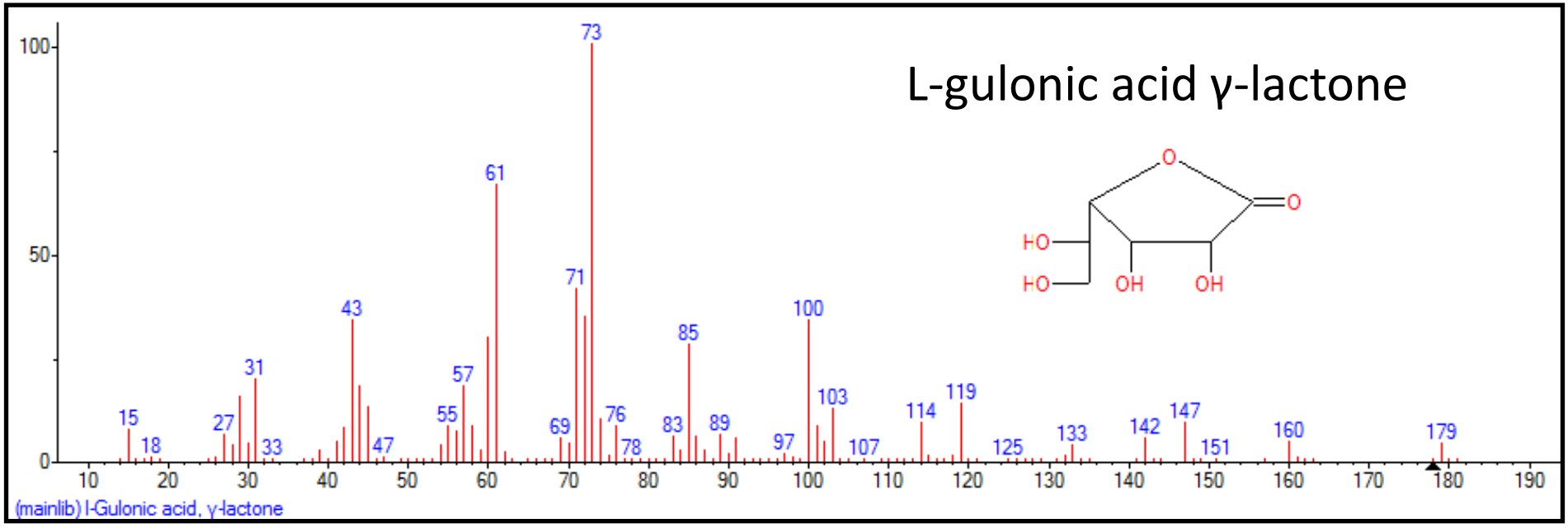
D-sorbitol



L-gulonic acid γ-lactone

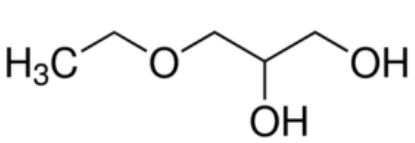
Analyte protectants

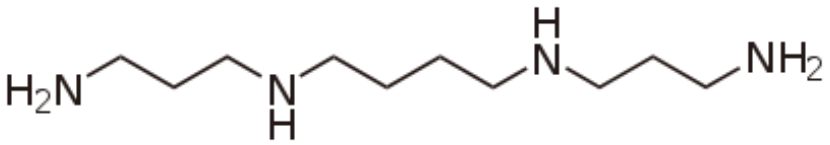

3-Ethoxy-1,2-propanediol
OH
O
OH
(mainlib) 3-Ethoxy-1,2-propanediol

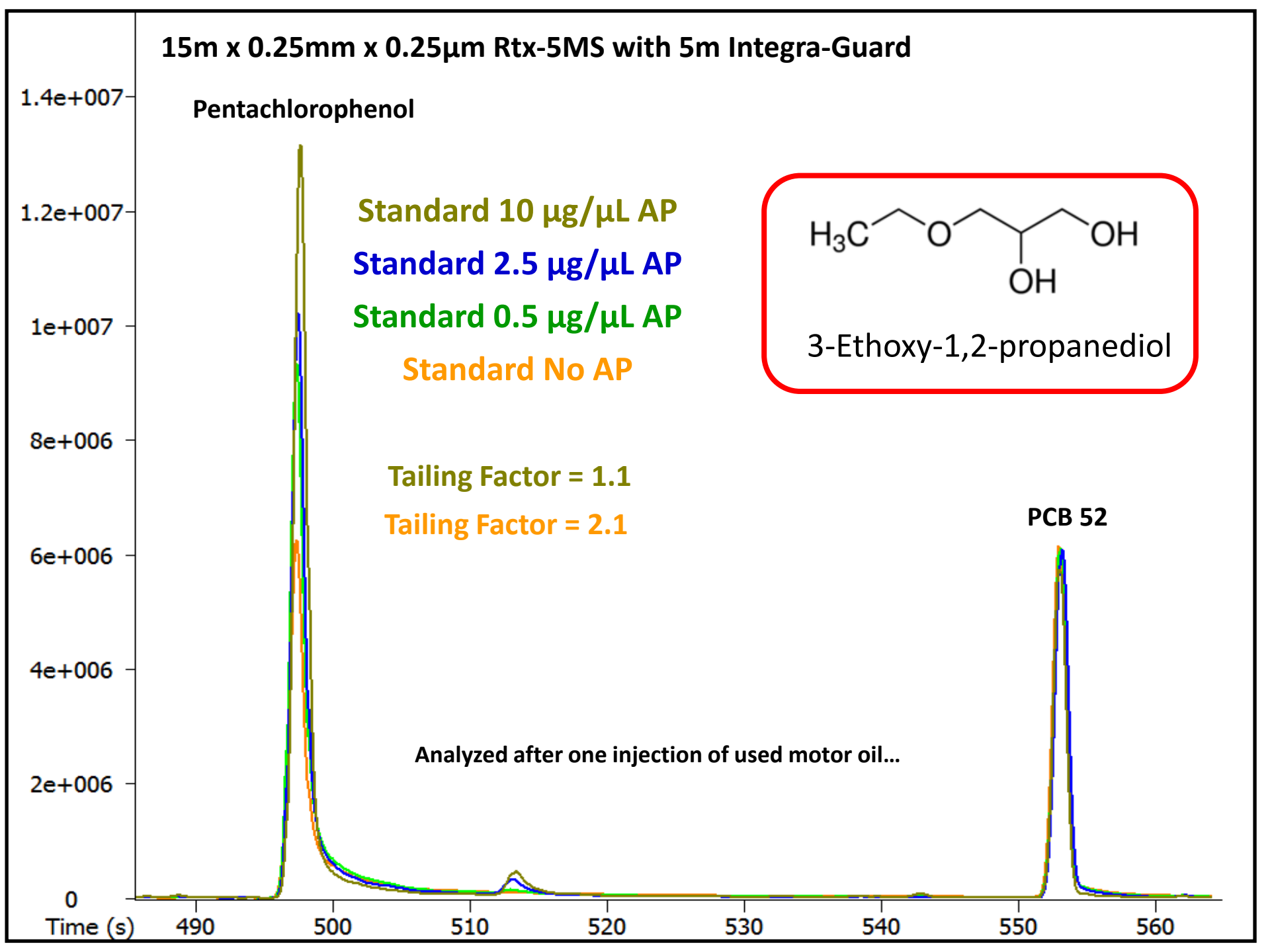

L-gulonic acid γ-lactone
O
O
HO
HO
OH
OH
(mainlib) l-Gulonic acid, γ-lactone

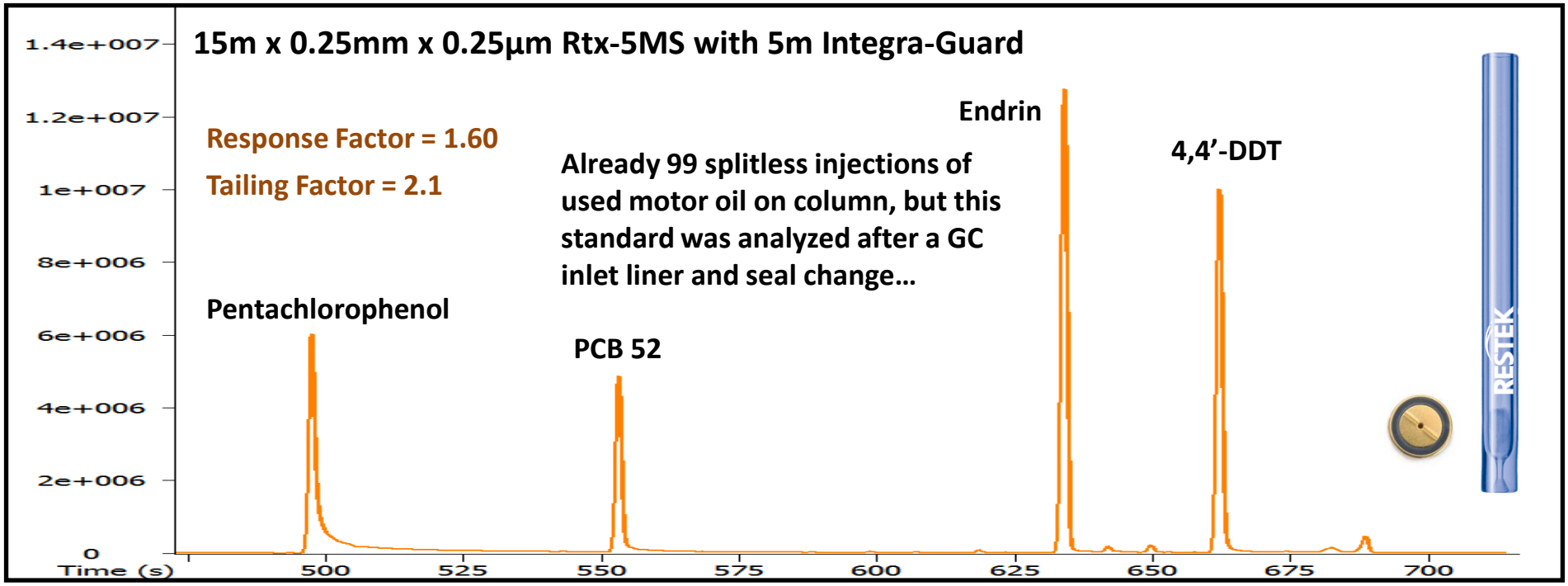
Analyte Protectant Criteria

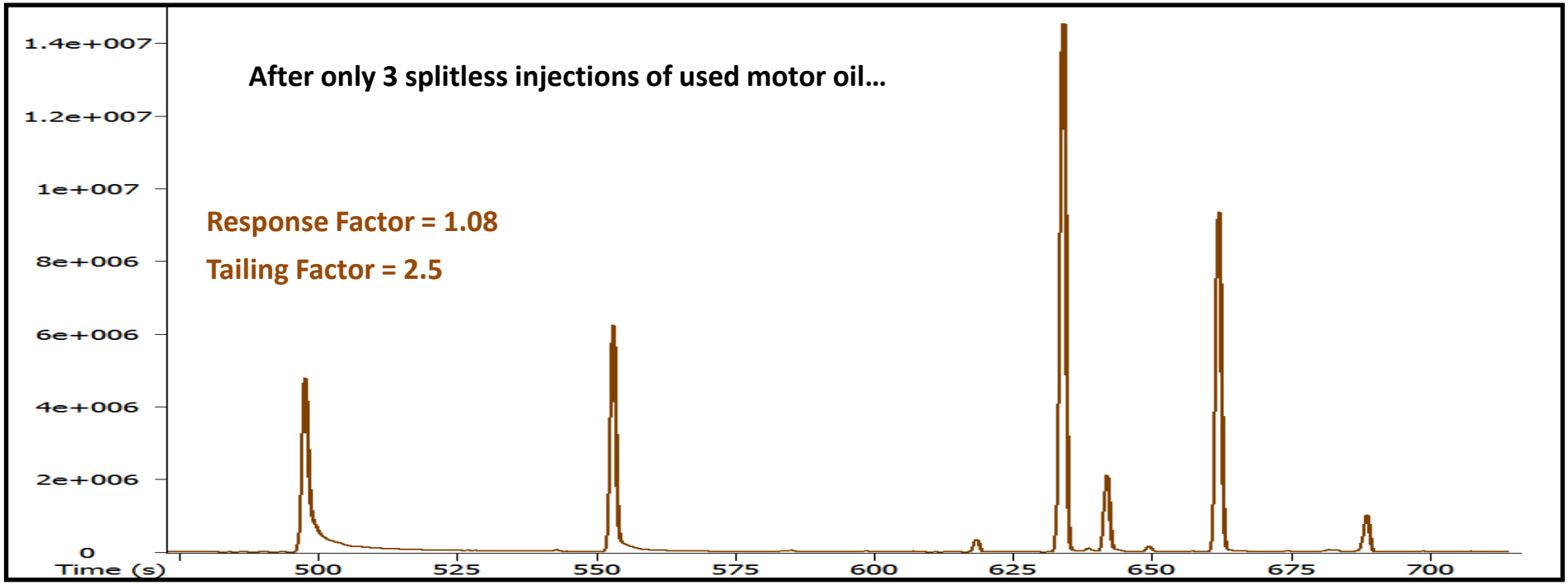
- Rich in hydroxys/aminos to deactivate systems
- Volatile so they gas chromatograph
- Several may perform better than one
- Volatility range similar to compounds analyzed
- Added in high concentration
- Low m/z ions to avoid quantification bias
- Inexpensive
- Injected with each standard and sample

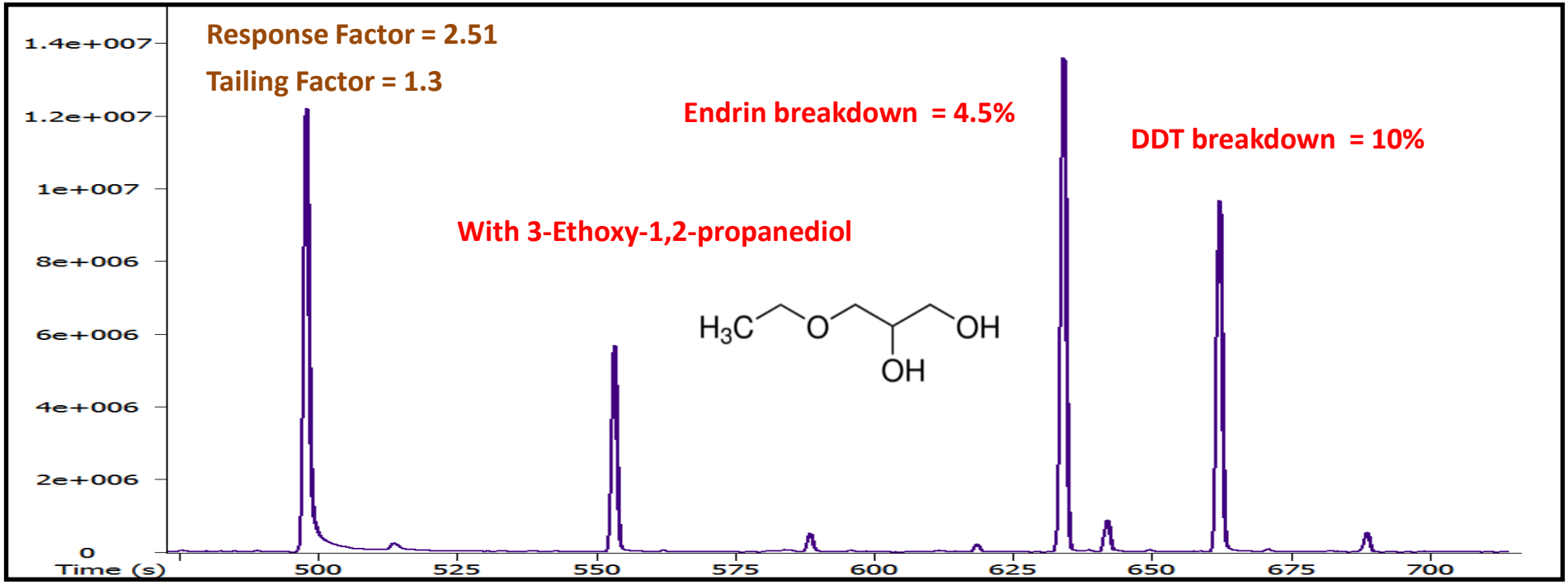


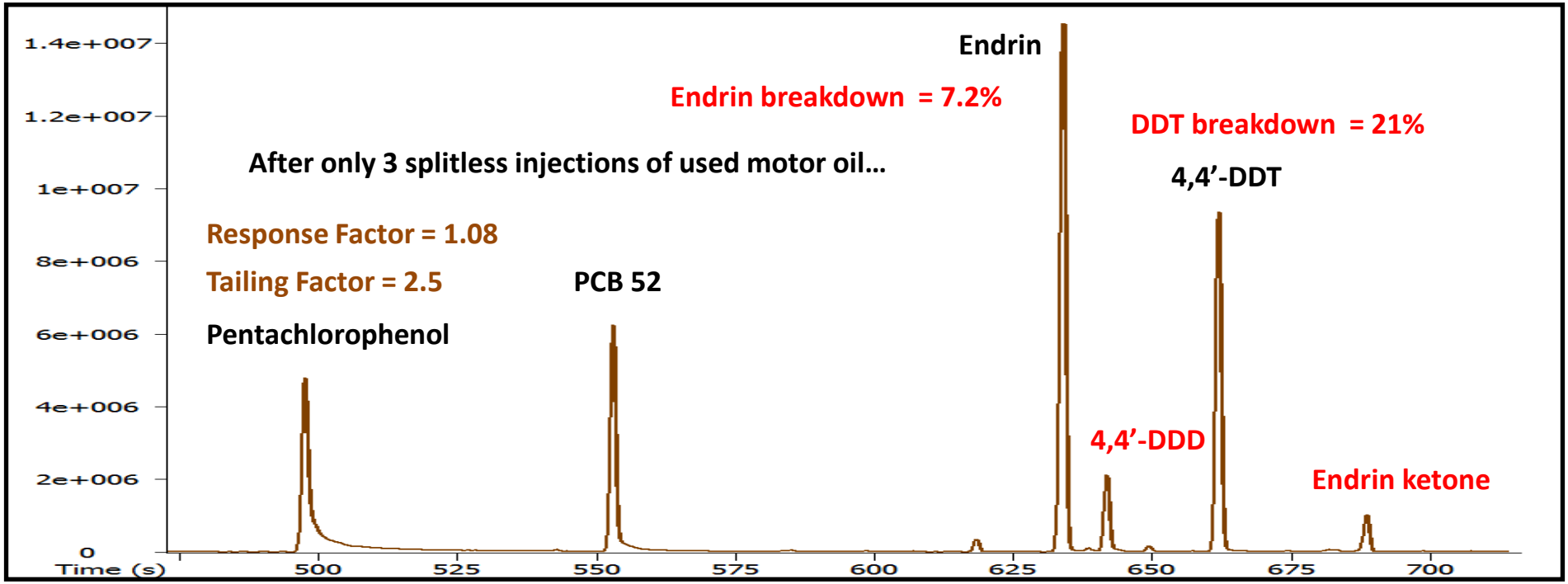


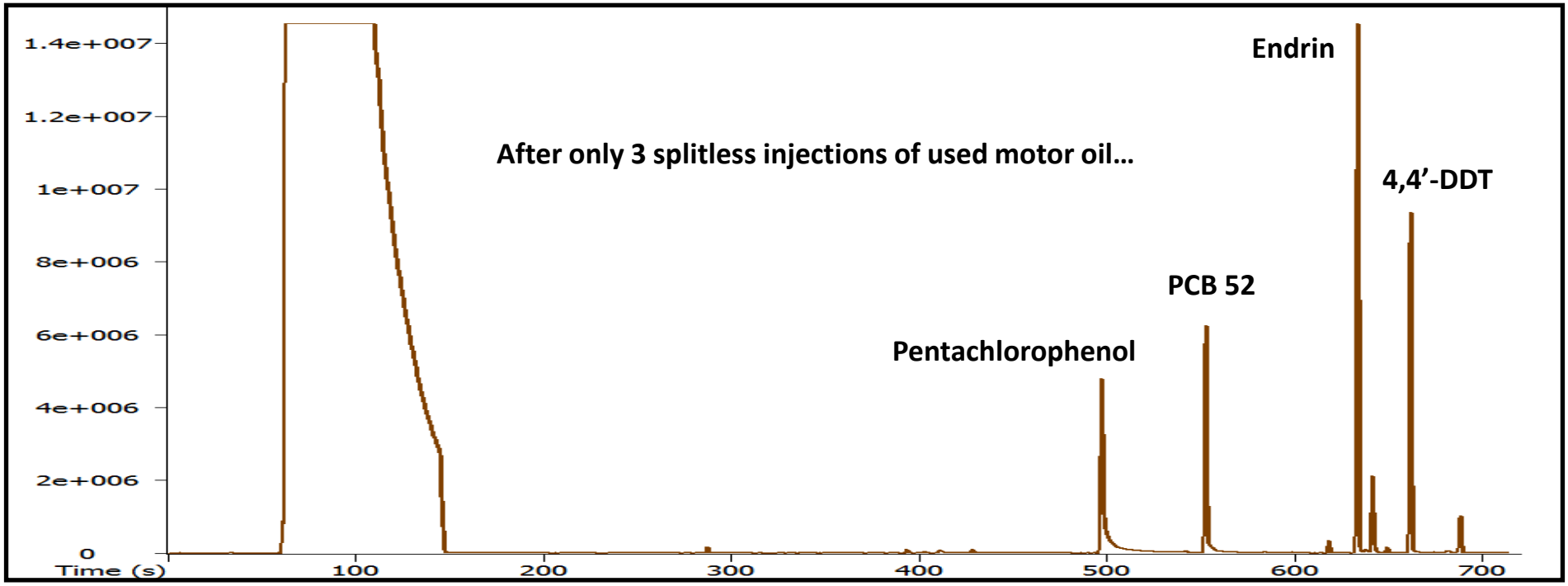

15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard
Pentachlorophenol
Standard 10 µg/µL AP
Standard 2.5 µg/µL AP
Standard 0.5 µg/µL AP
Standard No AP
3-Ethoxy-1,2-propanediol
Tailing Factor = 1.1
Tailing Factor = 2.1
PCB 52
Analyzed after one injection of used motor oil…
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
490
500
510
520
530
540
550
560

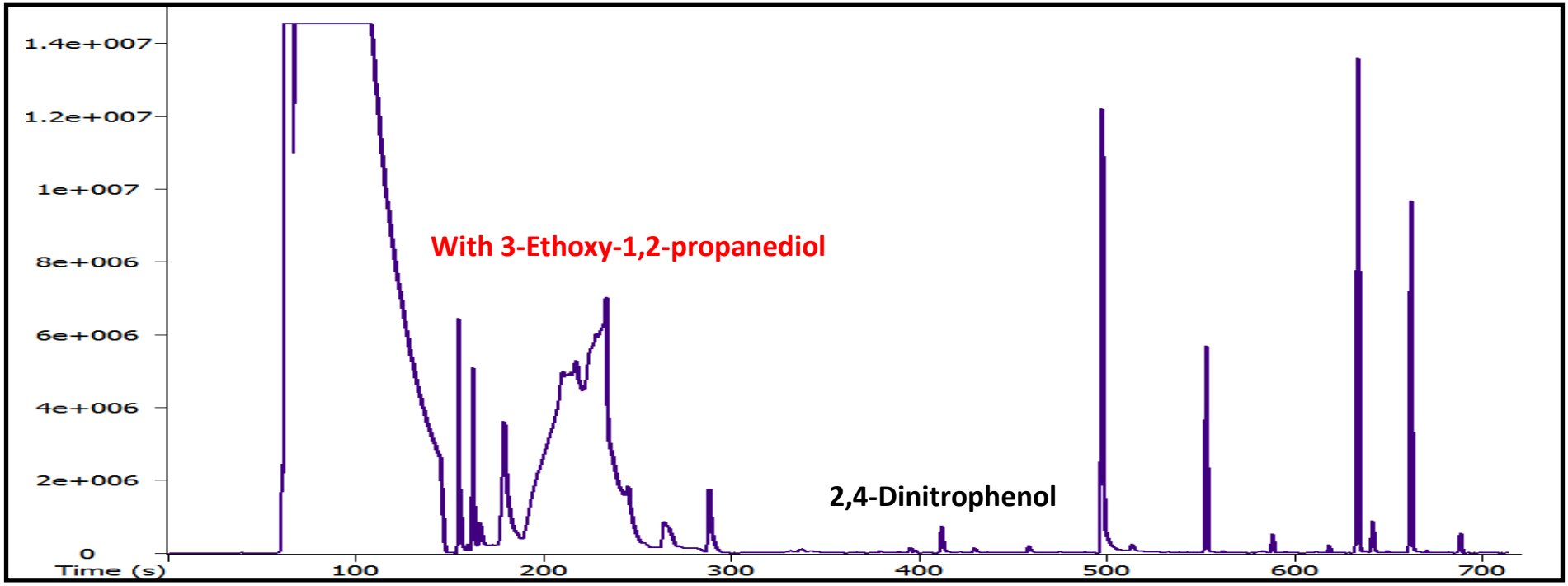

15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard
Response Factor = 1.60
Tailing Factor = 2.1
Already 99 splitless injections of used motor oil on column, but this standard was analyzed after a GC inlet liner and seal change…
Pentachlorophenol
PCB 52
Endrin
4,4'-DDT
RESTEK
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
500
525
550
575
600
625
650
675
700

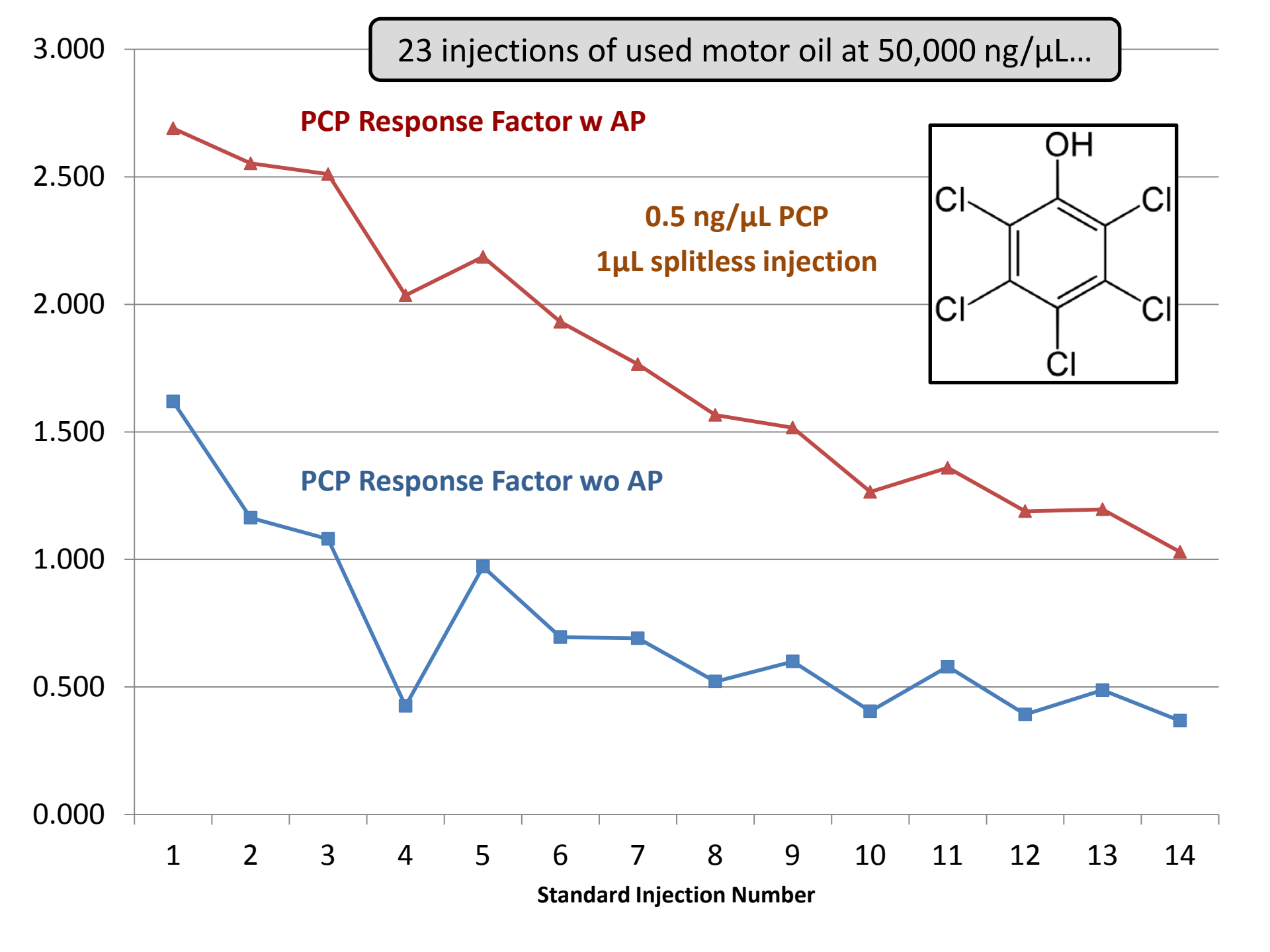

After only 3 splitless injections of used motor oil…
Response Factor = 1.08
Tailing Factor = 2.5
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
500
525
550
575
600
625
650
675
700

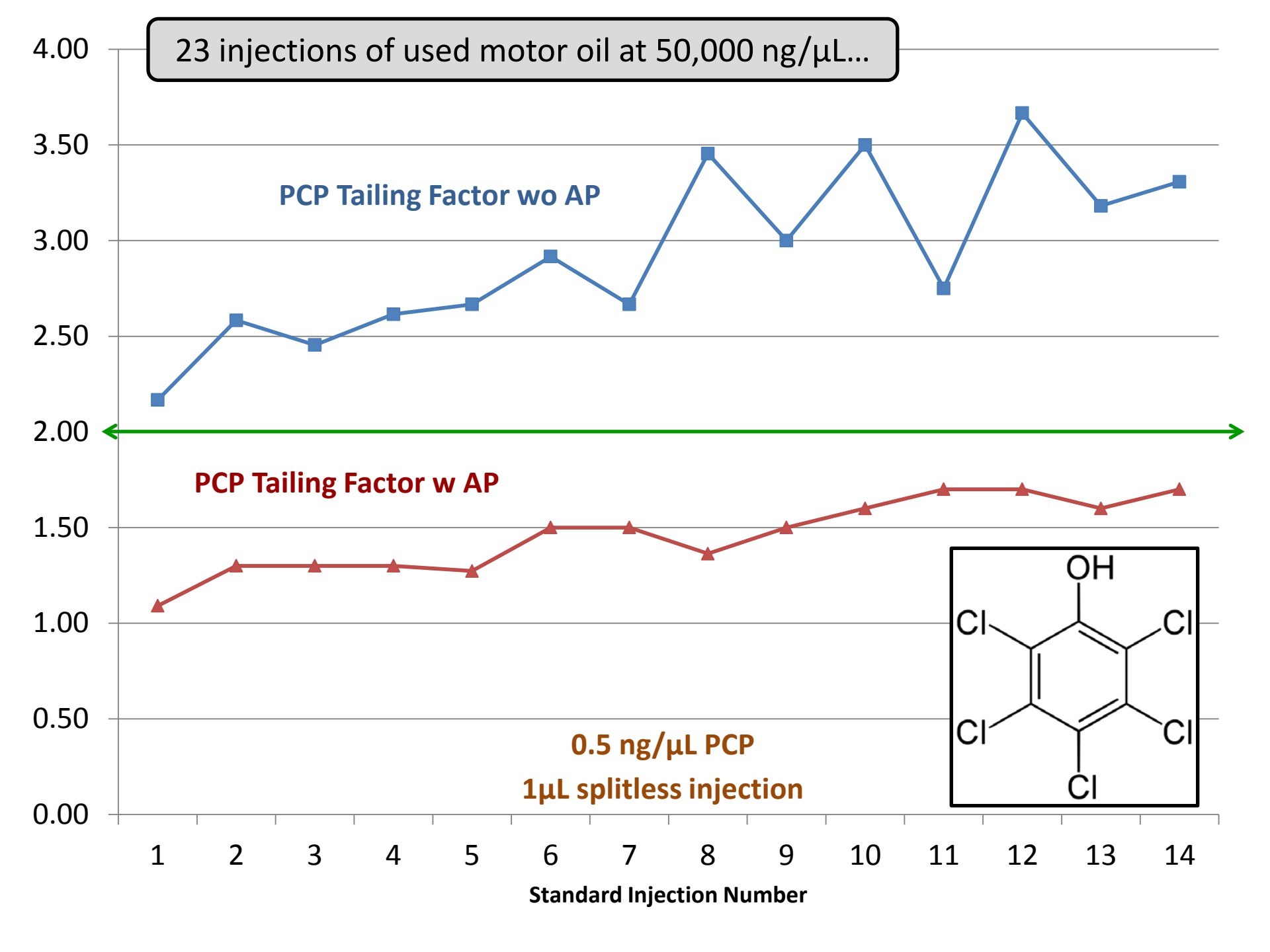

Response Factor = 2.51
Tailing Factor = 1.3
Endrin breakdown = 4.5%
DDT breakdown = 10%
With 3-Ethoxy-1,2-propanediol
H3C
O
OH
OH
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
500
525
550
575
600
625
650
675
700

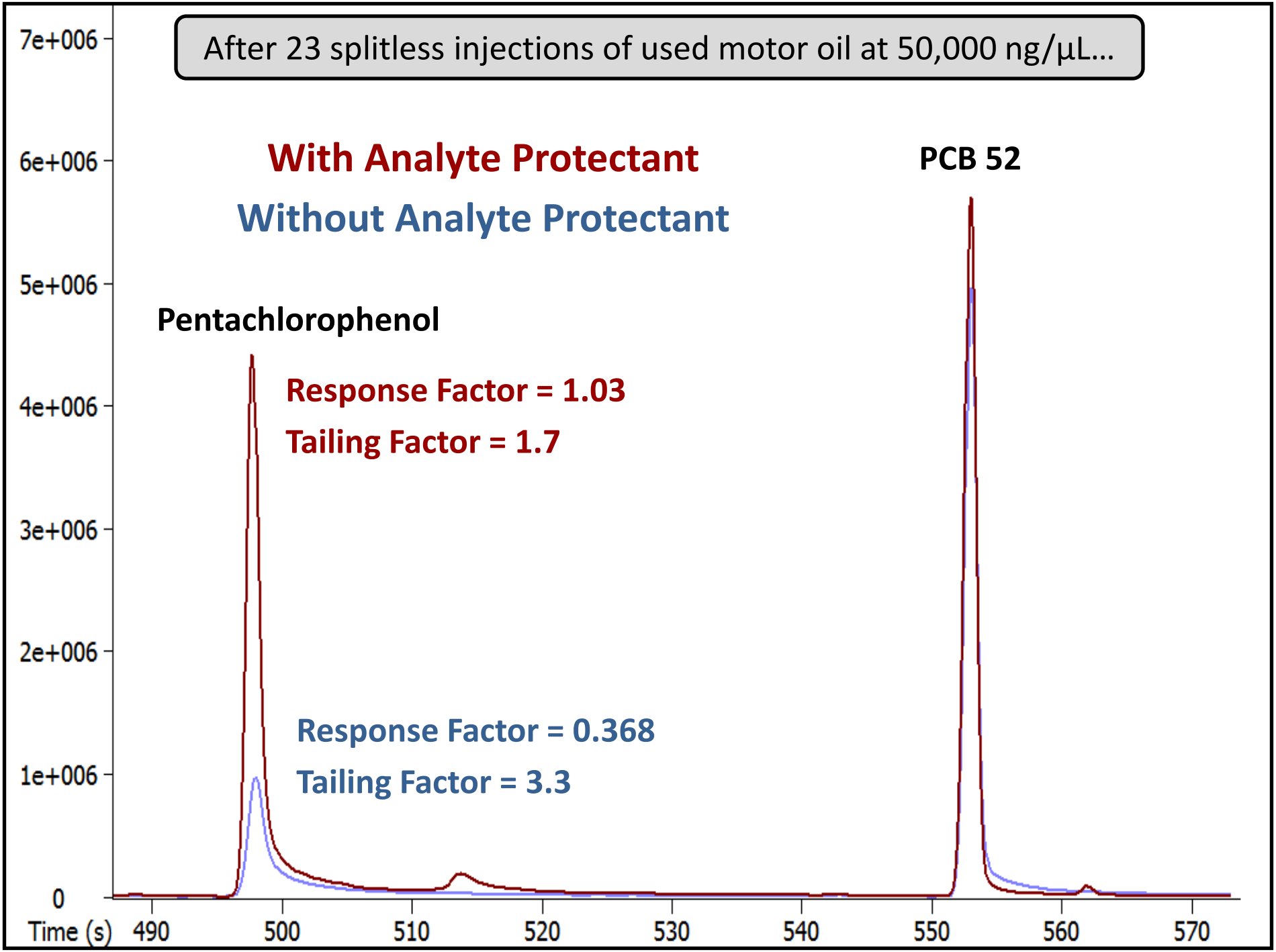

Endrin
Endrin breakdown = 7.2%
DDT breakdown = 21%
4,4'-DDT
After only 3 splitless injections of used motor oil…
Response Factor = 1.08
Tailing Factor = 2.5
PCB 52
Pentachlorophenol
4,4'-DDD
Endrin ketone
1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
500
525
550
575
600
625
650
675
700

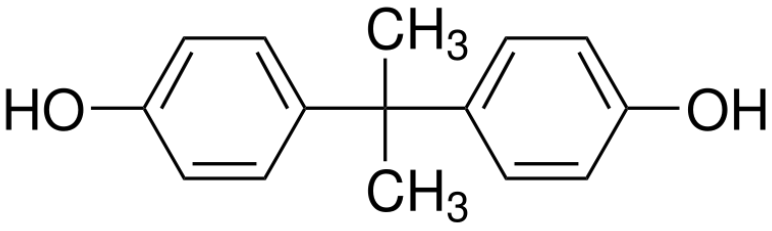

1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
100
200
300
400
500
600
700
After only 3 splitless injections of used motor oil…
Endrin
4,4'-DDT
PCB 52
Pentachlorophenol


1.4e+007
1.2e+007
1e+007
8e+006
6e+006
4e+006
2e+006
0
Time (s)
100
200
300
400
500
600
700
With 3-Ethoxy-1,2-propanediol
2,4-Dinitrophenol

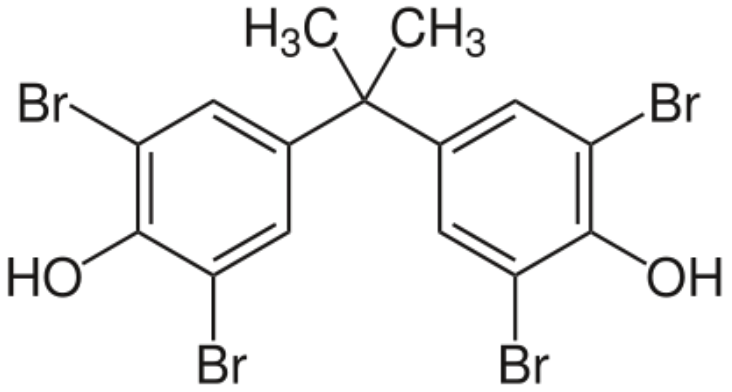

23 injections of used motor oil at 50,000 ng/µL…
PCP Response Factor w AP
0.5 ng/µL PCP
1µL splitless injection
PCP Response Factor wo AP
OH
Cl
Cl
Cl
Cl
Cl
3.000
2.500
2.000
1.500
1.000
0.500
0.000
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Standard Injection Number


23 injections of used motor oil at 50,000 ng/µL…
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
PCP Tailing Factor wo AP
PCP Tailing Factor w AP
0.5 ng/µL PCP
1µL splitless injection
OH
Cl
Cl
Cl
Cl
Cl
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Standard Injection Number

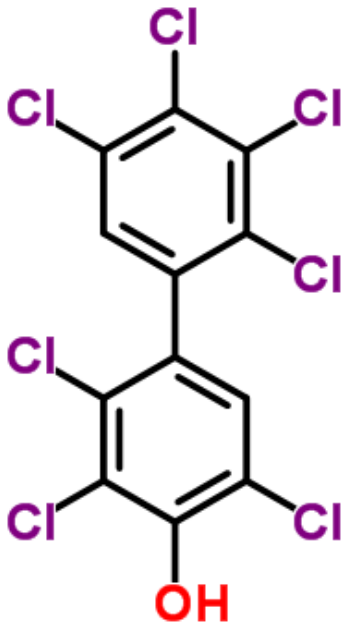

After 23 splitless injections of used motor oil at 50,000 ng/µL…
With Analyte Protectant
Without Analyte Protectant
PCB 52
Pentachlorophenol
Response Factor = 1.03
Tailing Factor = 1.7
Response Factor = 0.368
Tailing Factor = 3.3
7e+006
6e+006
5e+006
4e+006
3e+006
2e+006
1e+006
0
Time (s)
490
500
510
520
530
540
550
560
570



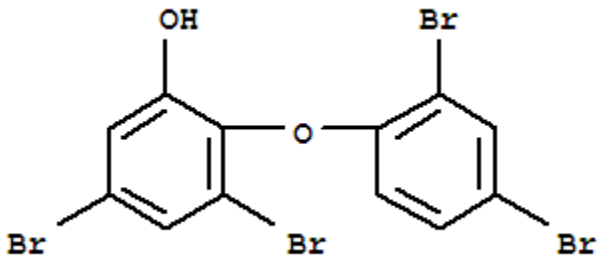
Bisphenol A



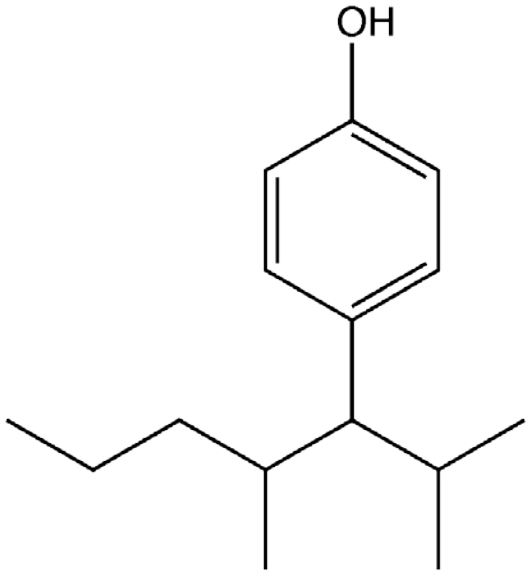
Tetrabromobisphenol A



Hydroxy PCB



Hydroxy BDE



Nonyl phenol

Analyte Protectants Summary for Splitless Injection GC

- Approach shows promise for better GC of active compounds, including POPs
  - Increased response factors and less peak tailing
- Less successful for reactive compounds
  - Endrin and DDT still subject to degradation
- Additional analyte protectants need testing
  - GC-MS will increase protectant choices

Splitless Injection GC Issues

- Active/polar/thermal sensitive analytes
- Dirty samples

- Matrix enhanced/degraded responses
- Limited transfer from GC inlet to column
- Poor detectability
- Significant quantification bias

- Frequent GC inlet and column maintenance
- Offline cleanup

Split injection

Matrix *effect* diluted to the point where it doesn't negatively impact *GC inlet* efficiency

Shoot-and-Dilute GC

Relies on detector sensitivity and selectivity improvements, e.g. MS/MS, HRMS

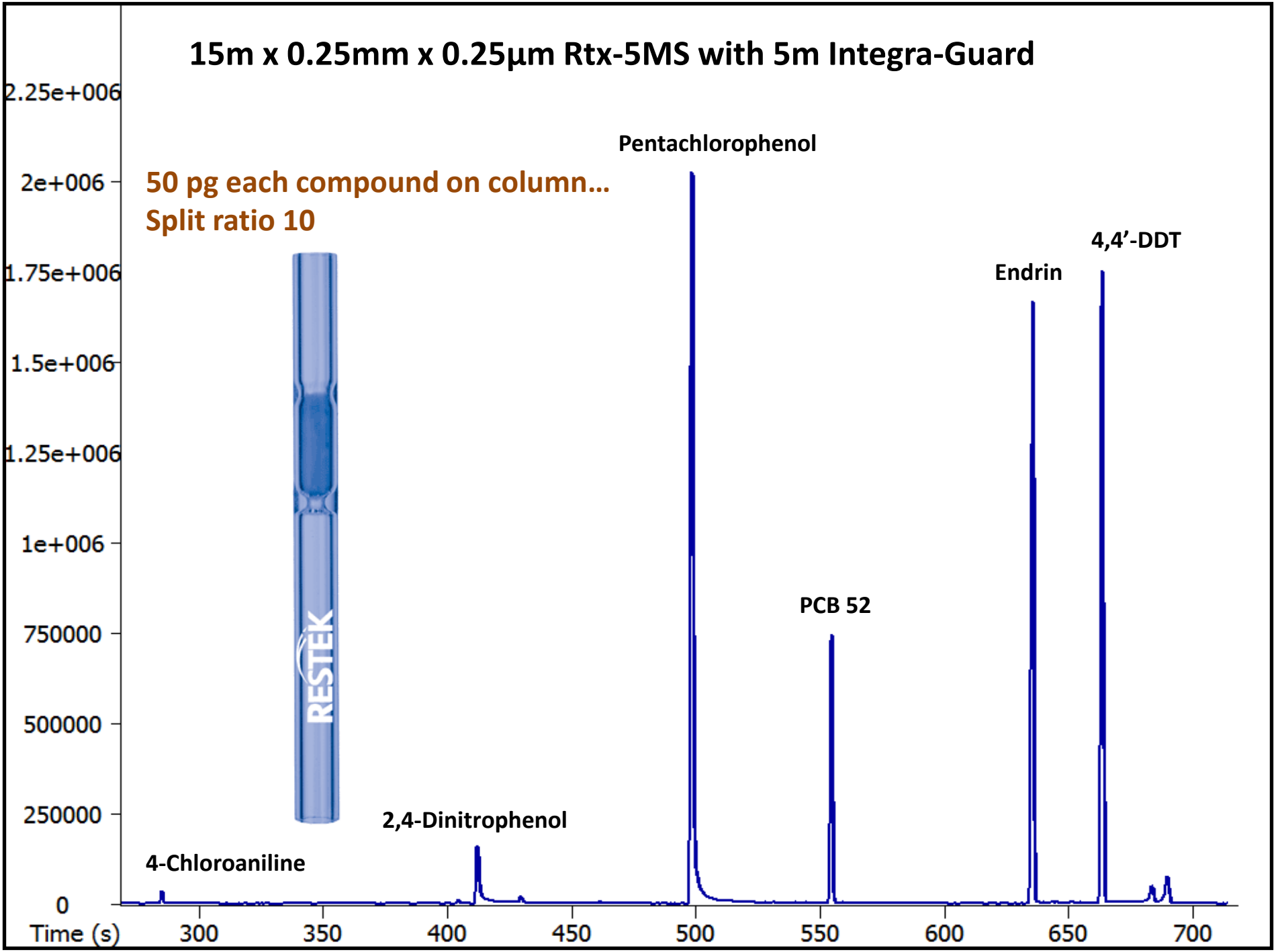
- Frequent GC inlet and column maintenance
- Offline cleanup

Sky® 4mm ID Single Taper Inlet Liner with Quartz Wool Splitless Injections

Sky® 4mm ID Precision® Inlet Liner with Quartz Wool Split Injections

- Goal is for part of sample to make it to GC column
  - Based on split ratio
- Wool wipes needle and helps homogenize sample
  - Excellent for repeatability
  - High split ratio reduces wool and “dirt” impact on compounds prone to breakdown or sorption losses

15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard

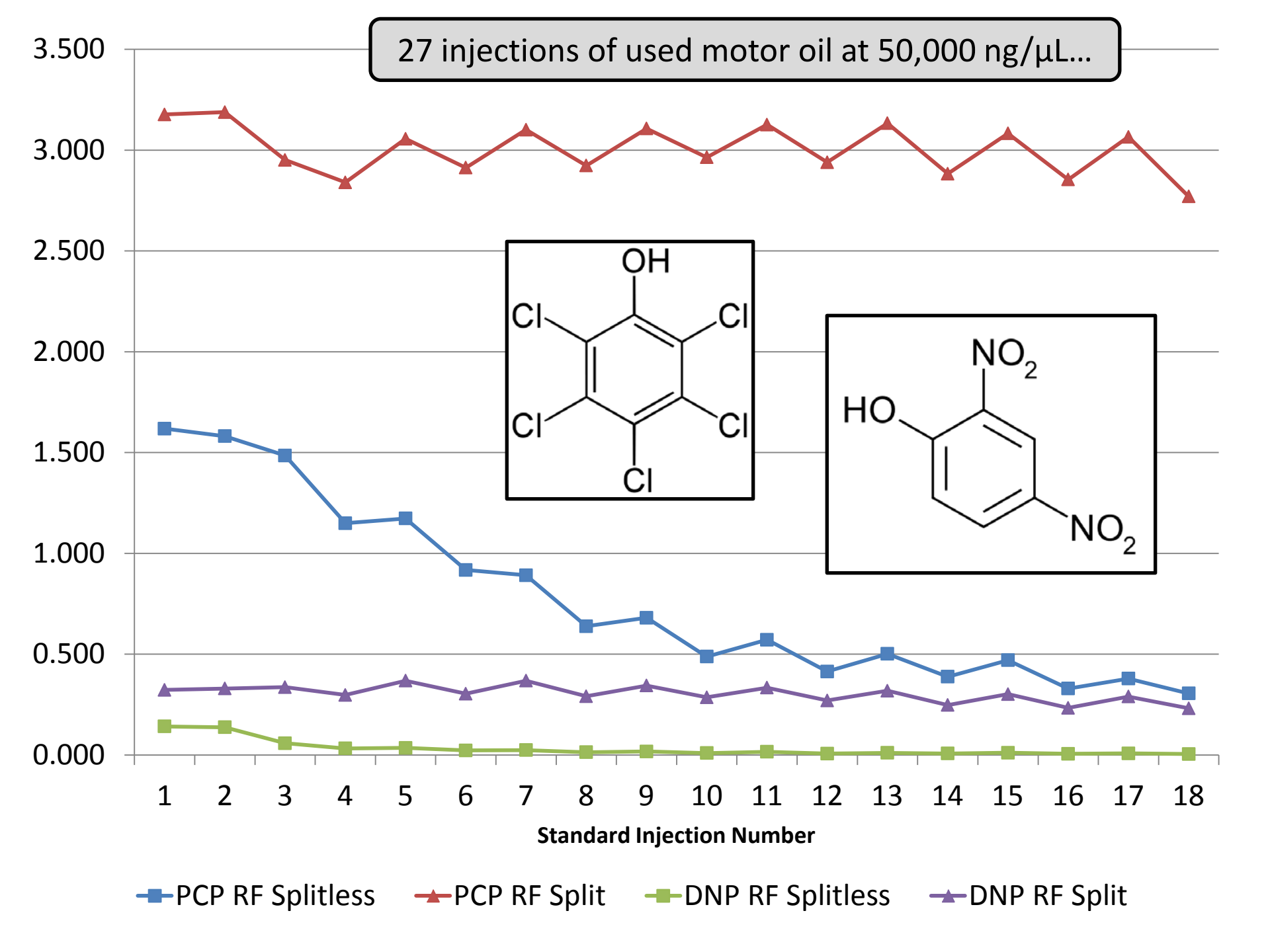


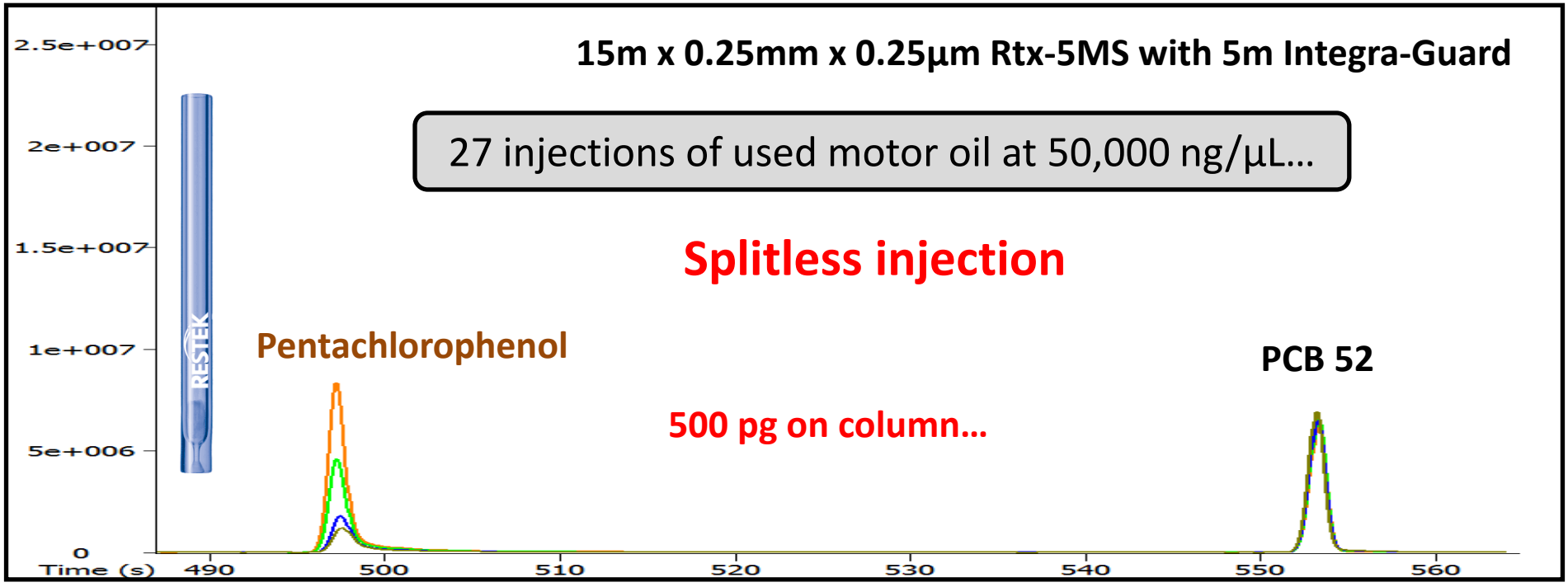
Splitless versus Split Injection GC Ruggedness Experiments

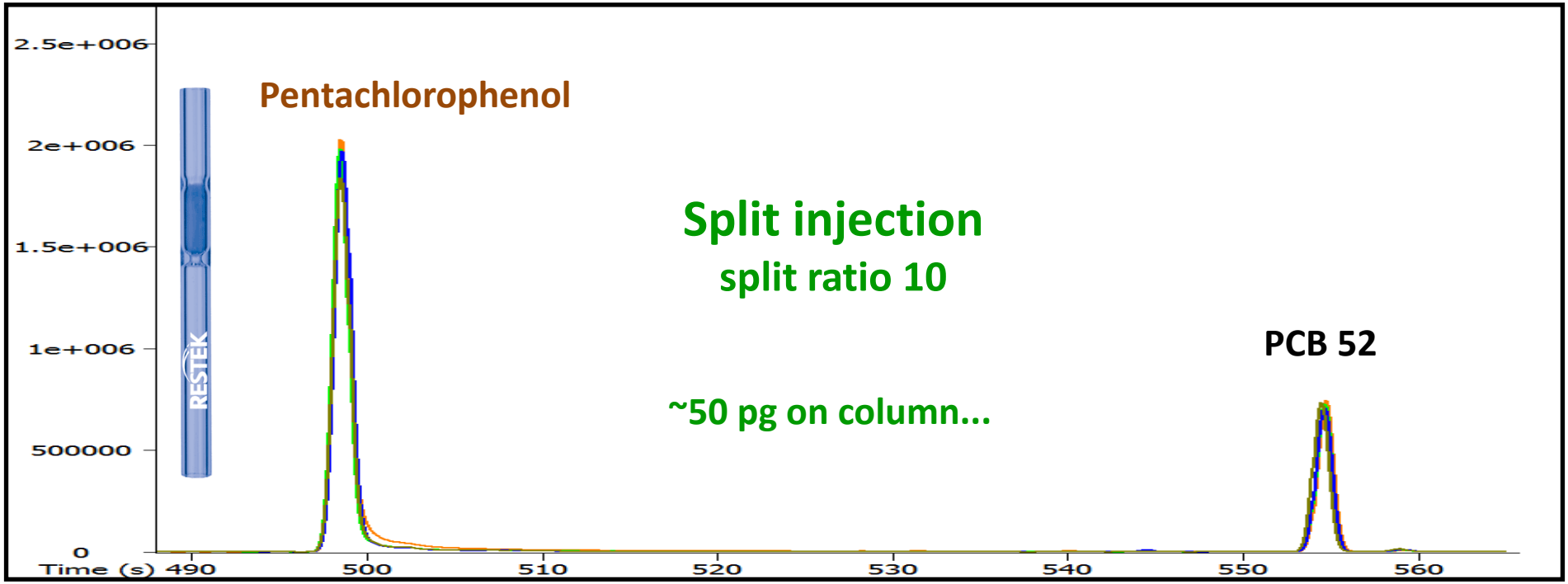
- Standard × 2
- Used Motor Oil × 1
- Standard × 2
- Used Motor Oil × 2
- Standard × 2
- Used Motor Oil × 4
- Standard × 2
- Used Motor Oil × 4
- Standard × 2
- Used Motor Oil × 4
- Standard × 2
- Used Motor Oil × 4
- Standard × 2
- Used Motor Oil × 4
- Standard × 2
- And more...

DNP PCP PCB Standard = 0.5 ng/µL

Used Motor Oil = 50,000 ng/µL


27 injections of used motor oil at 50,000 ng/µL…
3.500
3.000
2.500
2.000
1.500
1.000
0.500
0.000
OH
Cl
Cl
Cl
Cl
Cl
NO2
HO
NO2
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
Standard Injection Number
PCP RF Splitless
PCP RF Split
DNP RF Splitless
DNP RF Split


15m x 0.25mm x 0.25µm Rtx-5MS with 5m Integra-Guard
27 injections of used motor oil at 50,000 ng/µL…
Splitless injection
Pentachlorophenol
PCB 52
500 pg on column…
2.5e+007
2e+007
1.5e+007
1e+007
5e+006
0
Time (s)
490
500
510
520
530
540
550
560
RESTEK


Pentachlorophenol
Split injection
split ratio 10
PCB 52
~50 pg on column…
2.5e+006
2e+006
1.5e+006
1e+006
500000
0
Time (s)
490
500
510
520
530
540
550
560
RESTEK

15m x 0.25mm x 0.25µm Rtx-5MS with 4.5m Integra-Guard

Pentachlorophenol

PCB 52

Split injection

~20 pg on column…

With analyte protectant

Without analyte protectant

1e+006
800000
600000
400000
200000
0
Time (s)
490
500
510
520
530
540
550
560

2,4-Dinitrophenol

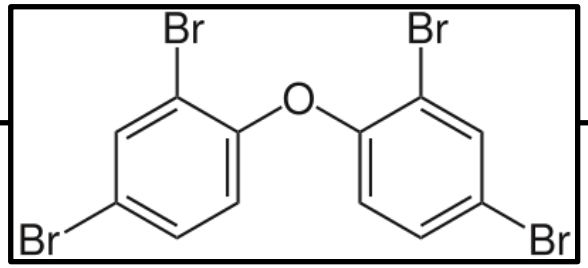
Pentachlorophenol

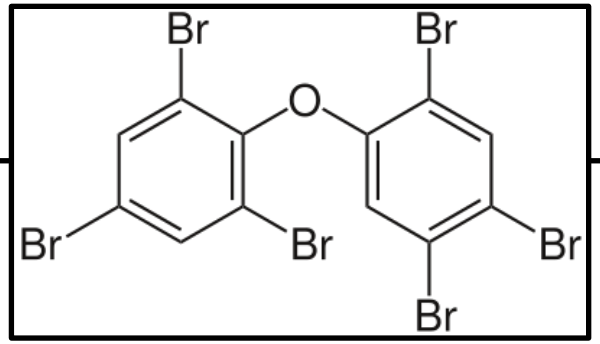
PCB 52

With analyte protectant

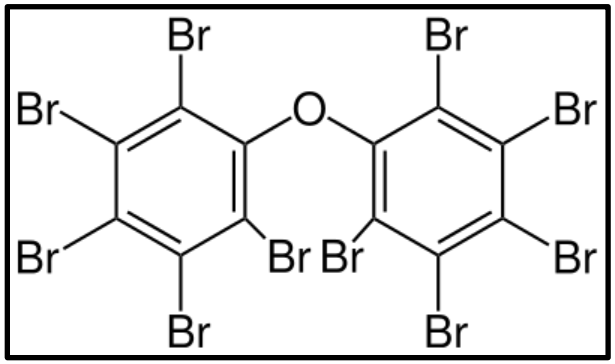
Without analyte protectant

120000
100000
80000
60000
40000
20000
0
Time (s)
400
420
440
460
480
500
520
540
560

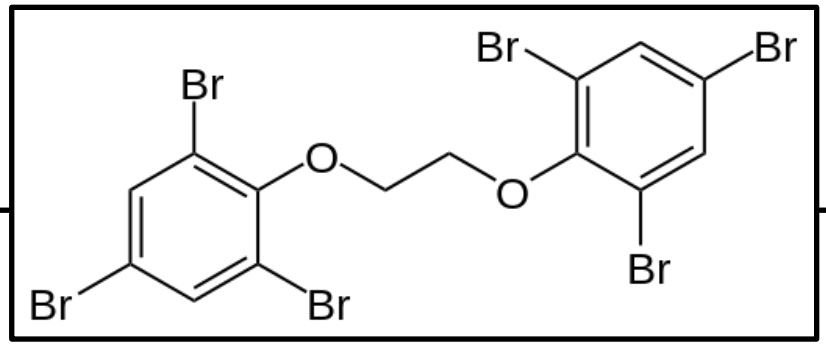


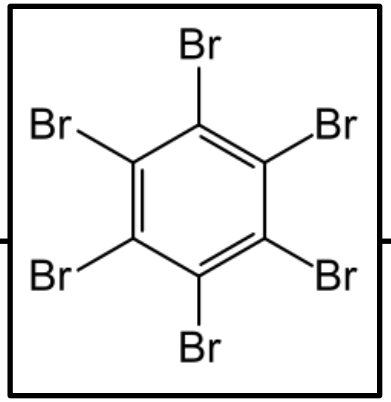


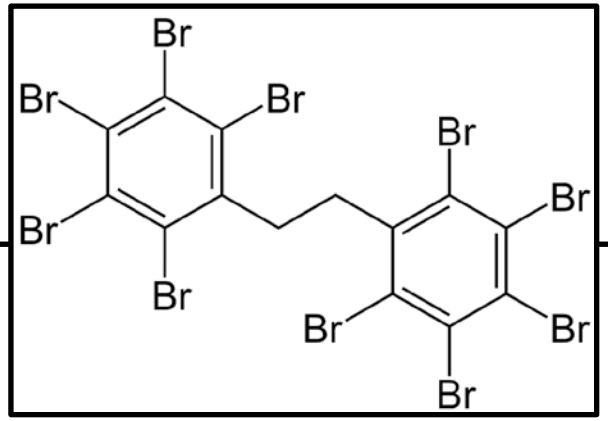
EPA
United States
Environmental Protection
Agency

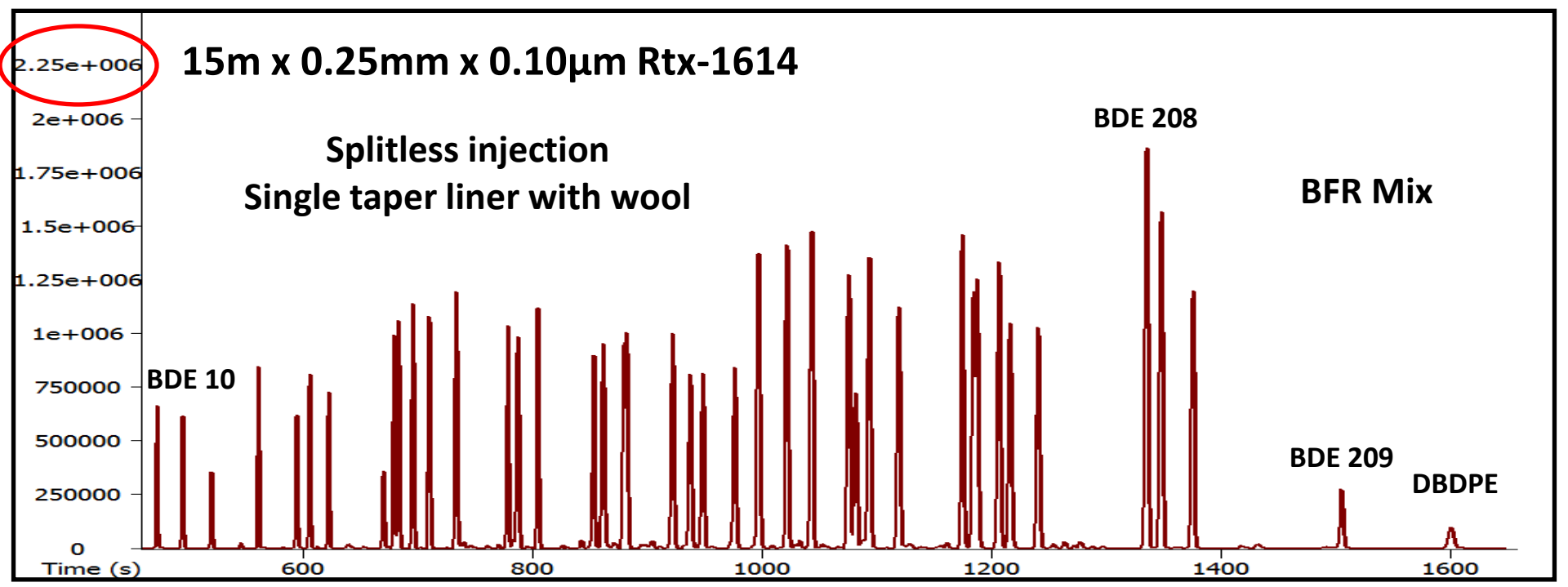


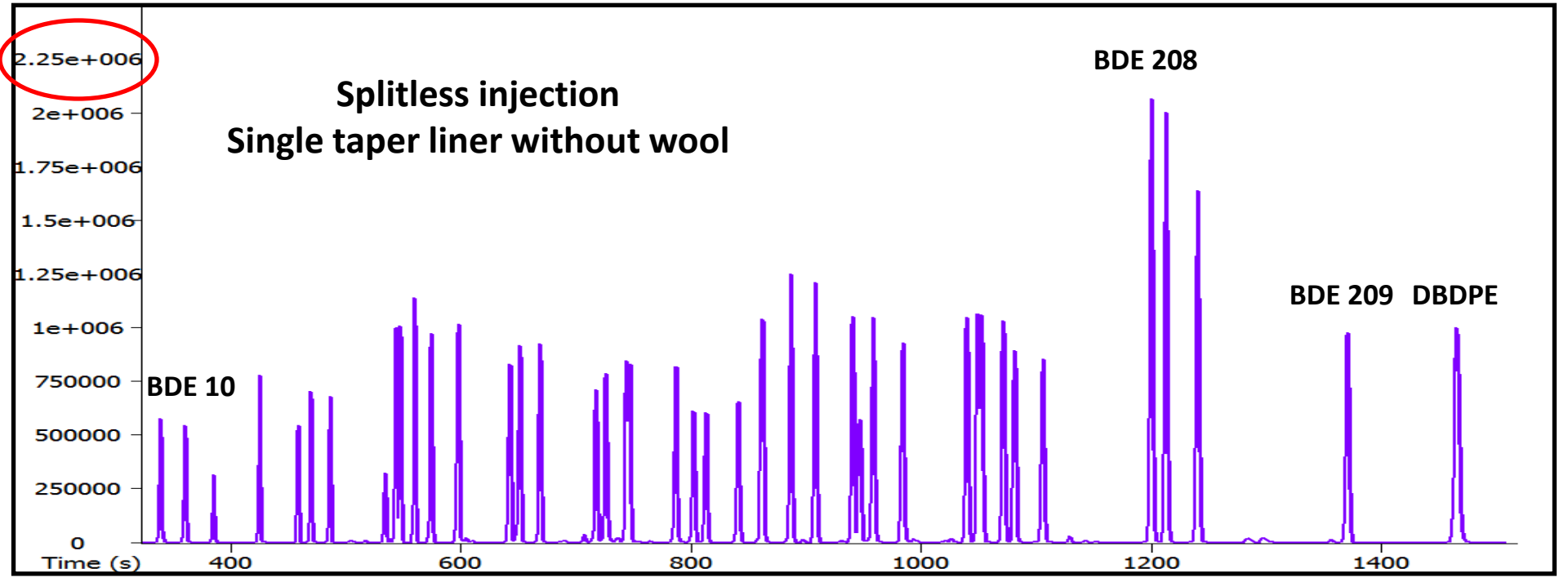
Method 1614
Brominated Diphenyl Ethers in Water Soil, Sediment and Tissue by HRGC/HRMS

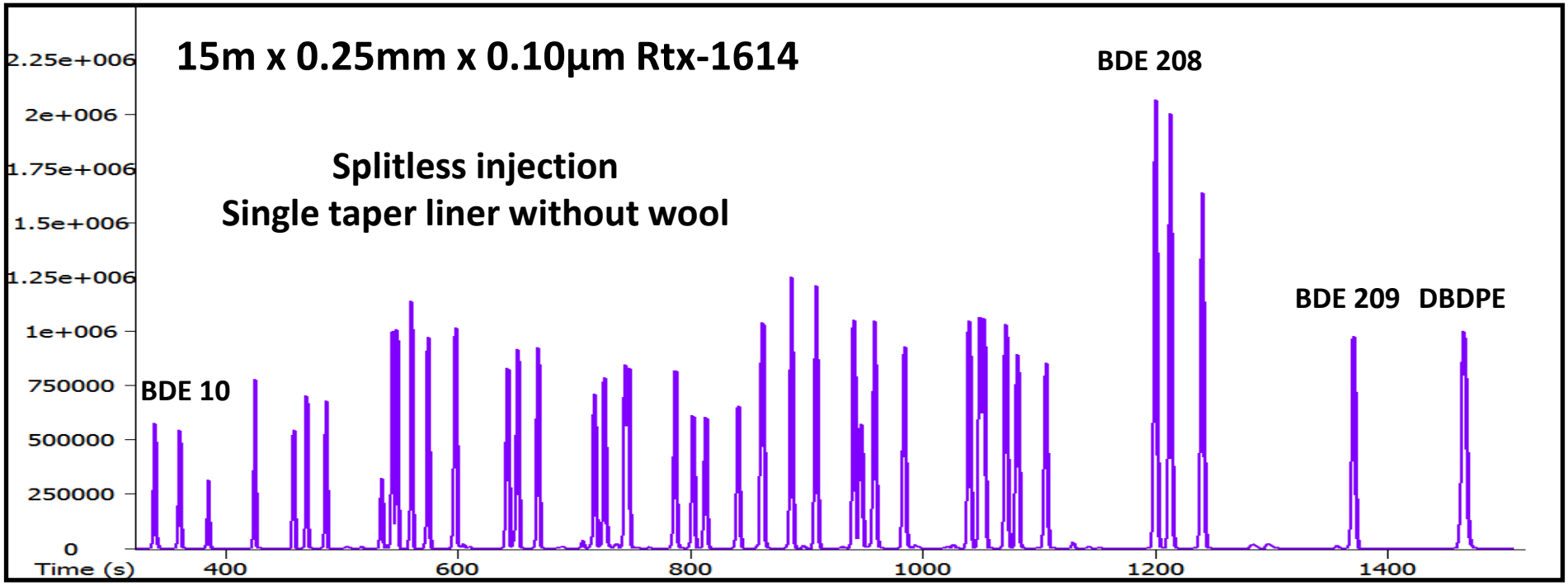


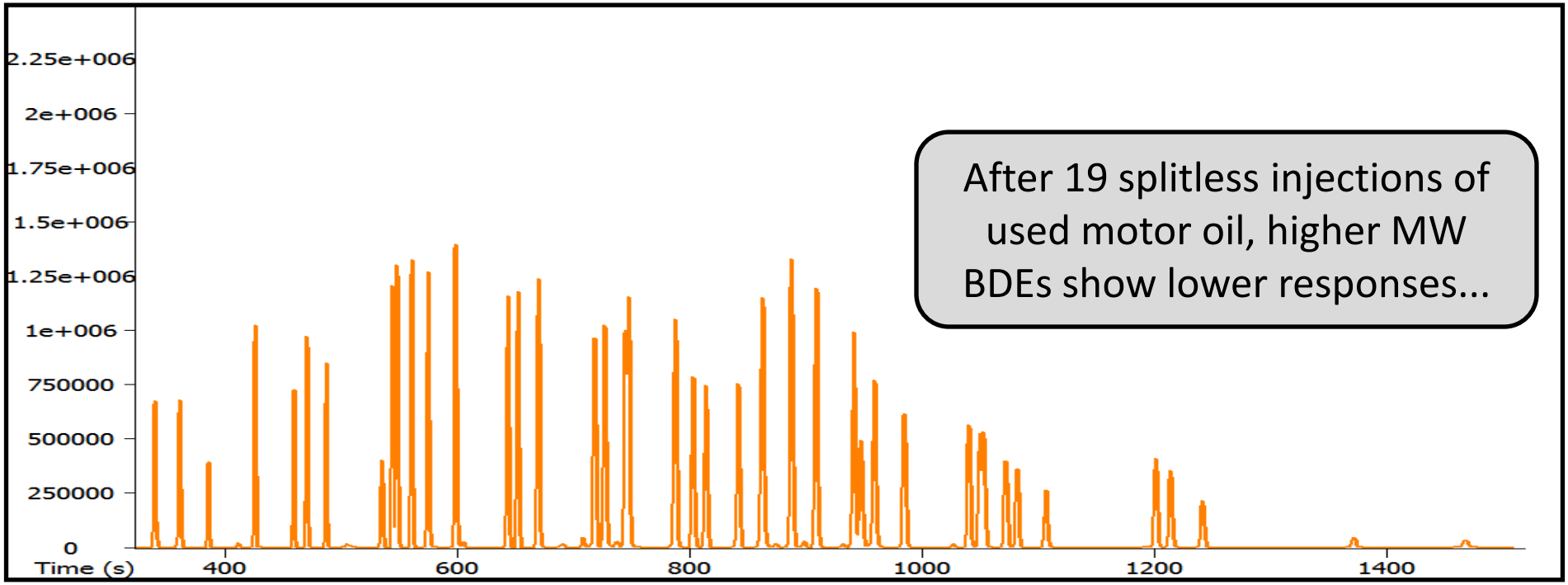


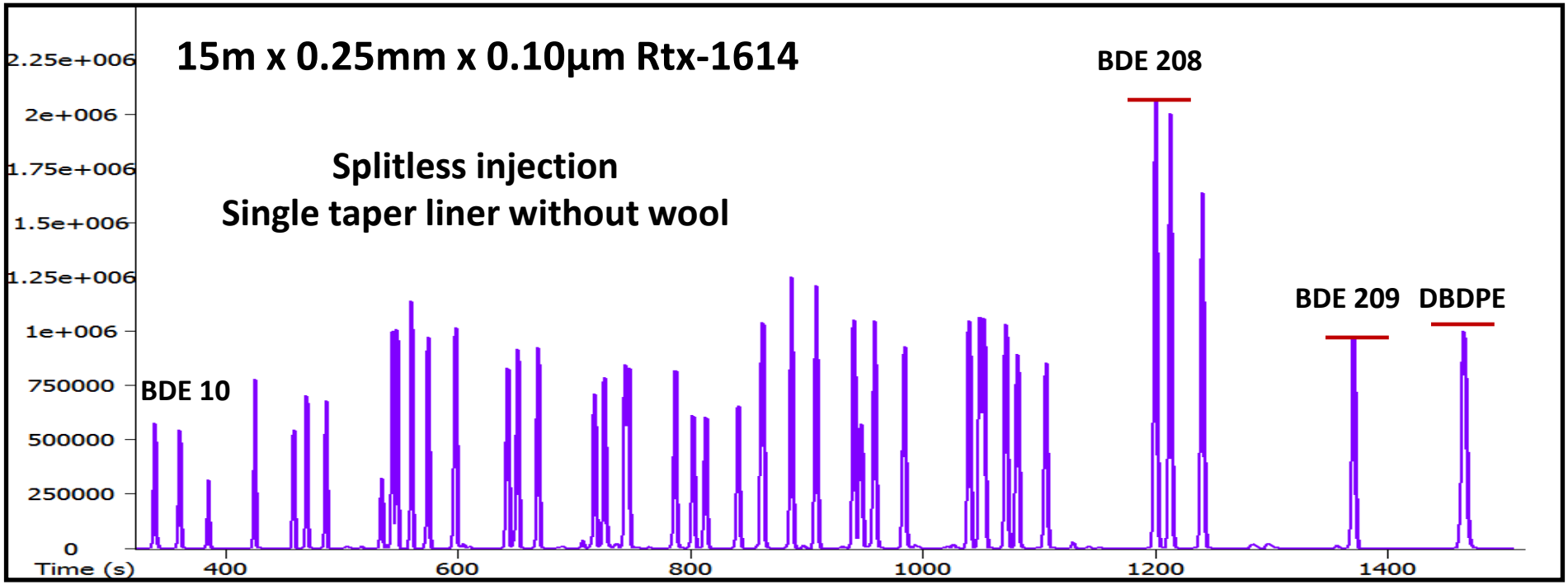


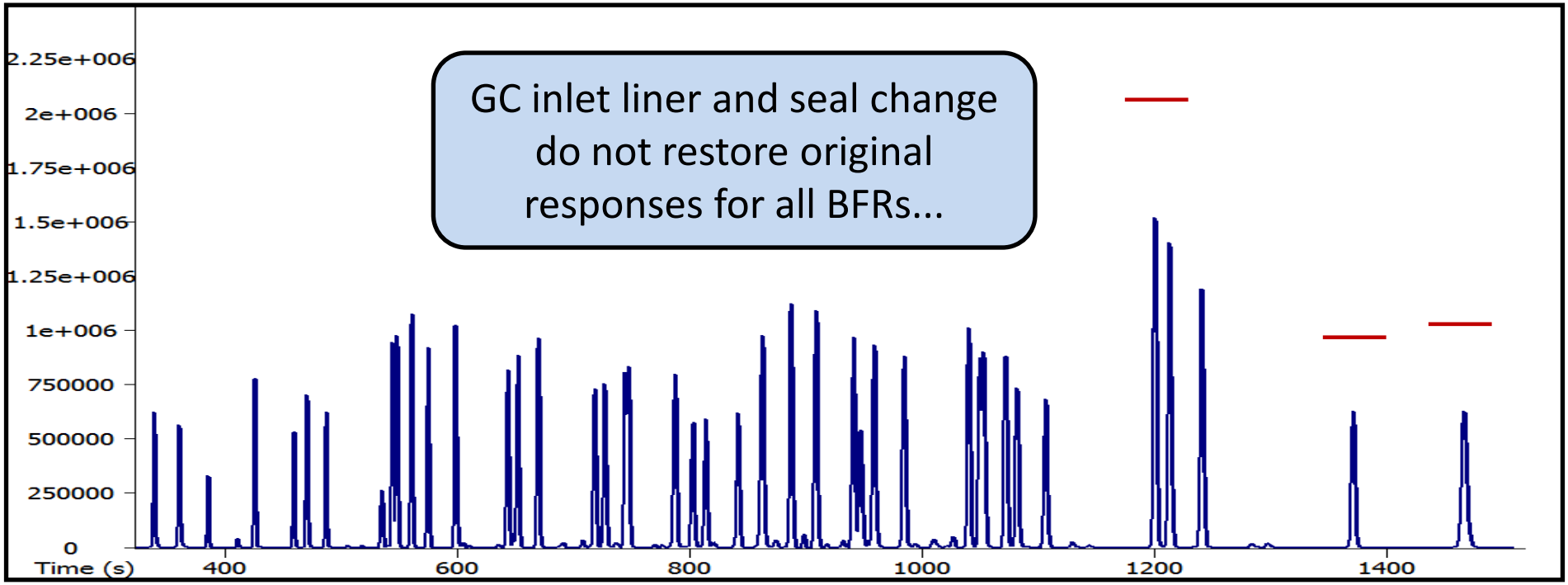

15m x 0.25mm x 0.10µm Rtx-1614
Splitless injection
Single taper liner with wool
BFR Mix
BDE 208
BDE 10
BDE 209
DBDPE
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
600
800
1000
1200
1400
1600

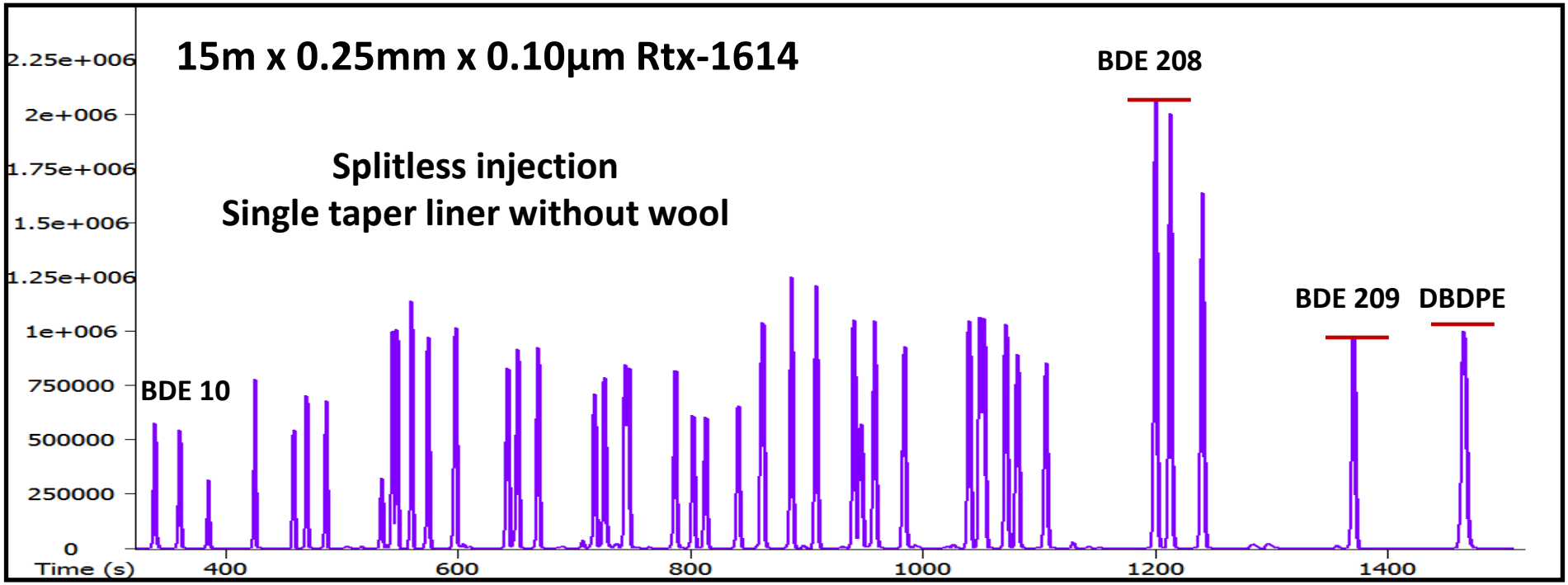

Splitless injection
Single taper liner without wool
BDE 208
BDE 10
BDE 209
DBDPE
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400

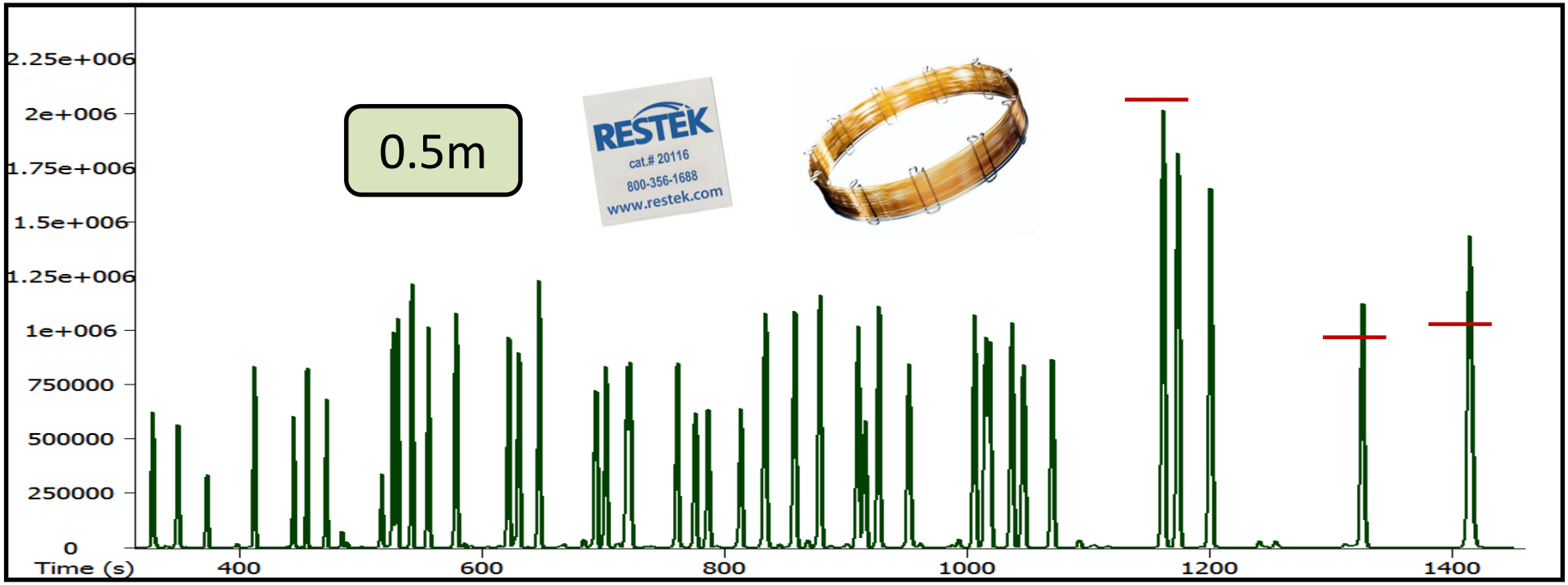

15m x 0.25mm x 0.10µm Rtx-1614
Splitless injection
Single taper liner without wool
BDE 10
BDE 208
BDE 209
DBDPE
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400

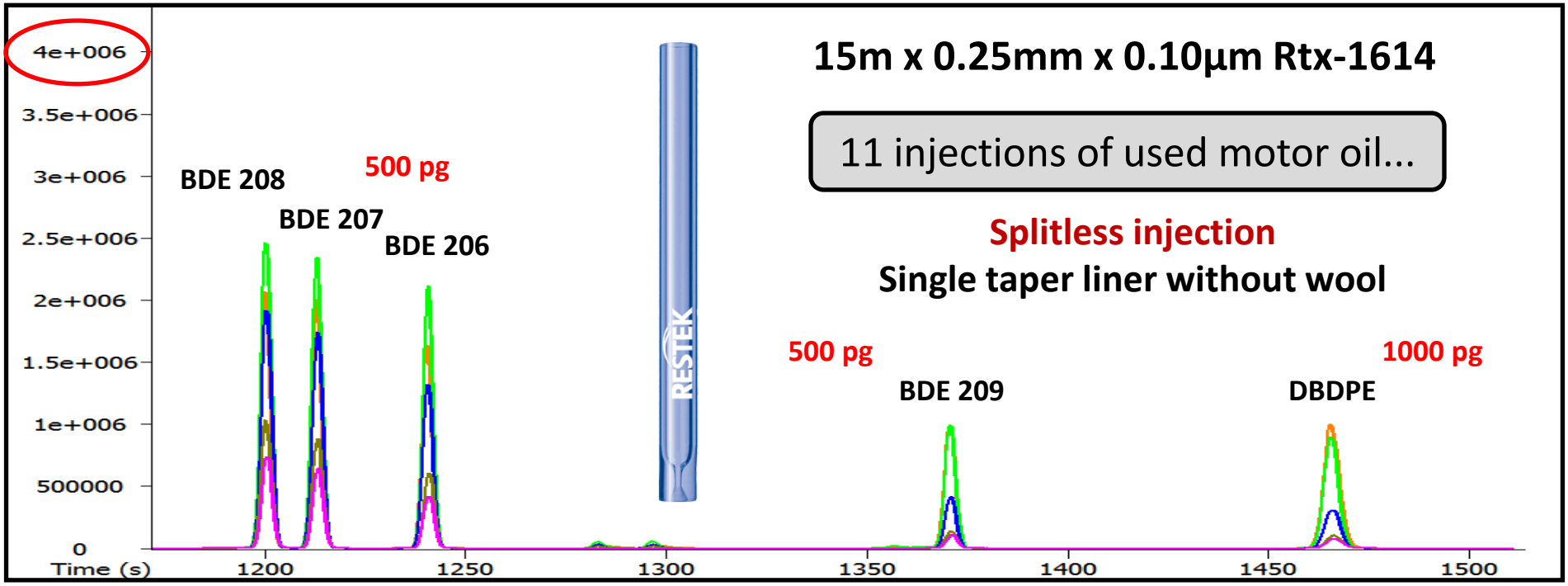

After 19 splitless injections of used motor oil, higher MW BDEs show lower responses...
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400

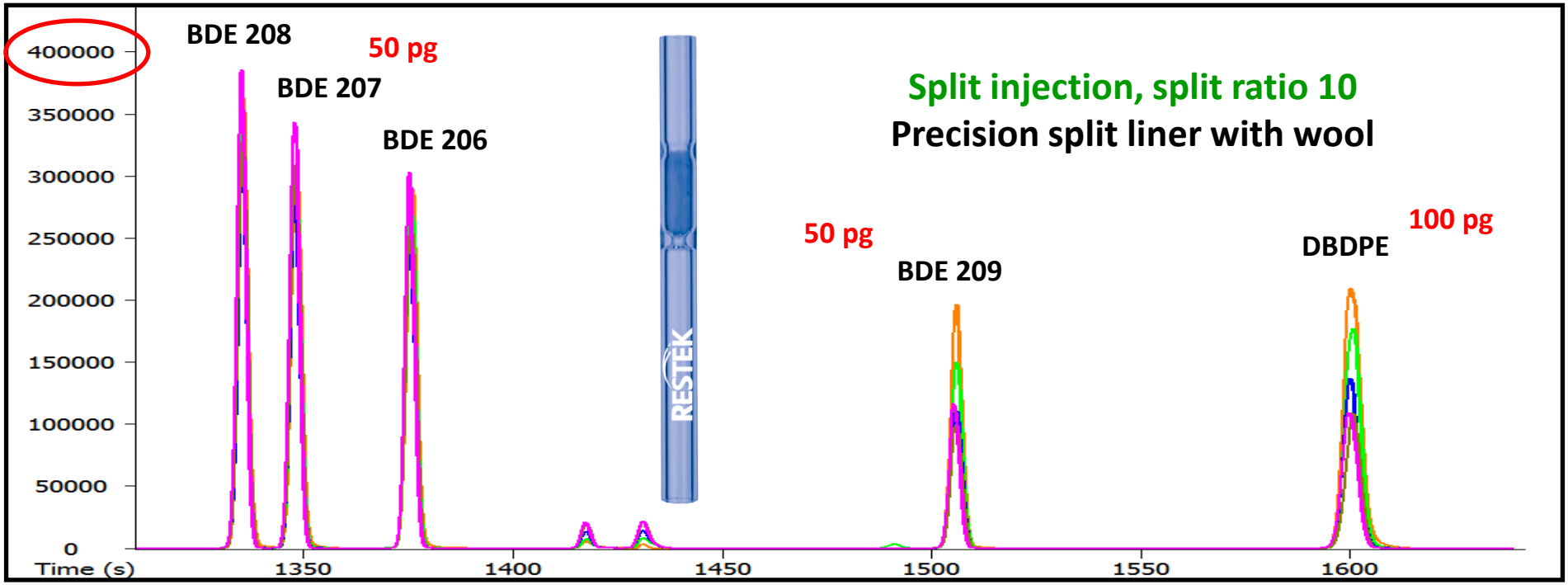

15m x 0.25mm x 0.10µm Rtx-1614
Splitless injection
Single taper liner without wool
BDE 208
BDE 209
DBDPE
BDE 10
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400

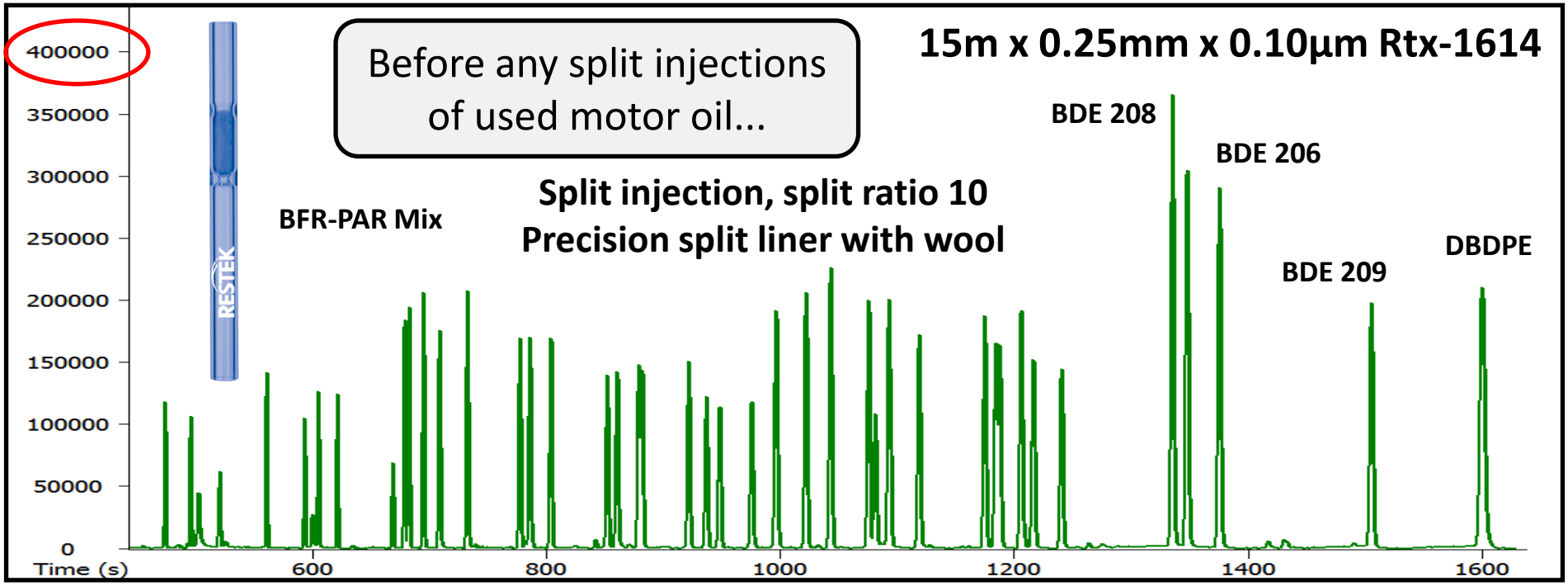

GC inlet liner and seal change do not restore original responses for all BFRs...
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400

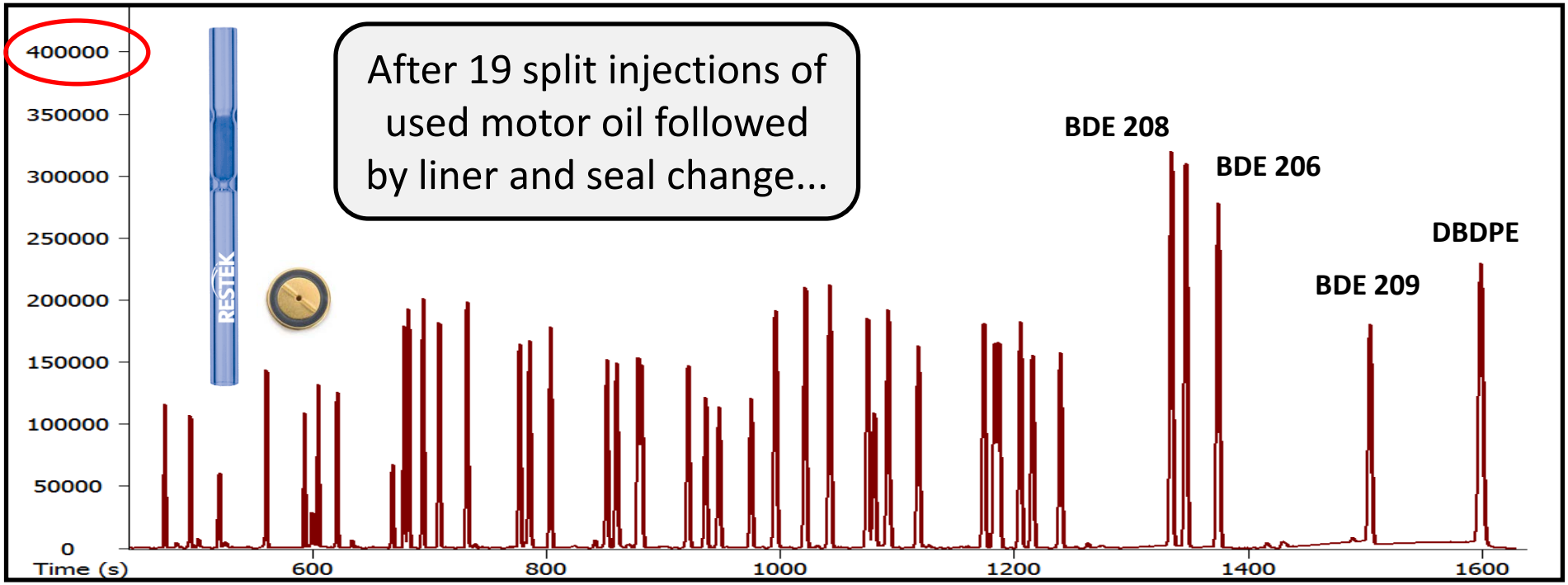

15m x 0.25mm x 0.10µm Rtx-1614
Splitless injection
Single taper liner without wool
BDE 10
BDE 208
BDE 209
DBDPE
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400


0.5m
RESTEK
cat.# 20116
800-356-1688
www.restek.com
2.25e+006
2e+006
1.75e+006
1.5e+006
1.25e+006
1e+006
750000
500000
250000
0
Time (s)
400
600
800
1000
1200
1400


15m x 0.25mm x 0.10µm Rtx-1614
11 injections of used motor oil...
Splitless injection
Single taper liner without wool
BDE 208
BDE 207
BDE 206
500 pg
500 pg
BDE 209
1000 pg
DBDPE
RESTEK
4e+006
3.5e+006
3e+006
2.5e+006
2e+006
1.5e+006
1e+006
500000
0
Time (s)
1200
1250
1300
1350
1400
1450
1500


Split injection, split ratio 10
Precision split liner with wool
BDE 208
BDE 207
BDE 206
50 pg
50 pg
BDE 209
100 pg
DBDPE
RESTEK
400000
350000
300000
250000
200000
150000
100000
50000
0
Time (s)
1350
1400
1450
1500
1550
1600


Before any split injections of used motor oil...
15m x 0.25mm x 0.10µm Rtx-1614
BFR-PAR Mix
Split injection, split ratio 10
Precision split liner with wool
BDE 208
BDE 206
BDE 209
DBDPE
RESTEK
400000
350000
300000
250000
200000
150000
100000
50000
0
Time (s)
600
800
1000
1200
1400
1600


After 19 split injections of used motor oil followed by liner and seal change...
BDE 208
BDE 206
DBDPE
BDE 209
RESTEK
400000
350000
300000
250000
200000
150000
100000
50000
0
Time (s)
600
800
1000
1200
1400
1600

Estimated LOQs Based on 10:1 Split Ratio

EPA Mtd	Ext Type	Smp Amt	Ext Vol	Detector	Det fg	~LOQ
8081B	LLE	1 L	10 mL	ECD	50	5 ppt
8081B	PFE	10 g	10 mL	ECD	50	0.5 ppb
1614	LLE	1 L	20 µL	HRMS DFS	25	5 ppq
1614	Soxhlet	10 g	20 µL	HRMS DFS	25	0.5 ppt
1668A	LLE	1 L	20 µL	HRMS DFS	10	2 ppq
1668A	Soxhlet	10 g	20 µL	HRMS DFS	10	0.2 ppt

Application Note: 30098

DFS - Analysis of Brominated Flame Retardants with High Resolution GC/MS

Dirk Krumwiede, Hans-Joachim Hübschmann, Thermo Fisher Scientific, Bremen, Germany

Shoot-and-Dilute GC Summary

- Split injection can minimize sorptive losses of active compounds
  - Better initial and sustained performance
- Split injection results in a better proportional and sustained transfer of less volatile analytes of interest (e.g., BDE 209)
  - Should lead to better accuracy and uptime
- Split injection reduces the amount of nonvolatile “dirt” placed on a GC column
  - Longer GC column uptime and lifetime

ChromaBLOGraphy

http://blog.restek.com

Only use split injection, never splitless, when detector is sensitive enough to meet LODs and LOQs

ChromaBLOGraphy

Topical and timely insights from top chromatographers.

Visit Restek.com

« GC compound responses lower than expected? Maybe this will help.

When should you use a buffer for HPLC, how does it work and which one to use? »

Keep your GC organochlorine pesticide analysis systems up longer by using split injection.

December 11th, 2013 by Jack Cochran

As noted in earlier blogs, "Fast Organochlorine Pesticide Analysis Using Hydrogen Carrier Gas with Split Injection GC-ECD" and "Split Inje... lzes Endrin and ...e pesticides DDT Breakdown for GC-ECD Analysis of Organochlorine Pesticides", split injection is a viable alternative to splitless injection for analysi... (e.g. when using EPA Method 8081).

Notable benefits include:

1. Higher throughput due to the ability to start the GC oven at a higher oven temperature (faster analysis time AND less oven...
2. Greater uptime through initial, and ongoing, decreased breakdown of sensitive pesticides Endrin and DDT (faster flow... splitless injection decreases compound residence time and chance for negative reactions or sorption).

Even after 145 used motor oil injections (4500 ng/µL – 1 µL injected, split 50:1), which led to heavily contaminated... work, EPA Method 8081 Endrin and DDT breakdown criteria were still easily met. In this case, used motor oil m... material that collects in the inlet liner and the front part of the GC column. This "dirt" is the common cause for... corruption in GC.

If you're worried about meeting detectability requirements at a higher split ratio, don't. The Agilent micr... post above to confirm.