Advanced Analytical Technologies for Analyzing Environmental Matrixes Contaminated with Petroleum Hydrocarbons

Sample Preparations

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Outline

- Legislation and Established procedures
  - **FDA and NOAA Legislation**
    - > Parameters used to measure contamination
    - > Sample preparation procedure

> QuEChERS sample preparation approach

- Sample preparation
- Analysis

References

”Extraction, Cleanup, and Gas Chromatography/Mass Spectrometry Analysis of Sediments and Tissues for Organic Contaminants”, Sloan, C.A., Brown, D.W., Pearce, R.W., Boyer, R.H., Bolton, J.L., Burrows, D.G., Herman, D.P., and Krahn, M.M.U.S. Dept. Commer., NOAA Tech. Memo. NMFS-NWFSC-59, 47 pp., 2004

“Protocol for Interpretation and Use of Sensory Testing and Analytical Chemistry Results for Re-Opening Oil-Impacted Areas Closed to Seafood Harvesting”, 2010_0529_NOAA Opening Protocol Final, 8 pp., 2010

“The Analysis of Poly Aromatic Hydrocarbons in Biota and Sediment Extracts Using GC-MS/MS with the Agilent 7000A GC-QQQ System” Chris Sandy, Agilent Technologies UK, 44 pp, Oct 2009

“GC/MS Analysis of European Union (EU) Priority Polycyclic Aromatic Hydrocarbons (PAHs) using an Agilent J&W DB-EUPAH GC Column with a Column Performance Comparison”, Doris Smith and Ken Lynam, Agilent Technologies, USA, 6 pp, pub 5990-4883EN, Oct 2009.

Legislative Authorities

US Food & Drug Administration (FDA)

- **Operates a mandatory safety program for all fish and fishery products**
  - **Under Federal Food, Drug & Cosmetic Act and related regulations**

National Oceanic and Atmospheric Administration (NOAA)

- **Legislative authority to open & close federal waters for seafood harvest**
  - **Operates the Seafood Inspection Program**

Established Procedures

After an oil spill occurrence

- **Federal and State agencies are faced with the issue of determining when seafood from the previously contaminated area may be safe for harvest and human consumption**
  - **NOAA and FDA work with other federal & state agencies to protect consumers from adulterated and unsafe seafood**
    - > Minimize undue economic burden on impacted seafood industries

Established Procedures

Once oil or chemical contamination visually observed on the surface

- **Recommend fishery closure until free of sheen and analytical testing has occurred: establishing seafood wholesome and safe**

  – **Testing includes: Organoleptic analysis of products (sensory testing)**

  Chemical analysis

    ▪ Fishery closure areas: buffer zone around contaminated waters to account for uncertainty

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Oil Contamination : Risks Assessments

Oil contamination presents 2 kinds of risks:

- **Presence of petroleum tainted seafood rendering unfit for human consumption**
- **Presence of polycyclic aromatic hydrocarbons (PAHs) that are chemical hazards**
  - **Established FDA levels**
    - > Persistence
    - > Potential Toxicity
    - > Carcinogenic effects
  - FDA's "List of 9 PAHs"
    - Including their 16 alkylated homologues

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FDA's " List of Nine-PAHs"

Criteria for Re-opening Areas Closed from Oil Spills Based on 160 g/day Seafood Consumption and Concentrations of Chemical Contaminants in Seafood

Chemical[1]	Level of Concern (ppm)	Basis[2]
Napthalene	20	EPA RfD; 70 kg bw; 160 g/day consumption
Fluorene	20	EPA RfD; 70 kg bw; 160 g/day consumption
Anthracene/phenanthrene	150	EPA RfD; 70 kg bw; 160 g/day consumption
Fluoranthene	0.15	10^{-6} Cancer risk estimate = 0.02B(a)P equivalency
Pyrene	0.025	10^{-6} Cancer risk estimate = 0.13B(a)P equivalency
Benz(a)anthracene	0.2	10^{-6} Cancer risk estimate = 0.014B(a)P equivalency
Chrysene	0.25	10^{-6} Cancer risk estimate = 0.013B(a)P equivalency
Benzo(a)pyrene	0.003	10^{-6} Cancer risk estimate = (34ng/p/d)(70/5yr)/160 g seafood/p/d

[1] Includes alkylated homologues, specifically C-1, C-2, C-3, C-4 napthalenes; C-1, C-2, C-3 fluorenes; C-1, C-2, C-3 anthracenes/phenanthracenes; C-1, C-2 pyrenes

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List of 29 PAHs

Naphthalene
1-Methylnaphthalene
2-Methylnaphthalene
Biphenyl
2,6-Dimethylnaphthalene
Acenaphthylene
Acenaphthene
2,3,5-Trimethylnaphthalene
Fluorene
Dibenzothiophene
Phenanthrene
Anthracene
1-Methylphenanthrene
Fluoranthene
Pyrene
Retene
Benz[*a*]anthracene
Chrysene + Triphenylene *
Benzo[*b*]fluoranthene
Benzo[*j*]fluoranthene + Benzo[*k*]fluoranthene *
Benzo[*e*]pyrene
Benzo[*a*]pyrene
Perylene
Indeno[1,2,3-*cd*]pyrene
Dibenz[*a,h*]anthracene + Dibenz[*a,c*]anthracene *
Benzo[*ghi*]perylene

* These analytes are quantitated and reported as the sum of their concentrations because they co-elute during GC/MS analysis.

NOAA Sample Preparation Procedure

Sediment or tissue sample

Mix a portion of sample with sodium sulfate and magnesium sulfate, then extract with dichloromethane using Accelerated Solvent Extraction

As needed, dry a portion of sample; gravimetrically determine the percent dry weight

Dichloromethane extract of sediment or tissue

For tissue extracts as needed, take 1/2 of extract and evaporate the solvent; gravimetrically determine the percent total extractables

Filter extract through a silica/alumina column, then concentrate it to 1 mL

Concentrated extract

Chromatograph 1/2 of the concentrated extract using size-exclusion HPLC; collect the AH/CH fraction, then concentrate it to 100 µL

Concentrated AH/CH fraction

Analyze fraction using GC/MS for AH quantitation, as needed

Analyze fraction using GC/MS for CH quantitation, as needed

Alternative Procedure: QuEChERS

QuEChERS: **Qu**ick, **E**asy, **Ch**eap, **E**ffective, **R**ugged and **S**afe

- **Initial purpose/validation was to determine pesticides in fruit and vegetables**
- **"QuEChERS works so well with pesticides can it work for other compound extracts"**
- **Advancements in QuEChERS has offered PAH determination in seafood**

  – **Why: Because of its "NAME"**

  > Takes 10 minutes versus overnight for the NOAA method

  ▪ Less time, Less solvent, Less glassware, Less cost, Less solvent disposal, Less subject to error, No chlorinated solvent

  – So let's take a look at QuEChERS

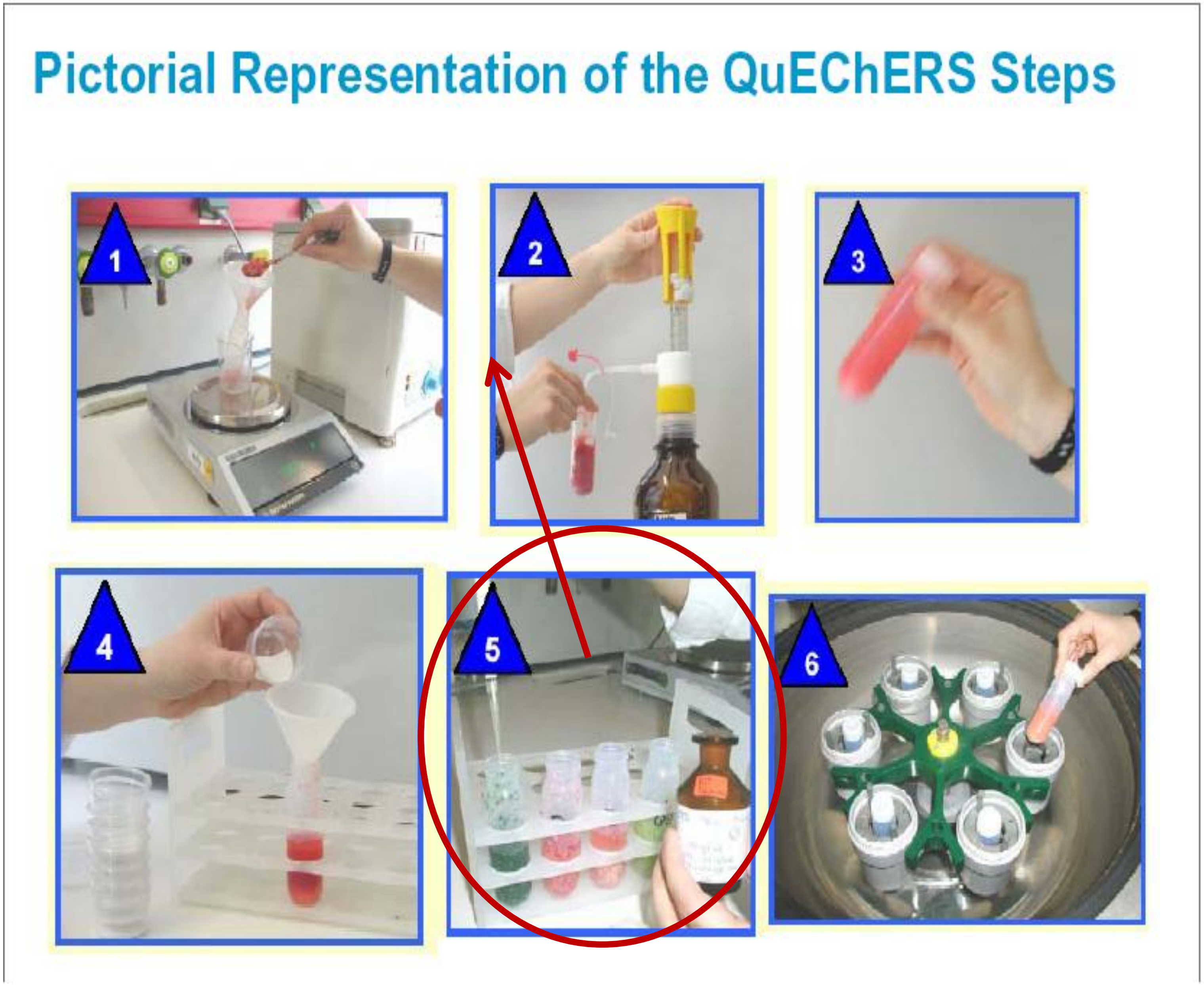
QuEChERS Procedure:

Chop then

Freeze

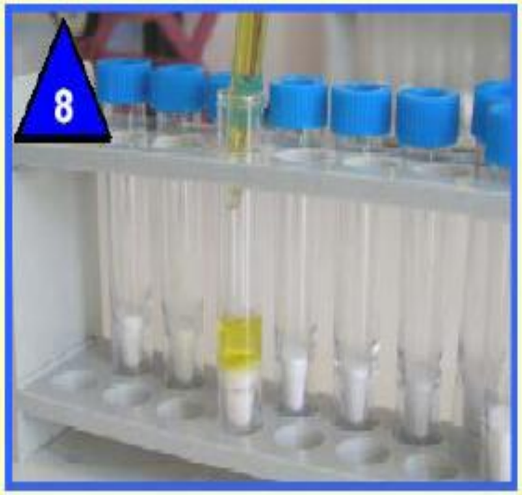
Grind

First Step – Extraction/Partitioning



1) Weigh sample
2) Add Ceramic Homogenizers
2) **Add standards**
3) **Vortex**
4) Add ACN (1% AA)
5) Vortex
6) Add salts
7) Shake 1 min
8) Centrifuge

Second Step – Dispersive SPE



1) Choose d-SPE
2) Transfer volume
3) Vortex 1 min
4) Centrifuge
5) Analyze

QuEChERS and d-SPE Sample Preparation Workflow

Weigh 3 g fish sample (+/- 0.1g) in 50 mL centrifuge tube

↓

Add Surrogate/IS solution, and QC spike solution if necessary, Vortex 1 min

↓

Add 12 mL of DI water and 2 ceramic bars to the sample (Agilent part #5982-9313), Vortex 1 min

↓

Add 15 mL of ACN containing 1%HAc

↓

Vortex 1 min

↓

Add Agilent SampliQ QuEChERS AOAC extraction salt packet (Agilent part #5982-5755)

↓

Cap and shake vigorously for 1 min on Geno/Grinder at1500 rpm

↓

Centrifuge at 4000 rpm for 5 min

↓

Transfer 1 mL of upper ACN layer to SampliQ AOAC Fatty dispersive SPE 2 ml tube (Agilent part # 5982-5122) Or 8 mL to SampliQ AOAC Fatty dispersive SPE 15 mL tube (Agilent part # 5982-5158)

↓

Vortex 1 min, Centrifuge at 13000 rpm for 2 min for 2 mL tubes, Or at 4000 rpm for 5 min for 15 mL tubes

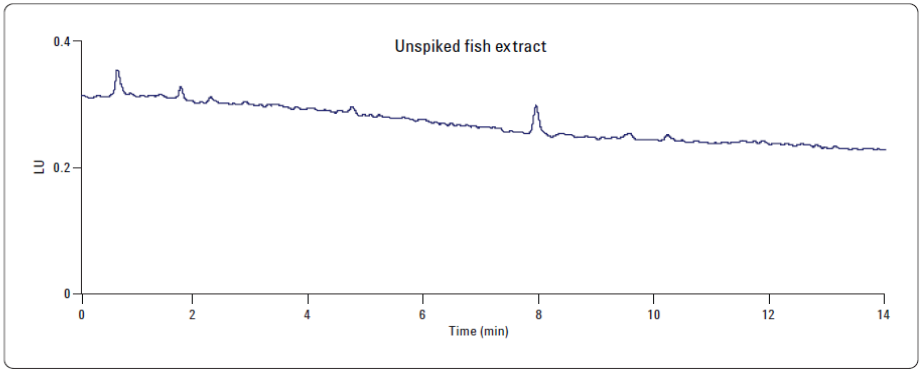
↓

Transfer 500 µL extract to autosampler vial

↓

Analyze by LC/FLD or GC/MS

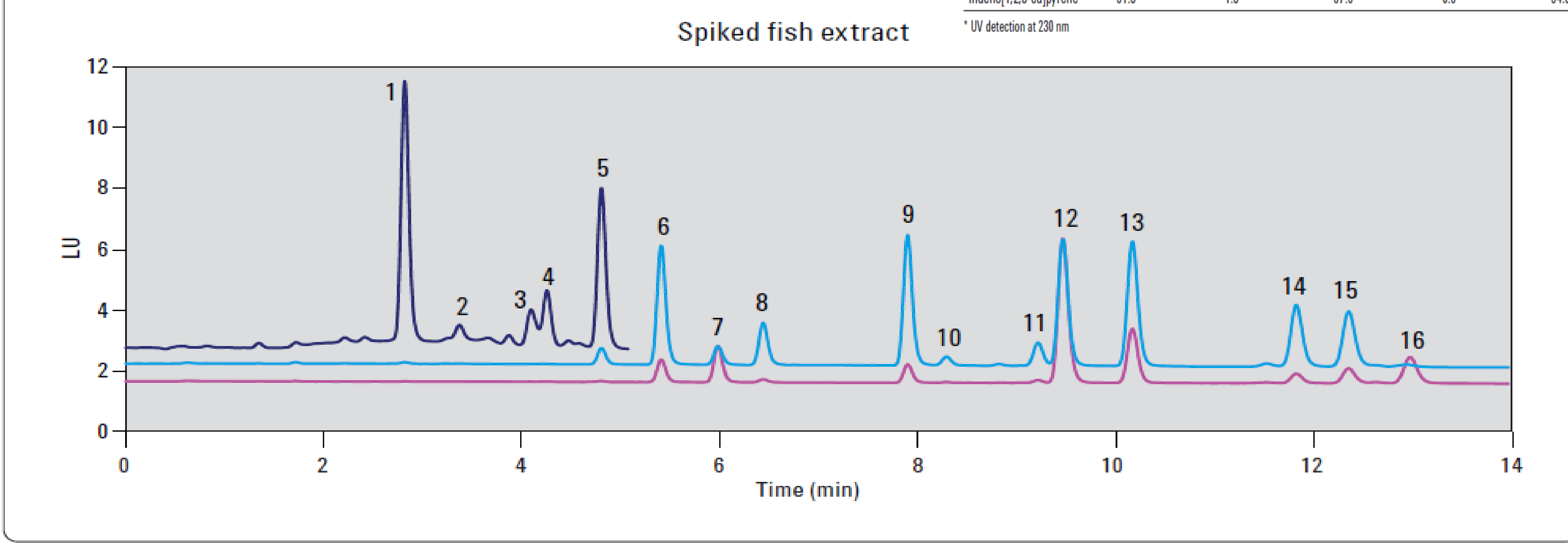
PAH Analysis by LC/FLD



Recoveries and RSDs for the Sixteen Polycyclic Aromatic Hydrocarbons in Fish Sample (n = 6)

PAH	Level of spiking (ng/g) (n = 6)					
	1		2		3	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Naphthalene	94.7	1.4	97.9	1.1	93.8	1.4
*Acenaphthylene	87.8	1.7	96.3	1.2	85.6	0.8
Acenaphthene	92.1	1.5	93.0	1.8	96.7	0.8
Fluorene	98.1	1.5	89.9	1.0	97.2	0.9
Phenanthrene	90.6	0.9	93.8	0.8	83.1	1.7
Anthracene	96.7	1.0	87.6	0.8	92.1	0.6
Fluoranthene	83.4	1.3	93.9	1.5	95.9	1.2
Pyrene	93.5	1.8	86.1	1.3	95.0	1.4
1,2-Benzanthracene	94.5	1.3	89.6	1.6	94.9	1.0
Chrysene	101.0	1.4	97.8	1.7	87.2	1.6
Benzo[e]pyrene	88.8	1.5	85.2	1.9	95.0	1.4
Benz[e]acenaphthylene	95.5	0.7	92.7	0.7	89.2	0.9
Benzo[k]fluoranthene	93.5	0.8	94.6	0.9	98.9	0.8
Dibenzo[a,h]anthracene	88.2	0.9	97.3	1.1	97.1	0.6
Benzo[g,h,i]perylene	98.4	0.8	95.5	1.6	98.2	0.7
Indeno[1,2,3-cd]pyrene	91.5	1.5	97.9	0.9	94.3	0.7

* UV detection at 230 nm



Overlay HPLC – FLD chromatograms of the spiked fish sample containing: 1. Nap 2. Acy 3. Ace 4. Flu 5. Phe 6. Ant 7. Fln 8. Pyr 9. BaA 10. Chr 11. BeP 12. BeA 13. BkF 14. DahA 15. BghiP 16. InP. The spiking level for this sample was level 1 The blue portion of the chromatogram used the following excitation/emission wavelengths: 260-nm/352-nm; the red portion 260-nm/420-nm; the light blue portion: 260-nm/440-nm. For acenaphthylene, UV detection at 230-nm was used

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Part II
PAH Analyzers
GC-Q and GC-QQQ

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July, 08, 2010

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References

”Extraction, Cleanup, and Gas Chromatography/Mass Spectrometry Analysis of Sediments and Tissues for Organic Contaminants”, Sloan, C.A., Brown, D.W., Pearce, R.W., Boyer, R.H., Bolton, J.L., Burrows, D.G., Herman, D.P., and Krahn, M.M.U.S. Dept. Commer., NOAA Tech. Memo. NMFS-NWFSC-59, 47 pp., 2004

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“Analysis of polycyclic aromatic hydrocarbons in fish: evaluation of a quick, easy, cheap, effective, rugged, and safe extraction method”, Ramalhosa M.J. et al, Journal of Separation Science, 2009, 32, 3529-3538

PAH Analyzer(s), 7890GC-7000B QQQ and GC-5975C Q

1. Compatible with **QuEChERS,** which is a fast and simple sample prep technique
2. Capillary Flow Technology based **backflush** reduces system maintenance needs even with dirty matrices. Method parameters are pre-set.
3. PAH **MRM acquisition method (QQQ)** has been optimized and preloaded
4. PAH **SIM target and qualifier ions (Q)** set in acquisition and data analysis
5. Analyzer is offered as a **turnkey system** that has been factory configured and undergone chemical testing prior to shipment
6. PAH **calibration standards** and **ISTDs** are included, reducing start up time
7. PAH-specific column used for **optimized PAH separation**

PAH Method for Productivity, GC-QQQ and GC-Q

1. **Multimode Inlet** for versatility. S/SL could be used for hot splitless PAHs but the MMI offers large volume injection if needed. Cold splitless also available when the system is used for thermally labile compounds.

2. **PAH specific column**, 20m x 0.18mm x 0.14um DB-EUPAH, p/n 121-9627. This offers separations that a DB5-MS does not, but the DB5-MS could be used. Run time is 18 minutes.

3. **Retention Time Locking** done on the method and column shipped. The system only needs to be relocked on installation.

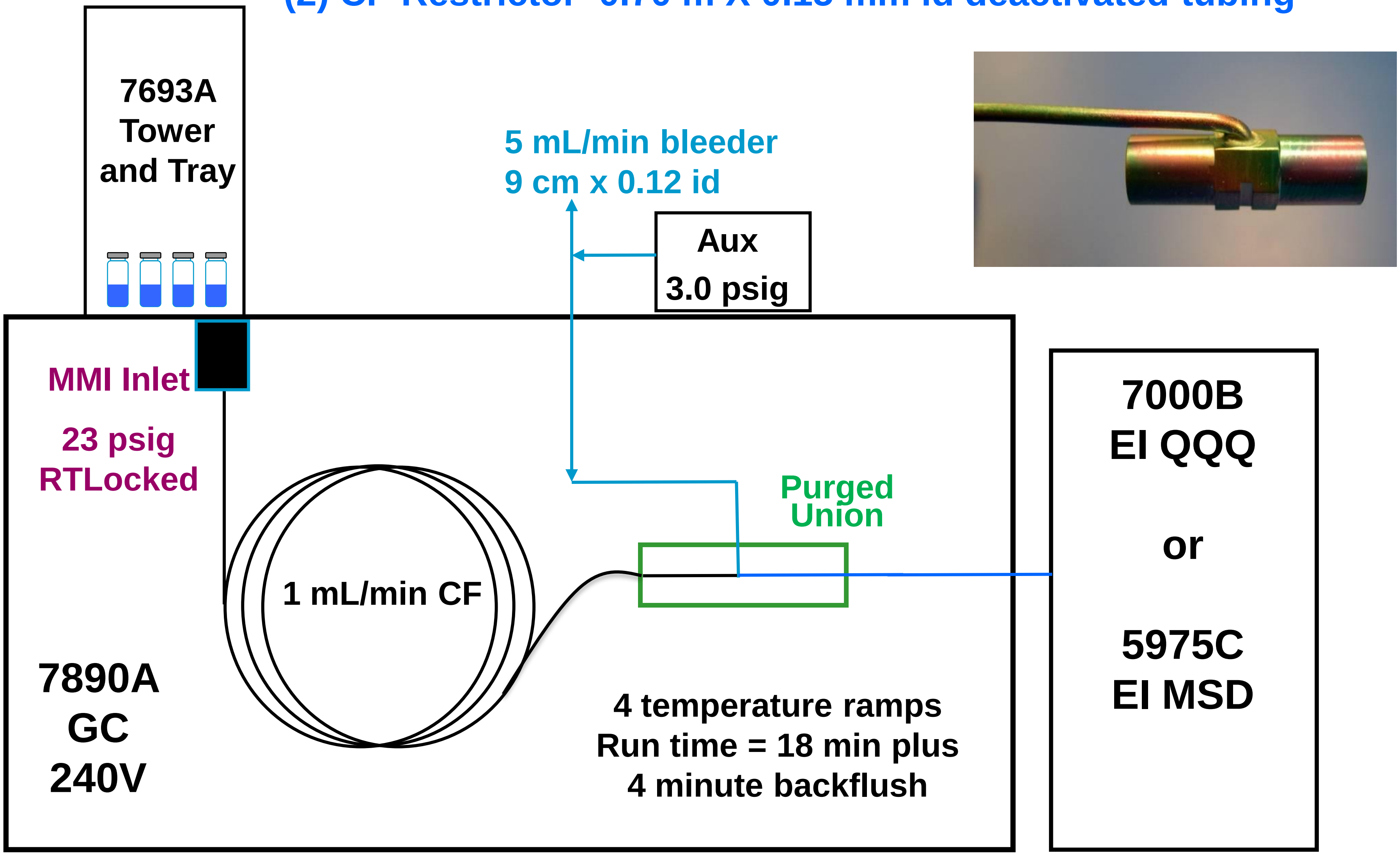
4. **Backflushing** is done via a Capillary Flow Technology purged union connected post column. Cycle time is reduced as column bake-out is eliminated. Source cleaning is reduced.

5. **SIM target ion (Q)** is the most abundant and qualifier ions are the next 3 most abundant. These can be optimized against matrix background using the Ion Optimization program in the latest software release.
6. **MRM (QQQ)** optimization is ongoing with collaborators

GC-QQQ (or GC-Q) PAH Analyzer

(1) CF Column 20 m X 0.18 mm id X 0.14 um DB-EUPAH part# 121-9627

(2) CP Restrictor 0.70 m X 0.15 mm id deactivated tubing



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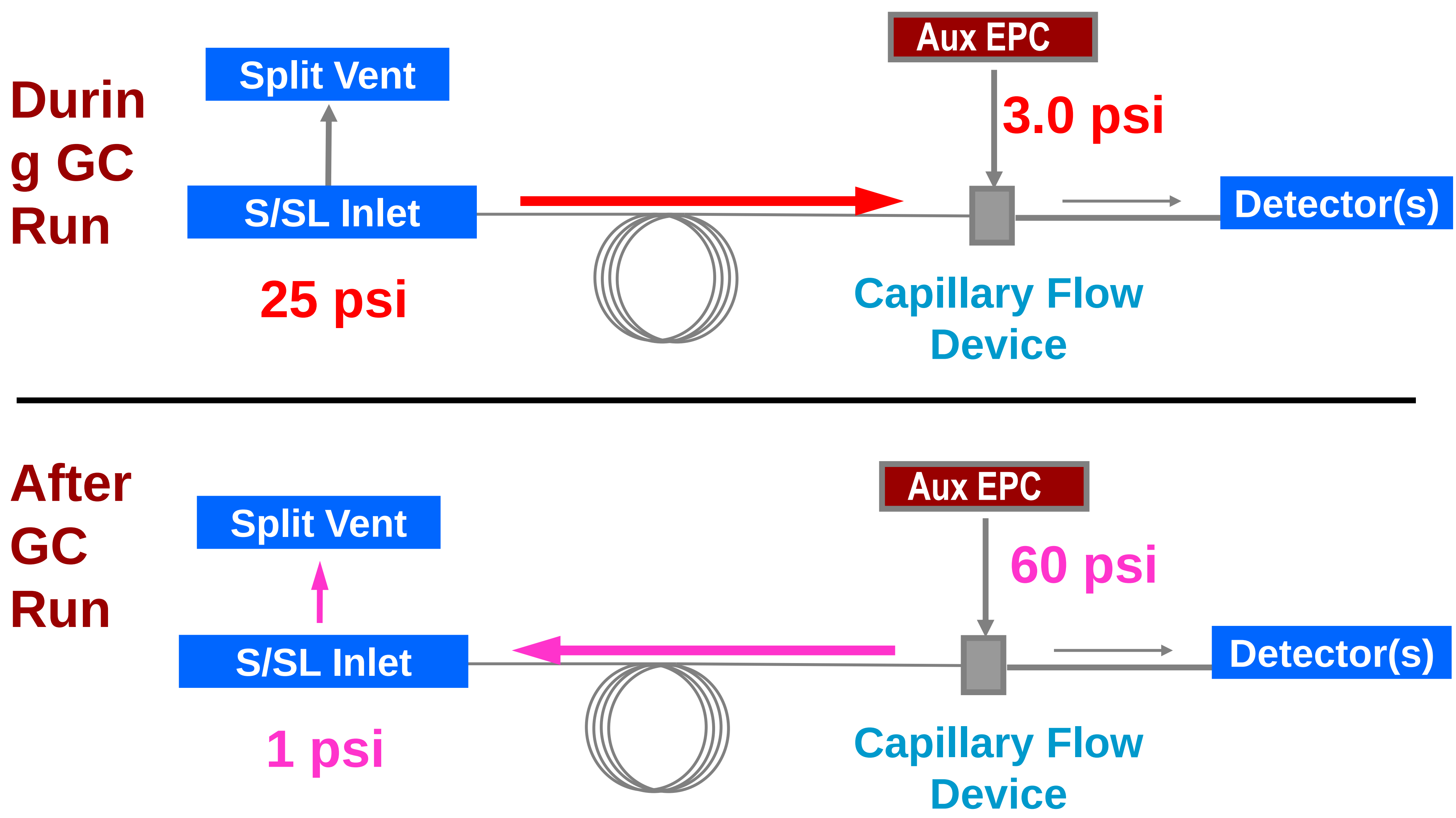
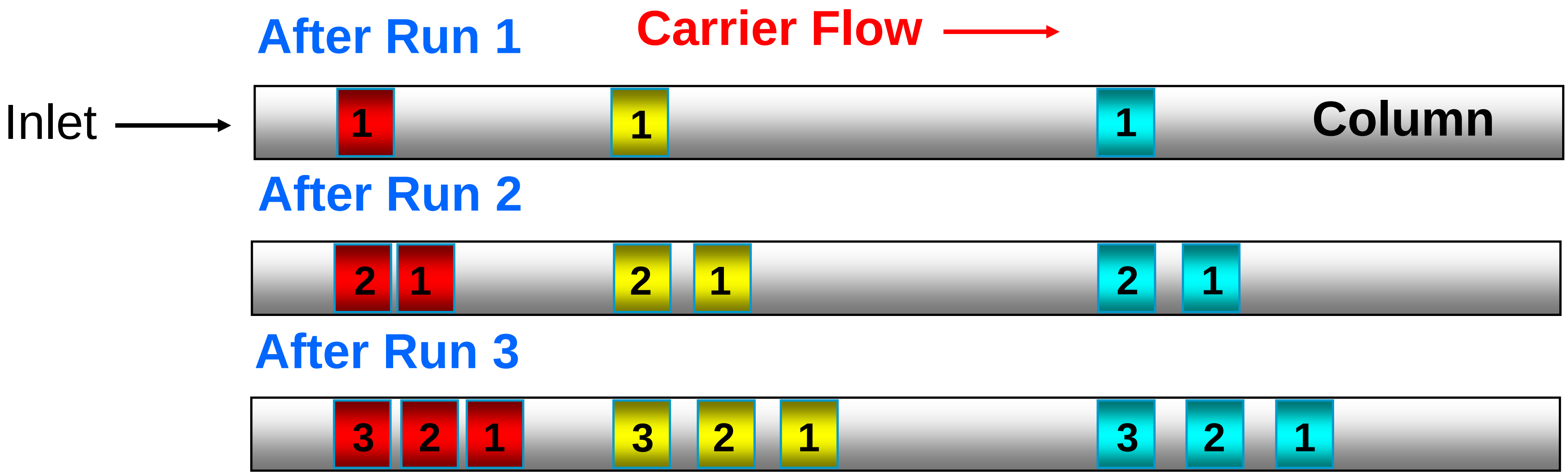
Principle Of Backflushing



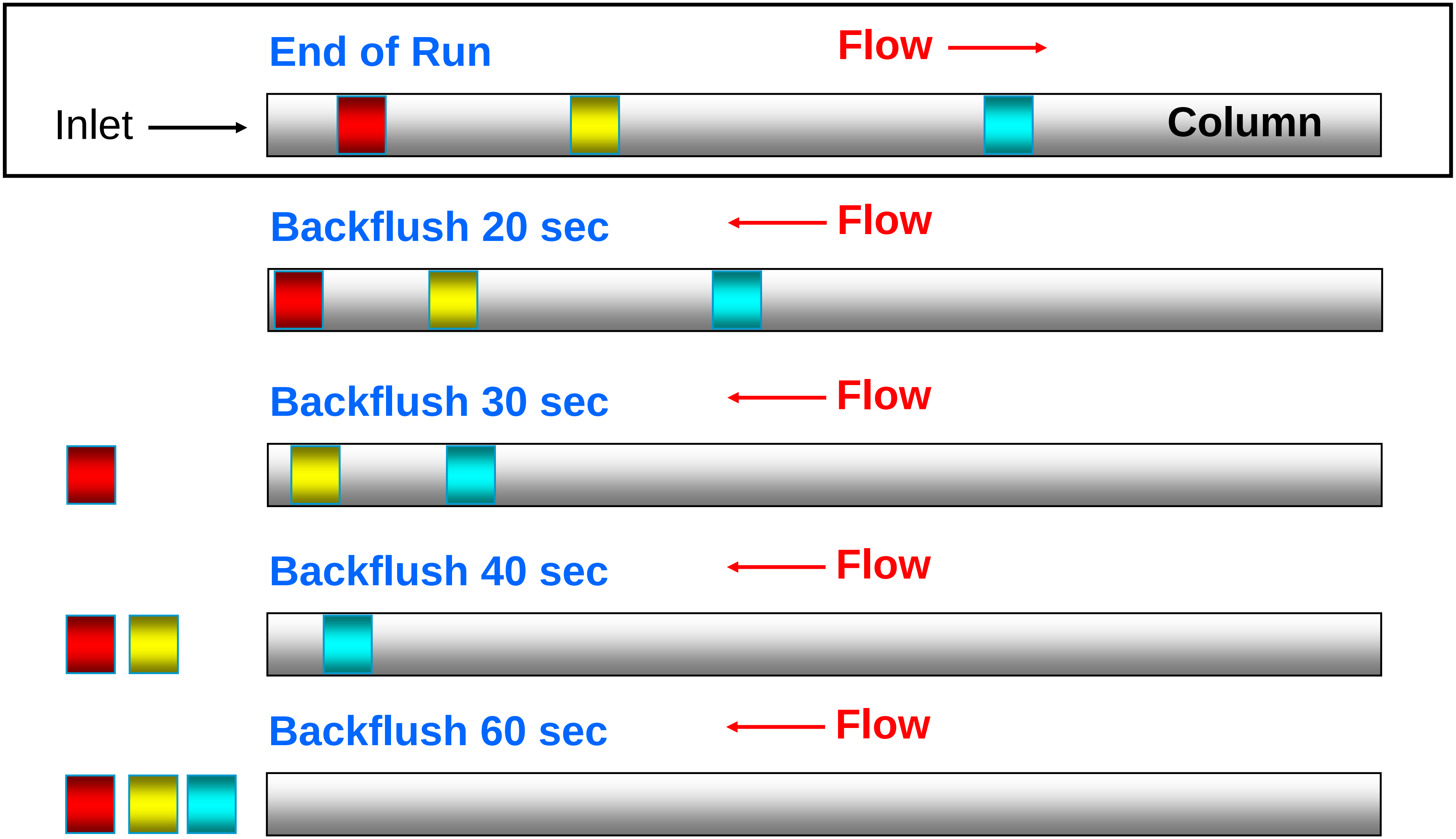
Heavy Compounds May Be Left in Head of Column After Each Injection



These heavy materials build up and travel further into the column with each injection.

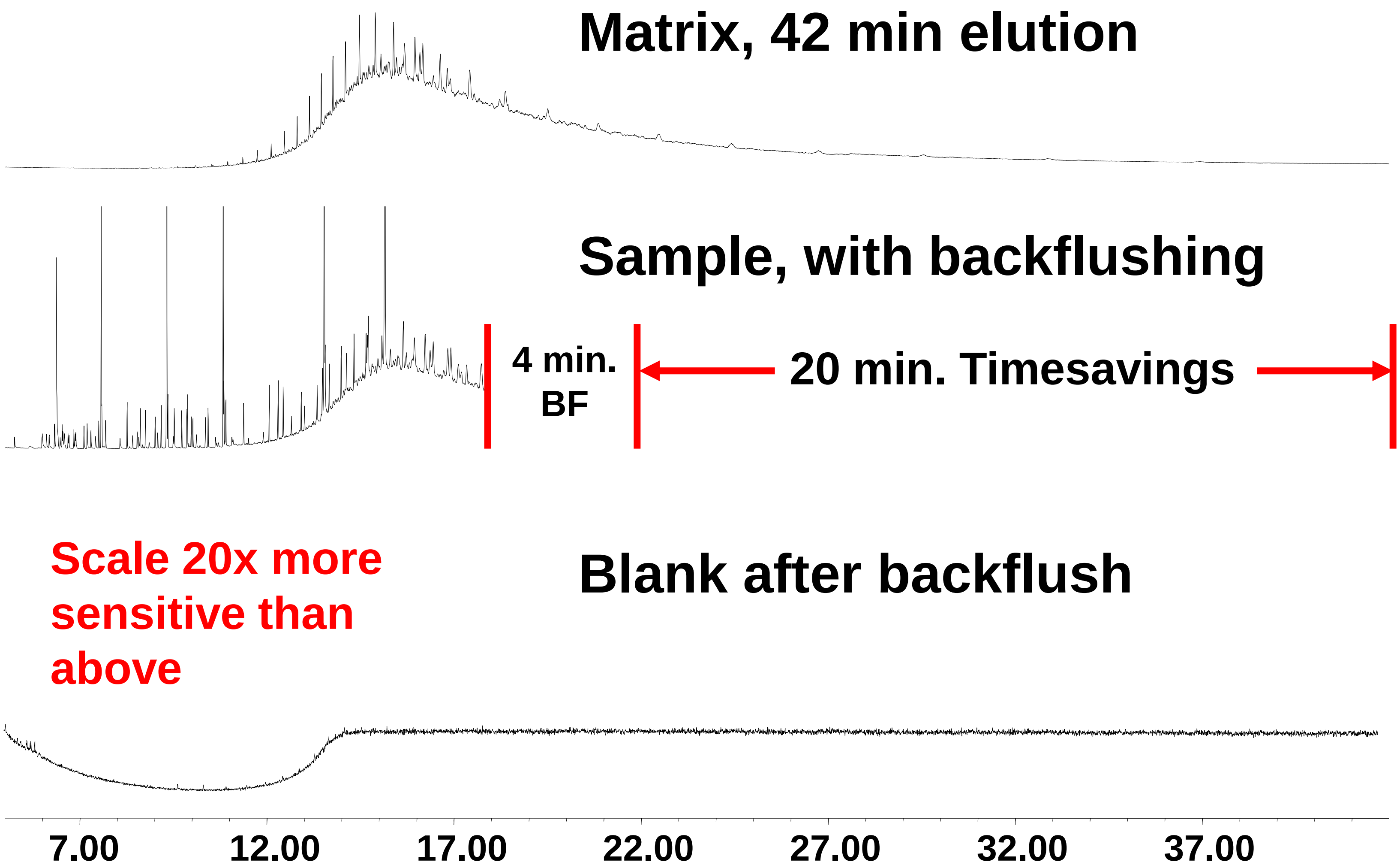
This buildup of heavy materials causes retention time shifts, peak distortion, higher bleed, and loss of sensitivity

Backflushing After Each Injection



Backflushing removes heavy materials after each injection.

Environmental - Gasoil Backflush Example



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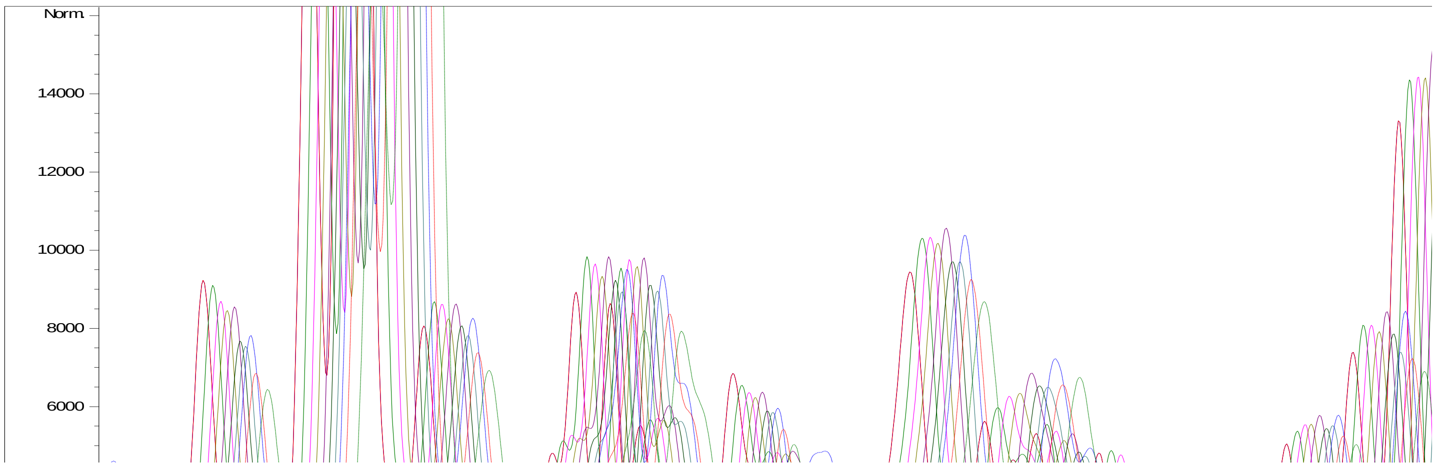
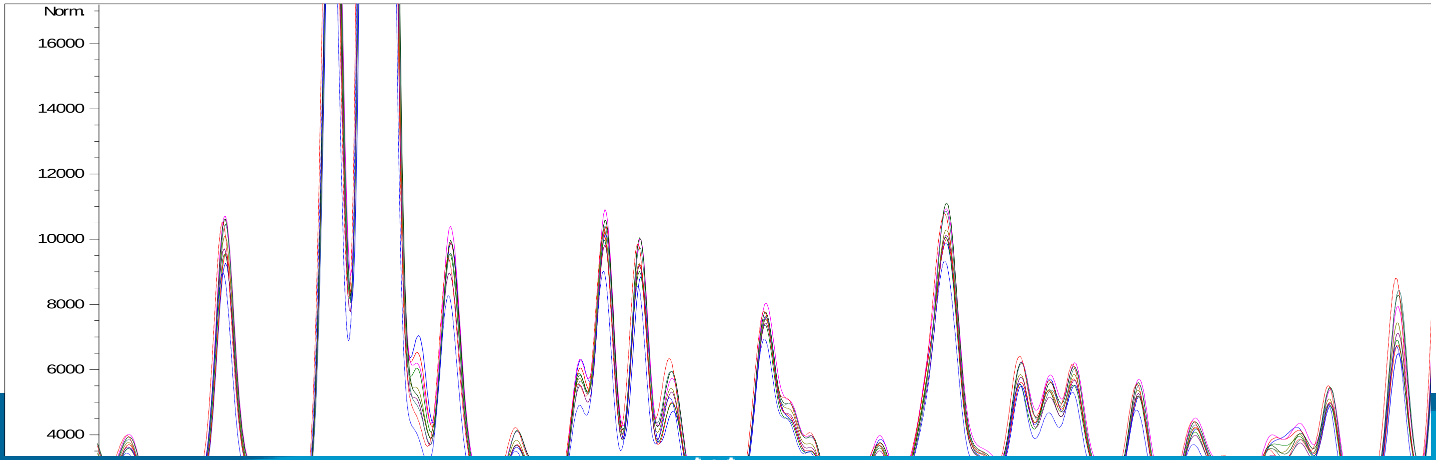
10% Fish Oil In Acetone: Retention Time Shifts Eliminated With Backflushing

10 Runs without Backflushing: Retention times shift ~4-5 sec during 10 runs



10 Runs with Backflushing: RT shift eliminated



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PAH Analysis, NOAA 29: GC/MS with Column Backflush

Oven Program
50 °C for 0.8 min
then 70 °C/min to 180 °C for 0 min; then 7 °C/min to 230 °C for 1 min
then 40 °C/min to 280 °C for 1 min; then 25 °C/min to 335 °C for 3 min

Run Time 18.25 min

MM Inlet

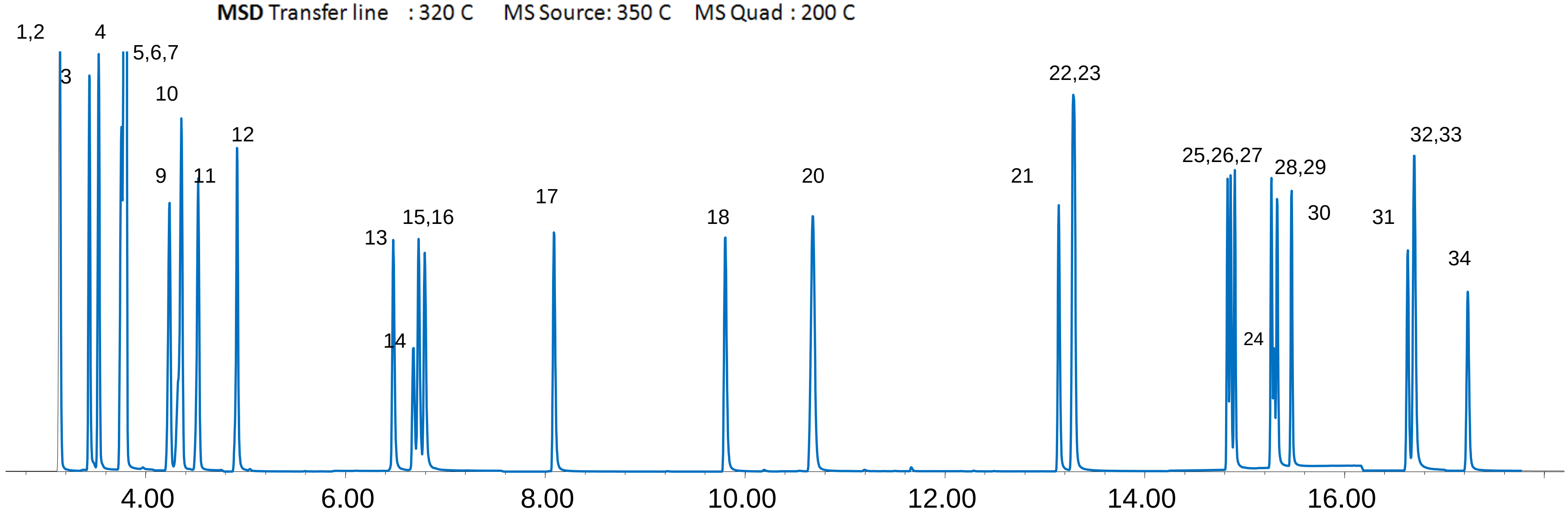
Mode Pulsed Splitless Temperature: 320 °C

Column DB-EUPAH, 20 m x 180 µm x 0.14 µm

Column Flow constant flow at 1 mL/min (pressure = 25.885 psi)

MSD Transfer line : 320 C MS Source: 350 C MS Quad : 200 C

-- Improved reliability and speed



	Internal Std	4	2-Methylnaphthalene	15	Phenanthrene	26	Benzo[k]fluoranthene
1	Naphthalene-d8	5	Biphenyl	16	Anthracene	27	Benzo[j]fluoranthene
9	Acenaphthene-d10	6	2,6-dimethylnaphthalene	17	1-methylphenanthrene	28	Benzo[e]pyrene
14	Phenanthrene-d10	7	HMB	18	Fluoranthene	29	Benzo[a]pyrene
24	Benzo[a]pyrene-d12	8	Acenaphthylene	20	Pyrene	30	Perylene
		10	Acenaphthene	21	Benz[a]anthracene	31	Dibenz[a,c]anthracene
	Target Compounds	11	2,3,5-trimethylnaphtha...	22	Triphenylene	32	Dibenz[a,h]anthracene
2	Naphthalene	12	Fluorene	23	Chrysene	33	Indeno[1,2,3-cd]pyrene
3	1-methylnaphthalene	13	Dibenzothiophene	25	Benzo[b]fluoranthene	34	Benzo[ghi]perylene

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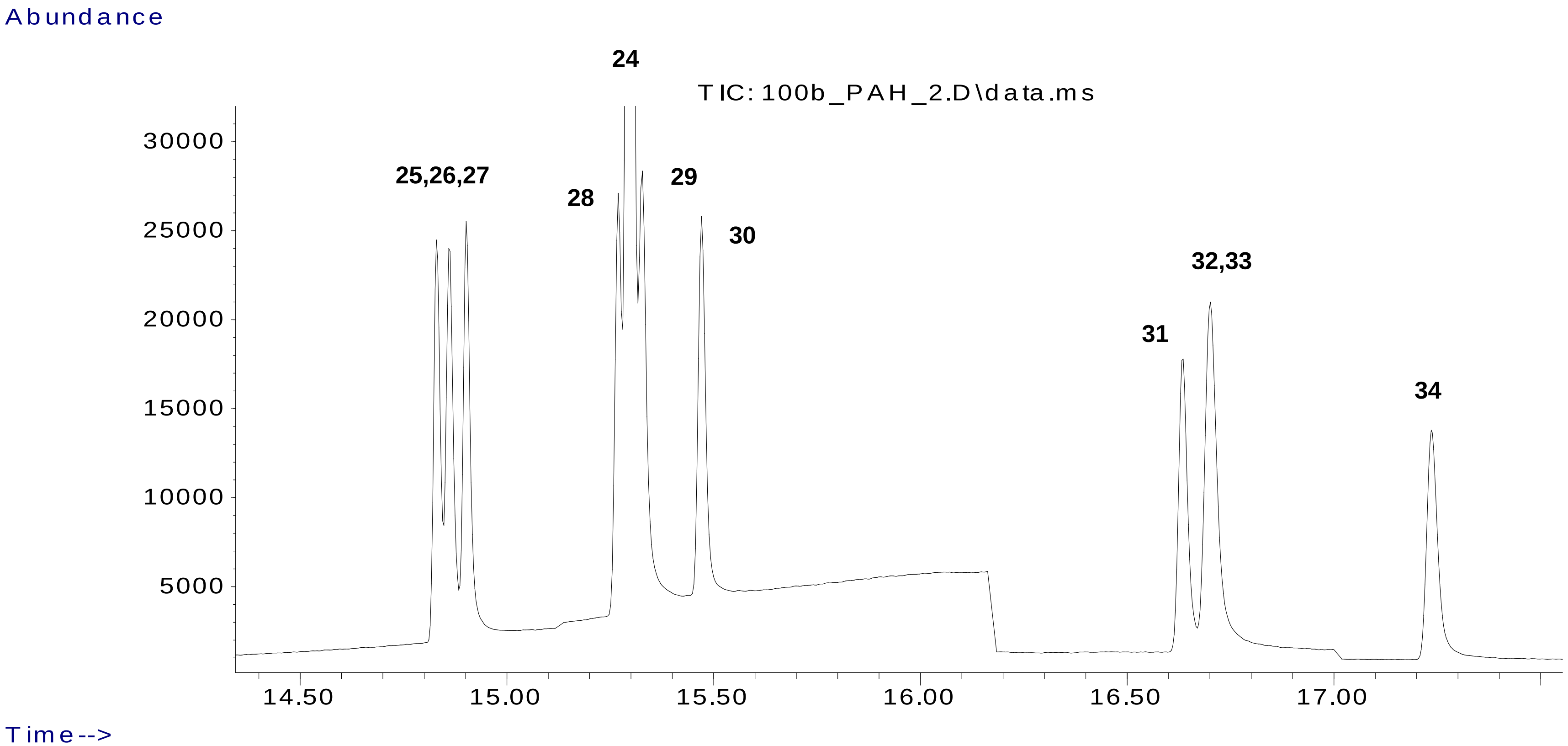
PAH Analysis: GC/MS SIM Late Eluters



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24	Benzo[a]pyrene-d12	8	Acenaphthylene	20	Pyrene	30	Perylene
		10	Acenaphthene	21	Benz[a]anthracene	31	Dibenz[a,c]anthracene
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2	Naphthalene	12	Fluorene	23	Chrysene	33	Indeno[1,2,3-cd]pyrene
3	1-methylnaphthalene	13	Dibenzothiophene	25	Benzo[b]fluoranthene	34	Benzo[ghi]perylene

r^2 values for 7 level cal curves, GC-QQQ and GC-Q

	7 levels --->	1 - 1000	1 - 100	1 - 1000
RT		QQQ A	QQQ V	Q
3.14	Napthalene	0.9998	0.9972	0.9997
3.43	1-methylnaphthalene	0.9998	0.9995	0.9998
3.53	2-methylnaphthalene	0.9999	0.9995	0.9996
3.76	Biphenyl	0.9998	0.9902	0.9998
3.78	2,6-dimethylnaphthalene	0.9998	0.9983	0.9999
4.24	Acenapthylene	0.9999	0.9994	0.9998
4.80	Acenapthene	0.9999	0.9999	0.9997
4.97	2,3,5-trimethylnaphthalene	0.9999	0.9998	0.9998
5.35	Fluorene	0.9999	0.9998	0.9998
6.48	Dibenzothiophene	0.9996	0.9989	0.9998
6.73	Phenanthrene	0.9997	0.9992	0.9999
6.79	Anthracene	0.9997	0.9985	0.9999
8.30	1-methylphenanthrene	0.9997	0.9996	0.9998
9.80	Fluoranthene	0.9960	0.9997	0.9998
10.68	Pyrene	0.9970	0.9998	0.9998
13.14	Benzo(a)anthracene	0.9930	0.9990	0.9998
13.29	Chrysene	0.9940	0.9997	0.9999
14.83	Benzo(b)fluoranthrene	0.9997	0.9980	0.9987
14.86	Benzo(k)fluoranthrene	0.9992	0.9983	0.9985
15.27	Benzo(e)pyrene	0.9999	0.9977	0.9987
15.33	Benzo(a)pyrene	0.9998	0.9971	0.9987
15.47	Perylene	0.9996	0.9977	0.9986
16.70	Indeno(1,2,3,-cd)pyrene	0.9997	0.9899	0.9996
16.69	Dibenz(a,h)anthracene	0.9980	0.9895	0.9996
17.23	Benzo(ghi)perylene	0.9888	0.9889	0.9991

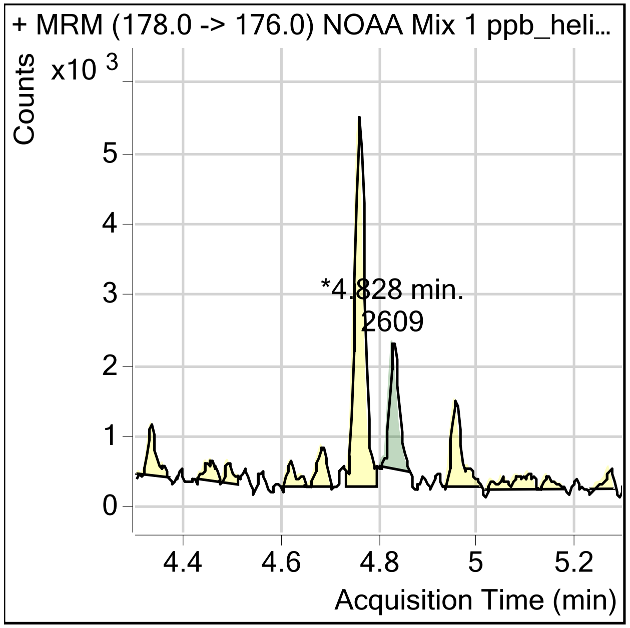
QQQ A and Q calibration stds were in isooctane solvent.

QQQ V calibration stds were in QuEChERS extract of fish at 1g/mL

Data from Ralph Hindle, Vogon Labs, 7000A

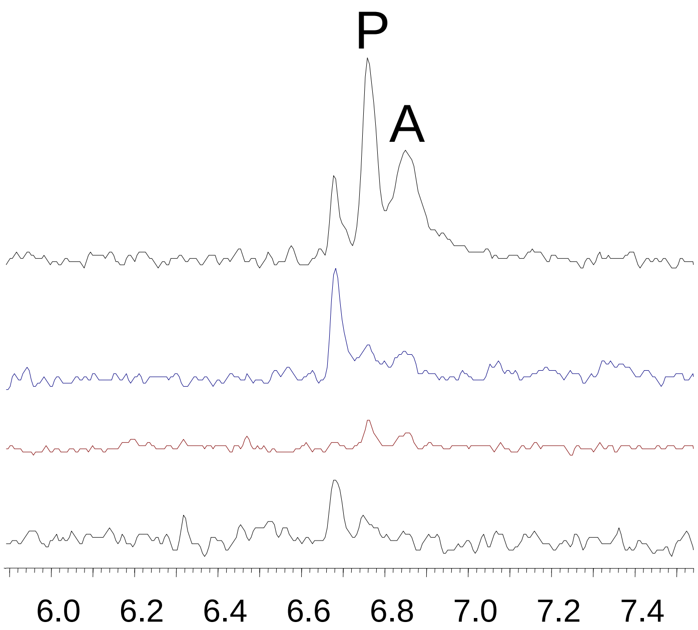
Phenanthrene and Anthracene 1.0 ppb Standard

7000A QQQ in QuEChERS fish extract



Vogon Labs

5975 Q in Isooctane



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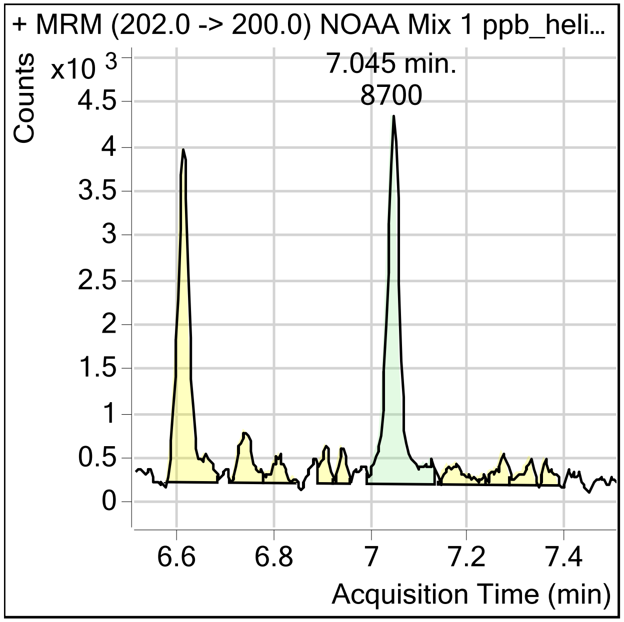
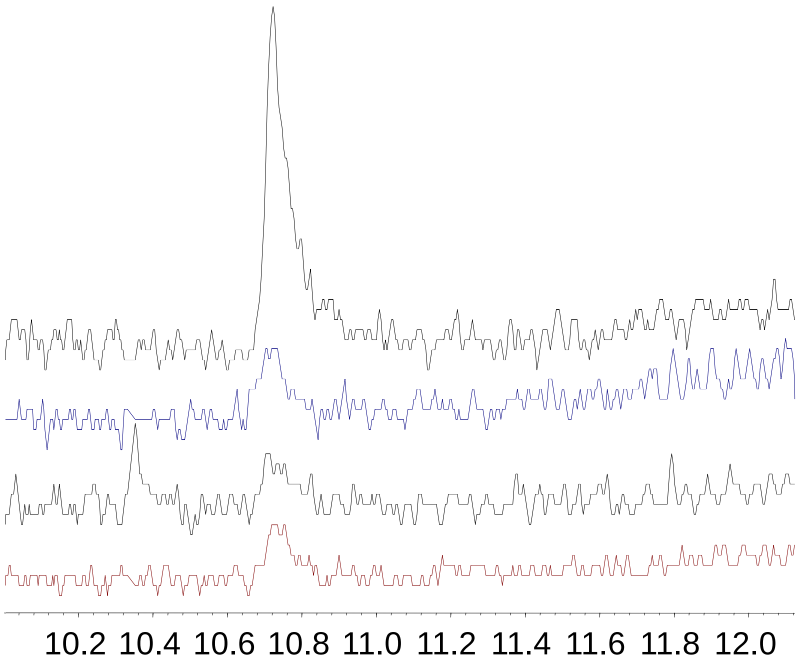
Pyrene 1.0 ppb Standard

7000A QQQ in QuEChERS fish extract



Vogon Labs

5975 Q in Isooctane



Agilent LFS

Recovery Values for PAHs, Spiked into Mussel Tissue at 125 ppb and Extracted Using QuEChERS + Dispersive SPE with no Additional Cleanup nor Concentration

	25 ppb spike 1	25 ppb spike 2	25 ppb spike 3	Avg % Rec
Acenaphthylene	23.8	25.0	25.7	99
Acenaphthene	23.3	24.8	22.5	94
Fluorene	31.3	30.6	29.2	121
Phenanthrene	24.5	27.1	26.4	104
Anthracene	22.5	23.6	24.3	94
Fluoranthene	25.7	25.9	26.8	105
Pyrene	22.9	22.9	24.1	93
Benz[a]anthracene	29.2	27.9	29.9	116
Chrysene	24.0	23.4	24.3	96
Benzo[b]fluoranthene	22.0	23.1	23.6	92
Benzo[k]fluoranthene	20.7	21.9	22.2	86
Benzo[a]pyrene	27.0	29.5	31.7	117
Dibenz[a,h]anthracene	18.8	19.4	19.9	77
Indeno[1,2,3-cd]pyrene	17.3	17.9	18.7	72
Benzo[ghi]perylene	17.3	18.0	18.7	72

Extracts measured by both GC-QQQ MRM and GC-Q SIM. Recovery values were the same.

Concentration in 3 g mussel tissue = 125 ppb

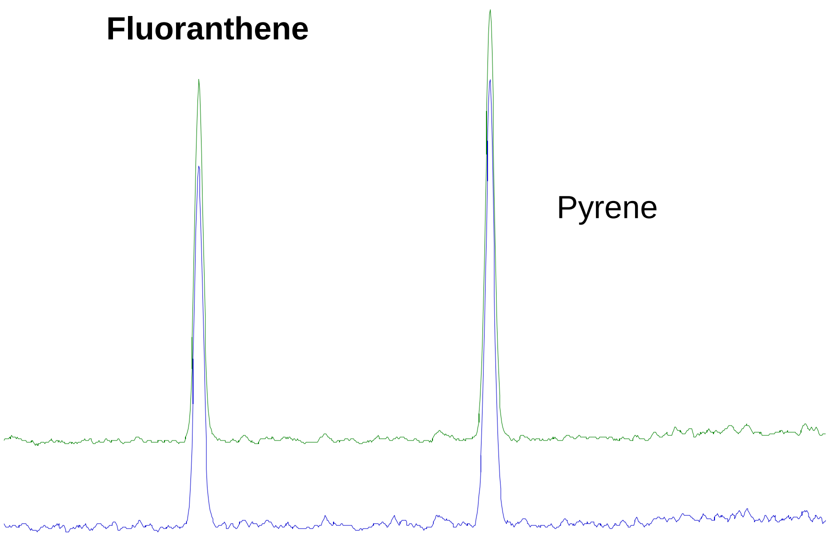
Signal to Noise (pk-pk) for NOAA PAHs (5/29/2010 list) GC-QQQ and GC-Q 1 ppb Standard and 125 ppb Spike in mussels

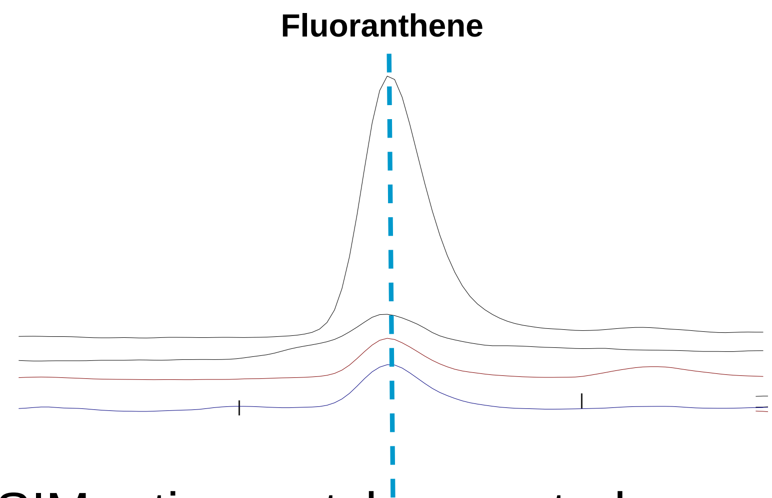
	7000B	5975C	7000B	5975C
	MRM	SIM	MRM	SIM
	Std	Std	Spike	Spike
	1 ppb	1ppb	25 ppb	25 ppb
Naphthalene	36	23	---	---
Fluorene	8.0	7.2	112	92
Phenanthrene	6.7	8.8	121	69
Anthracene	6.8	5.7	100	60
Fluoranthene	8.0	5.3	88	43
Pyrene	6.3	4.6	105	39
Benz[a]anthracene	22	5.0	130	128
Chrysene	21	5.1	130	121
Benzo[a]pyrene	15	10	60	11

Sensitivity for standards is similar in the 2 systems but better in the QQQ when matrix is present. Spiked mussel tissue extracted with QuEChERS + dispersive SPE.

What if my QuEChERS extract does not have enough sensitivity ? Fluoranthene at ~ 15 pg is Okay.

Both sets of EICs are from a QuEChERS extract concentrated 10x in ACN. Background is still low.





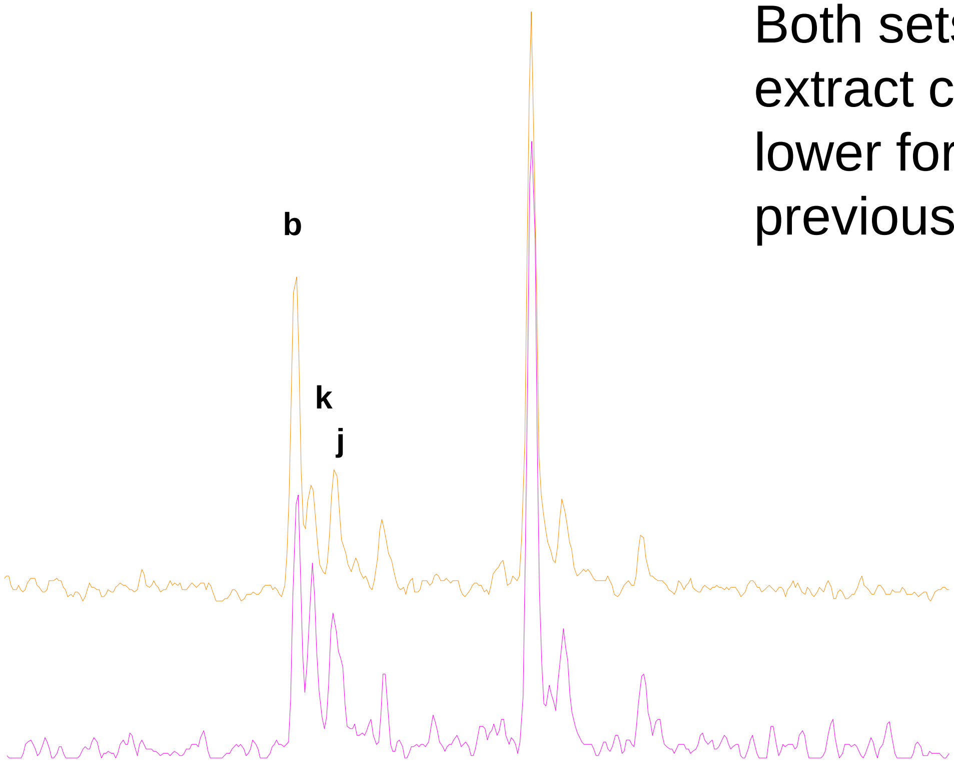
MRM ratios match expected on QQQ

SIM ratios match expected on GC-Q. RTs align.

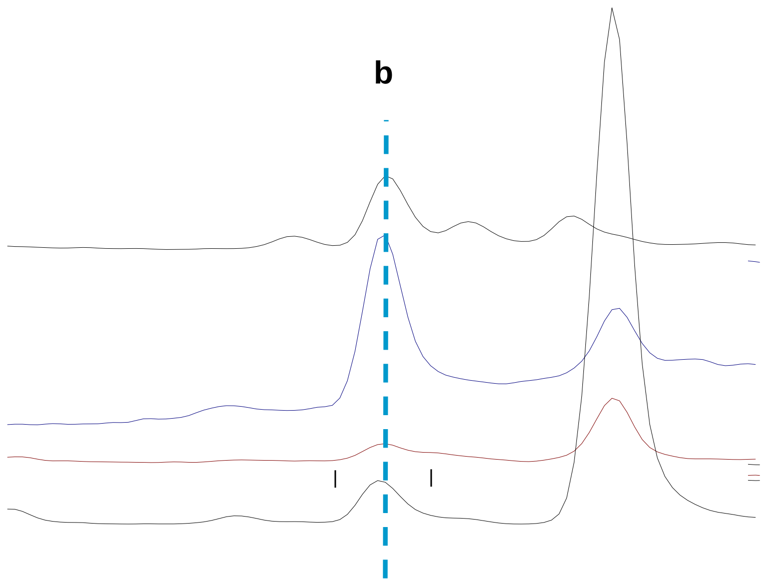
These also shows how a 10 uL solvent vent injection, of a non-concentrated extract, might appear using an MMI.

What if my QuEChERS extract does not have enough sensitivity ? Benzo[b,k,j]fluoranthenes at ~1-6 pg.

Both sets of EICs are from a QuEChERS extract concentrated 10x in ACN. S/N is lower for these ions compared to previous slide.



MRM ratios match expected on QQQ



SIM ratios do not match expected on GC-Q. RTs do not align

These also shows how a 10 uL solvent vent injection, of a non-concentrated extract, might appear using an MMI.

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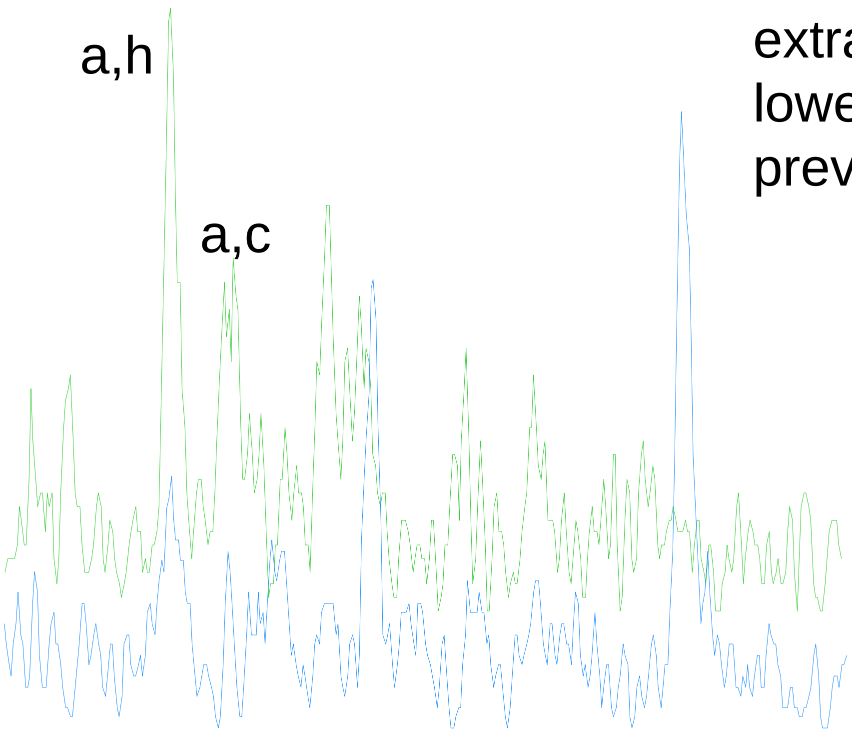
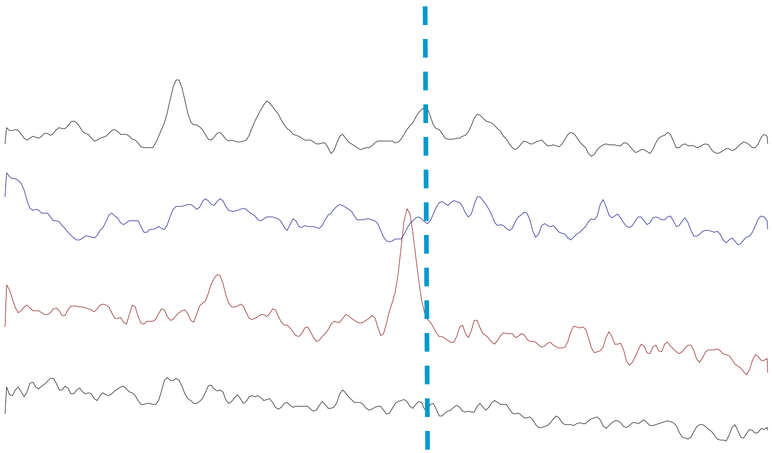
What if my QuEChERS extract does not have enough sensitivity ? Dibenz(a,h) & (a,c) anthracene at ~ 0.2 pg



Both sets of EICs are from a QuEChERS extract concentrated 10x in ACN. S/N is lower for these ions compared to previous slide.



MRM ratios do not match expected on QQQ, but s/n is better than Q

SIM data useful if you squint.

These also shows how a 10 uL solvent vent injection, of a non-concentrated extract, might appear using an MMI.

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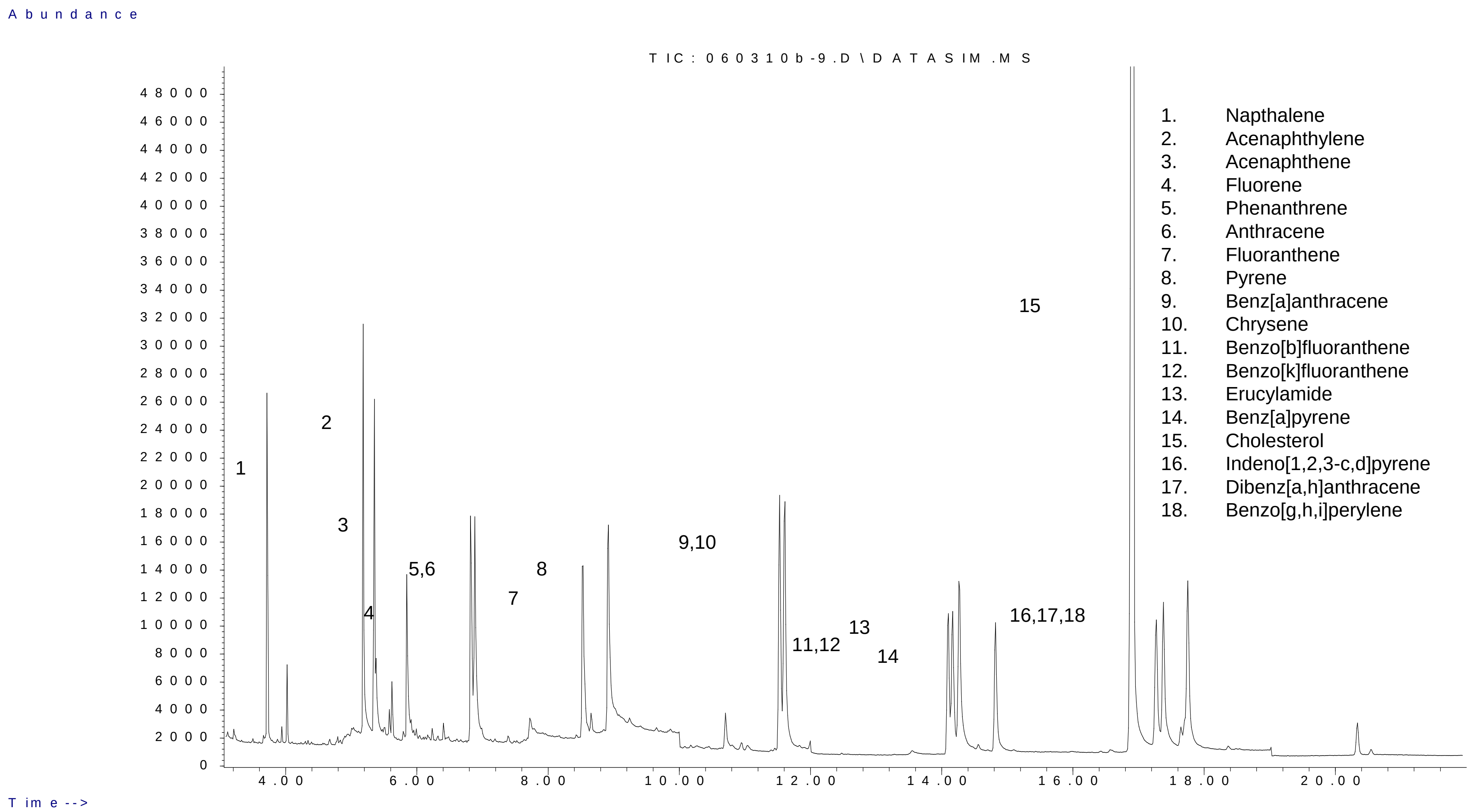
125 ppb EPA PAHs extracted from Swai fish using QuEChERS DB-5ms 20m 0.18mm 0.18µm GC/MS SIM TIC



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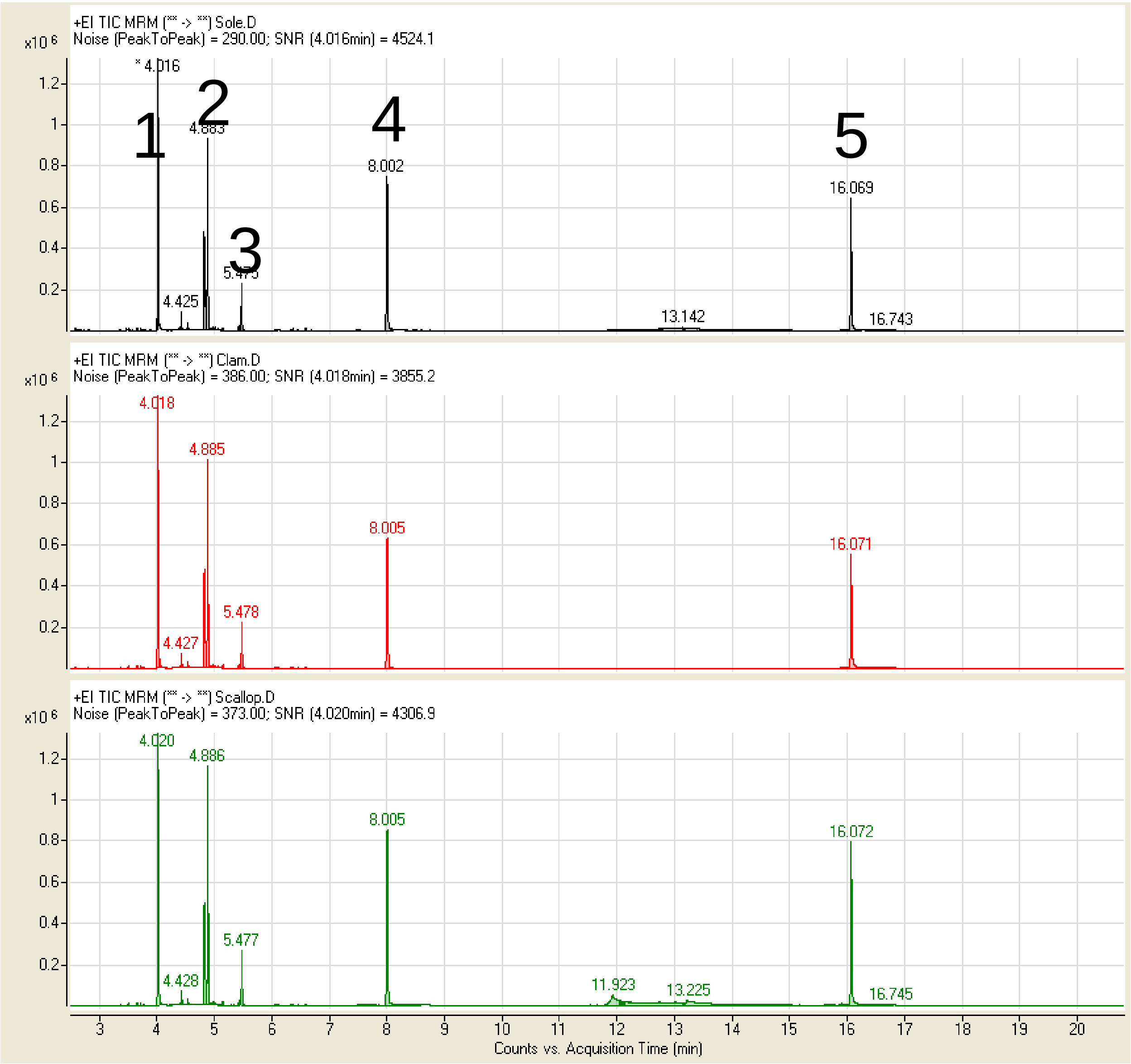
Sole, Clam & Scallop Samples – Spiked with ISTDs at 67 ppb and Extracted using Agilent QuEChERS



<u>Internal Standards</u>

1. Naphthalene-d8
2. Hexamethylbenzene
3. Acehaphthene-d10
4. Phenanthrene-d10
5. Benzo[a]pyrene-d12

Data from Arkansas DOH on 7000B QQQ-A. Jeffrey Moran and John Blevins

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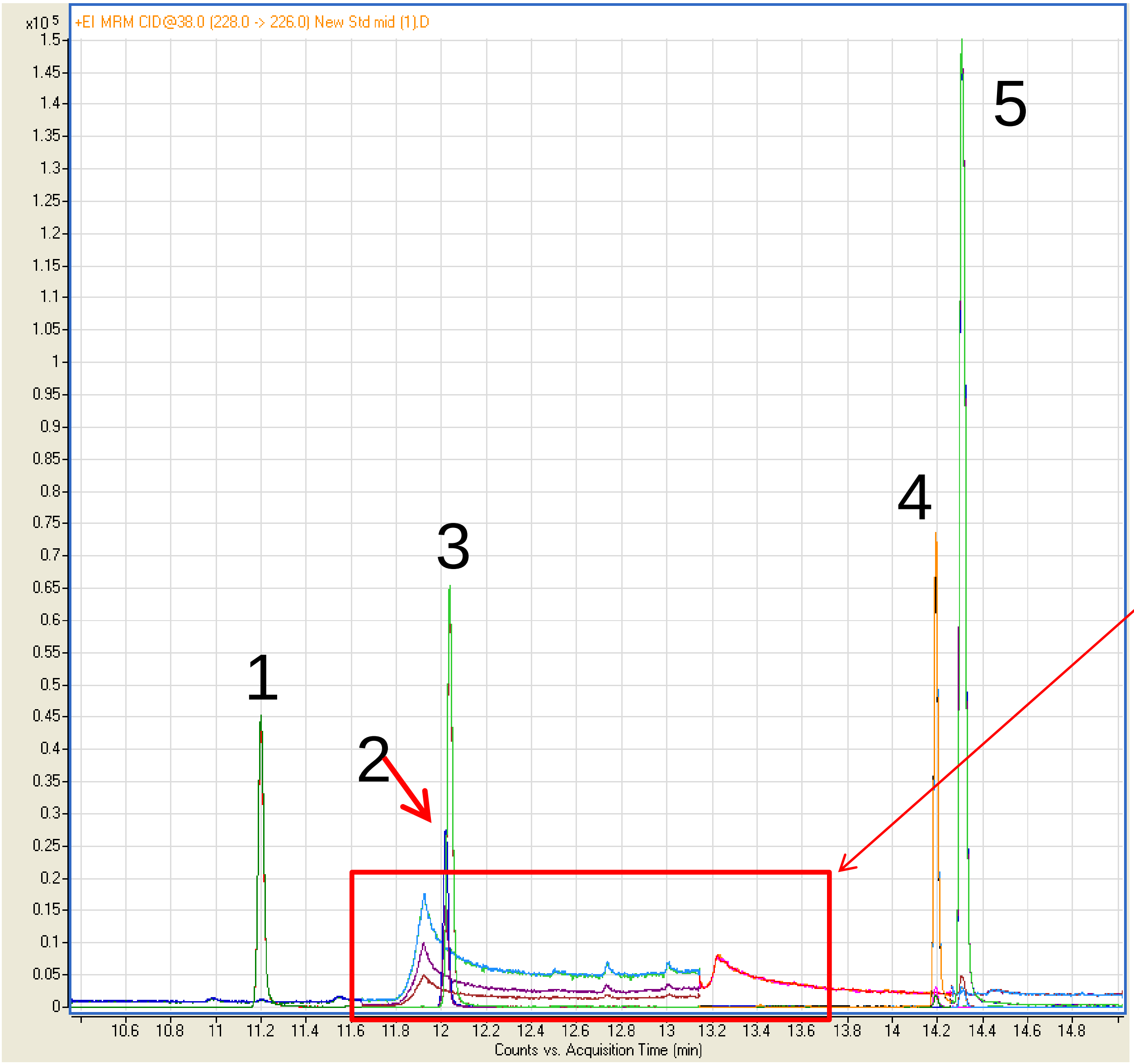
Background in Scallop Extract vs. Blank Spiked at 67 ppb Before Extraction



PAHs

1. Fluoranthene
2. Retene
3. Pyrene
4. Benz[a]anthracene
5. Chrysene + Triphenylene

Low level background

Data from Arkansas DOH on 7000B QQQ-A. Jeffrey Moran and John Blevins

Summary

- QuEChERS: offers a simple sample preparation approach to the extraction and analysis of PAHs in finfish and shellfish
- The simplicity and quickness associated with QuEChERS sample preparation allows multitudes of samples to be processes per day versus weeks
- A preconfigured analyzer can help your lab start running PAHs with higher productivity
- Backflushing will reduce cycle time and instrument maintenance for samples with matrix
- Signal-to-noise is about the same on a 5975C-Q using SIM compared to a 7000B-QQQ using MRM for clean samples
- The 7000B-QQQ analyzer can reach lower detection limits for PAHs, with greater confidence, than the 5975C-Q for QuEChERS extracts of seafood