

A Sustainable Approach to SVOC Analysis: Modernizing EPA 8270

Session: Innovative Solutions for Reducing Methylene Chloride

Monday, August 4, 2024

Julian Martinez & Sarah Choyke Ph.D.

Eurofins Environment Testing



Environment Testing

Emerging Tech Leads to Operational Tech



**Technology
Exploration**

**Demonstration
Phase**

**Routine
Analysis**



Categorical Innovations



Innovate or Get Left Behind

New Technology:

Better Sensitivity

More Selectivity

More Efficient

Cost Savings:

Smaller sample volume

Less Solvent use
(Methylene Chloride)

Easier shipping and storage

Cons:

Change is scary

NPDES Permits
mandated
methods/technology

Regulations slow to adapt

Reduce Dichloromethane use

- DCM (MeCl_2) is a hazardous solvent with strict handling and disposal requirements.
- Regulatory pressure from the 2024 EPA DCM Rule demands reduction.
- EPA **3510** (LLE), **3546** (Microwave), and **3550** (Ultrasonication) use DCM for EPA Methods 8081, 8082, 8141 & 8270
- Laboratories face increasing demand for sustainable, safer alternatives.



EPA METHYLENE CHLORIDE RULE

- Most commercial and industrial uses prohibited
- Workplace Chemical Protection Program required for continued critical uses
- All consumer uses banned

A Safer, Compliant Water SVOC Workflow

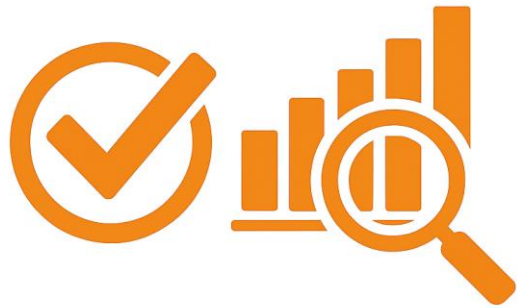


GC-MS/MS
GC-Triple Quad
GC- QQQ

- GC-MS/MS meets QA/QC and MDL/LOQ requirements.
- Align EPA's DCM Rule and ESG/sustainability goals.
- More sensitive & selective
- Developed in partnership with Shimadzu Scientific Instruments for GC-MS/MS method implementation.

EPA Method 3511 > Performance

PERFORMANCE VERIFICATION



- MDLs and LOQs comparable (or better)
- Less matrix
 - More passing QC
 - More stable internal standard recovery
 - Lower RPD/RSD
 - Less instrument maintenance

© 2005 Blackwell Publishing Ltd *Journal of Internal Medicine* 258: 103–110

[illegible]

Flavors of Gas-Chromatography – Mass Spectrometry

Criteria	Full Scan	SIM	MRM
Description	Full mass range	Fragments for target analytes	Full mass range & Fragments
Mass Range	Wide, Comprehensive	Specific ions	Wide and specific
Sensitivity	Lower	Very high	High
Data output	Complete mass spectrum	Selected ions	Selected ions for the full list
Quantitation	Less robust	More robust	Very robust
Library Search	Yes, including TIC	No	Yes, can include TICs

GC-QQQ is the best of both worlds: Selectivity of SIM & Breadth of Full Scan

Multiple Reaction Monitoring (MRM)

Gold Standard for selectivity and sensitivity

High Throughput

Fast scanning, 100s compounds in single analysis

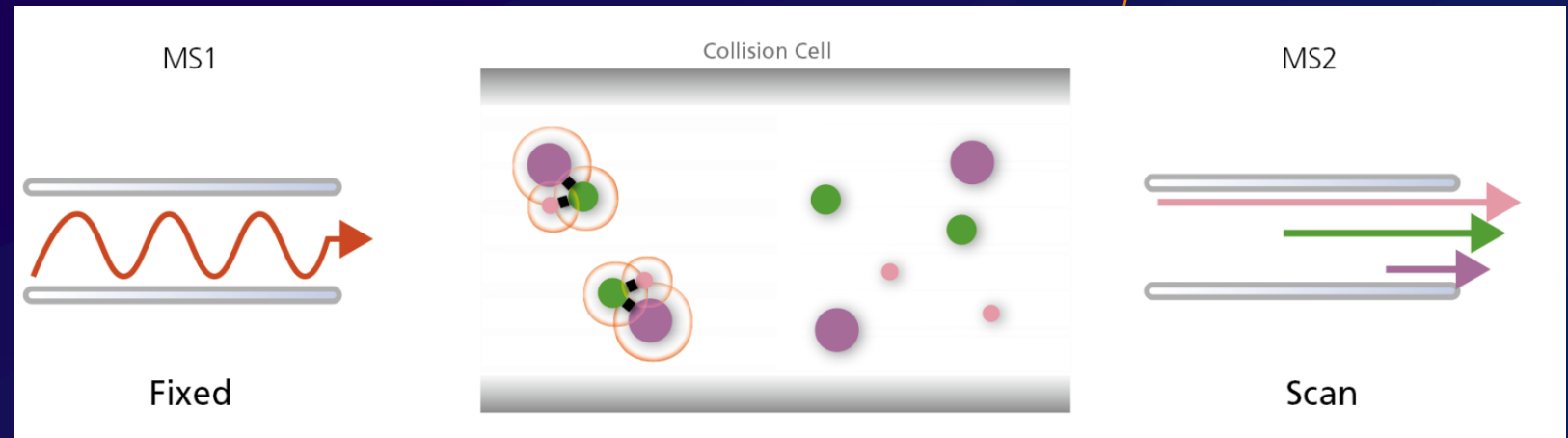
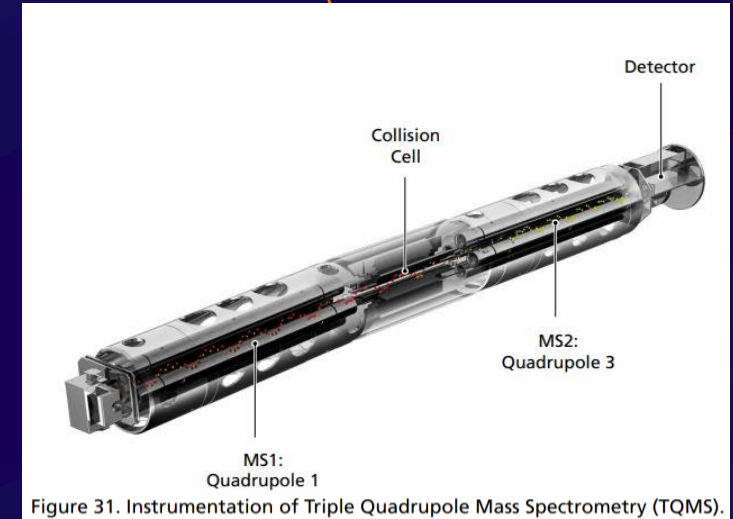
SemiVolatiles

Organophosphorus Pesticides

Organochlorine Pesticides

PCBs

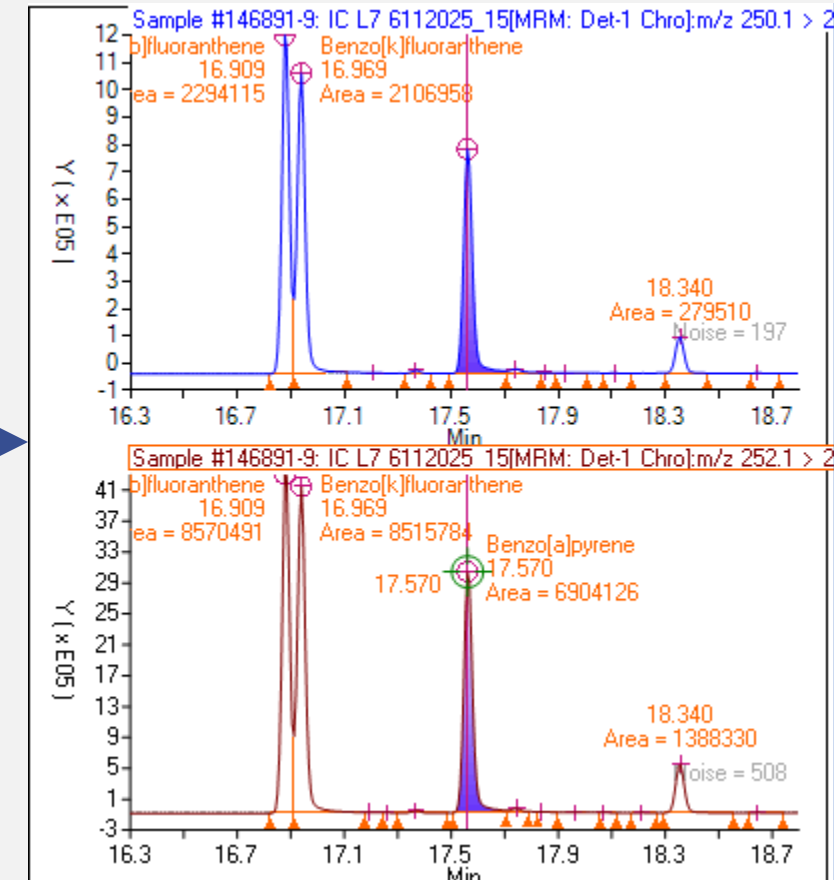
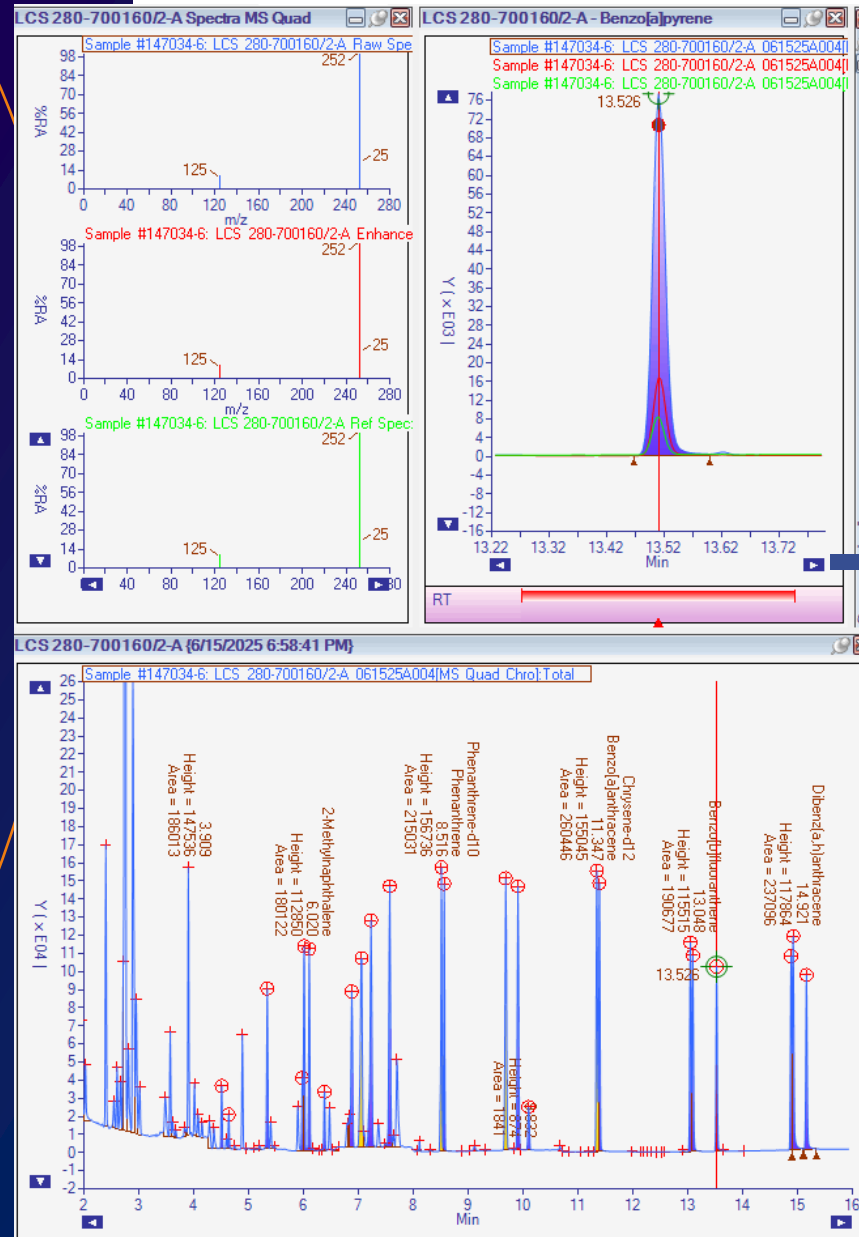
Low Detection Limits



Paradigm Shift: Reading the Tea Leaves: Benzo[a]pyrene



- Peak Identification
- Performance Checks
- Calibration Curve
- MDL/LOD/LOQ
- %R of LCS, MS/D



Instrument Performance Check

- Peak Identification
- Performance Checks
- Calibration Curve
- MDL/LOD/LOQ
- %R of LCS, MS/D

Breakdown Curve: 4,4'- DDT

SW-846 Method

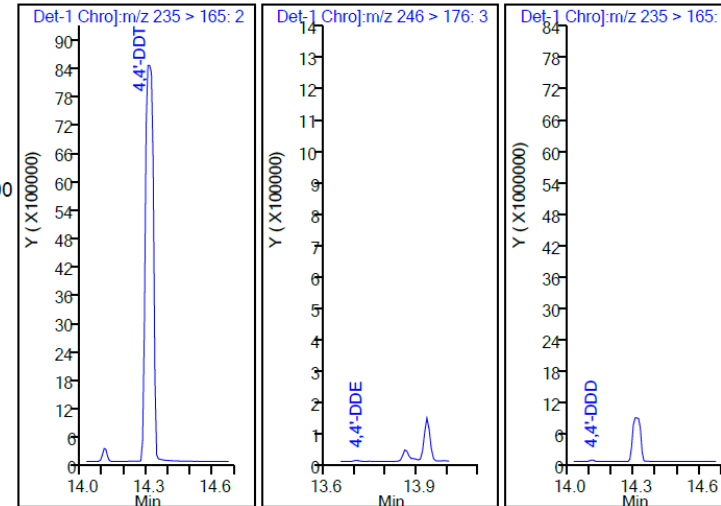
%Breakdown =
$$\left(\frac{\text{Area Breakdown Cpnds}}{\text{Total Area Breakdown Cpnds}} \right) * 100$$

145 4,4'-DDT, Area = 21496544

139 4,4'-DDE, Area = 5519

142 4,4'-DDD, Area = 408520

%Breakdown: 1.89%, <= 20.00%
Passed



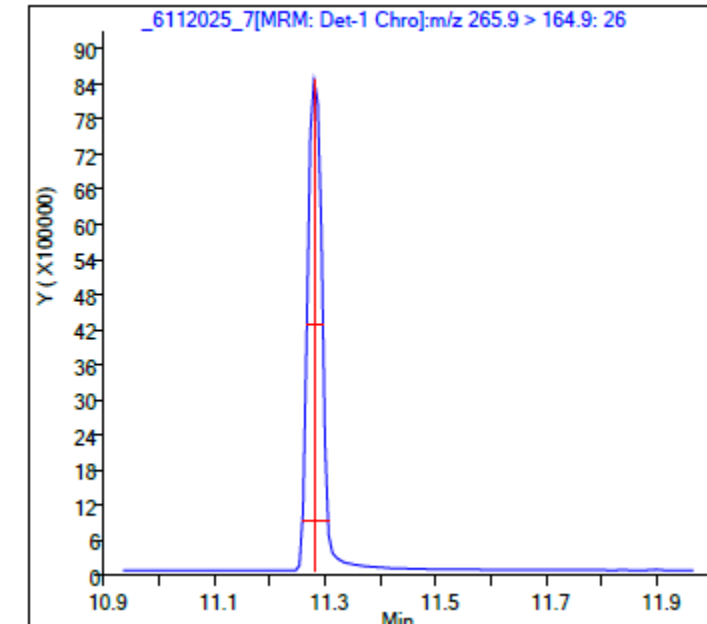
Peak Tailing Factor =
BackWidth/FrontWidth @ 10% Peak Height

Back Width = 0.026 (min.)

Front Width = 0.021 (min.)

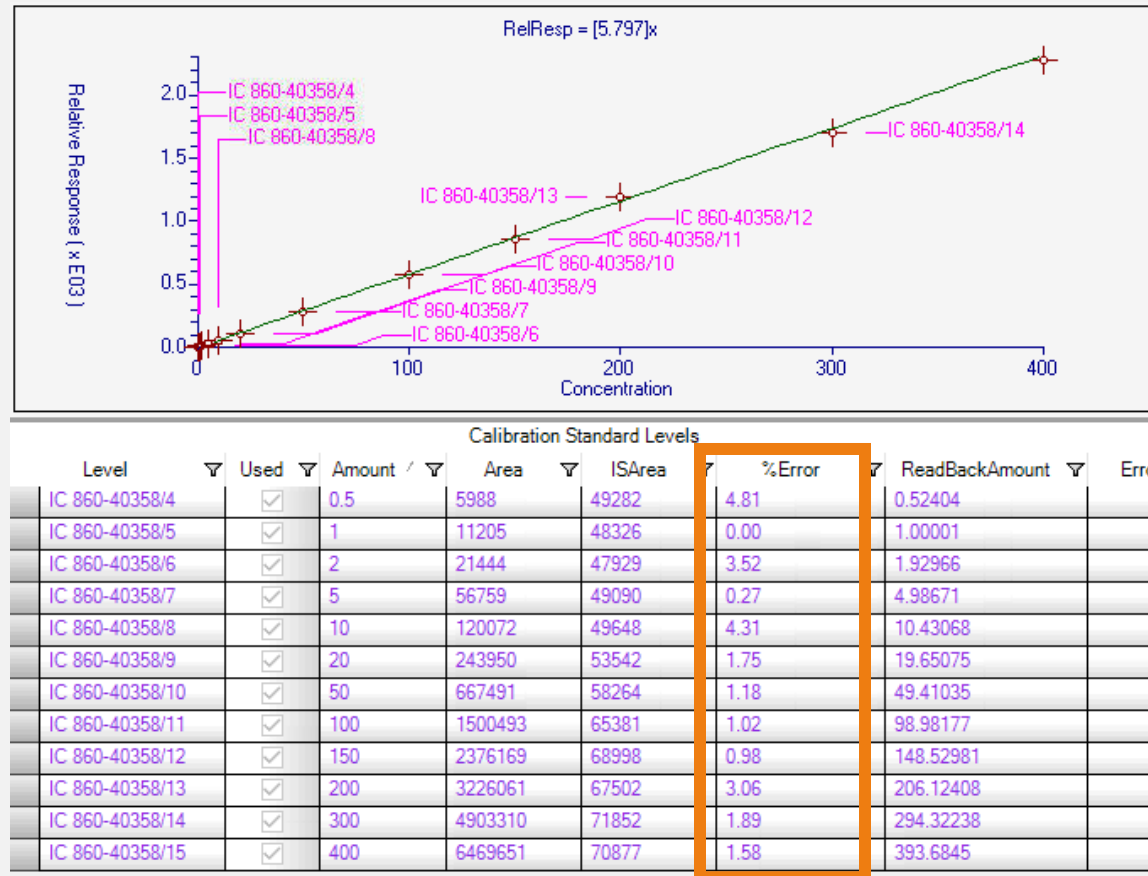
Tailing Factor = 1.24, Max. Tailing <= 2.00
Passed

Tailing Factor: Pentachlorophenol



Reading the Tea Leaves: Benzo[a]pyrene Calibration Curve

- Peak Identification
- Performance Check
- **Calibration Curve**
- MDL/LOD/LOQ
- %R of LCS, MS/D



Locked

Analyte: **Benzo[a]pyrene**

Expected RT: 18.944

Analyte Type: target

Curve Select: AverageLS ☐

Apply Curve to All LG ☐

Mode: ISTD

ISTD Ref: Perylene-d12

Curve Fit: Average

Origin: Force

Weighting: 1/Conc²

Dependent on: ☒ Resp. ☐ Conc.

Primary: %RSD 2.6 ≤ 20.0

*Tune Check is not necessary for instrument stability assessment

- Peak Identification
- Performance Check
- Calibration Curve
- **MDL/LOD/LOQ**
- %R of LCS, MS/D

Reading the Tea Leaves: Benzo[a]pyrene

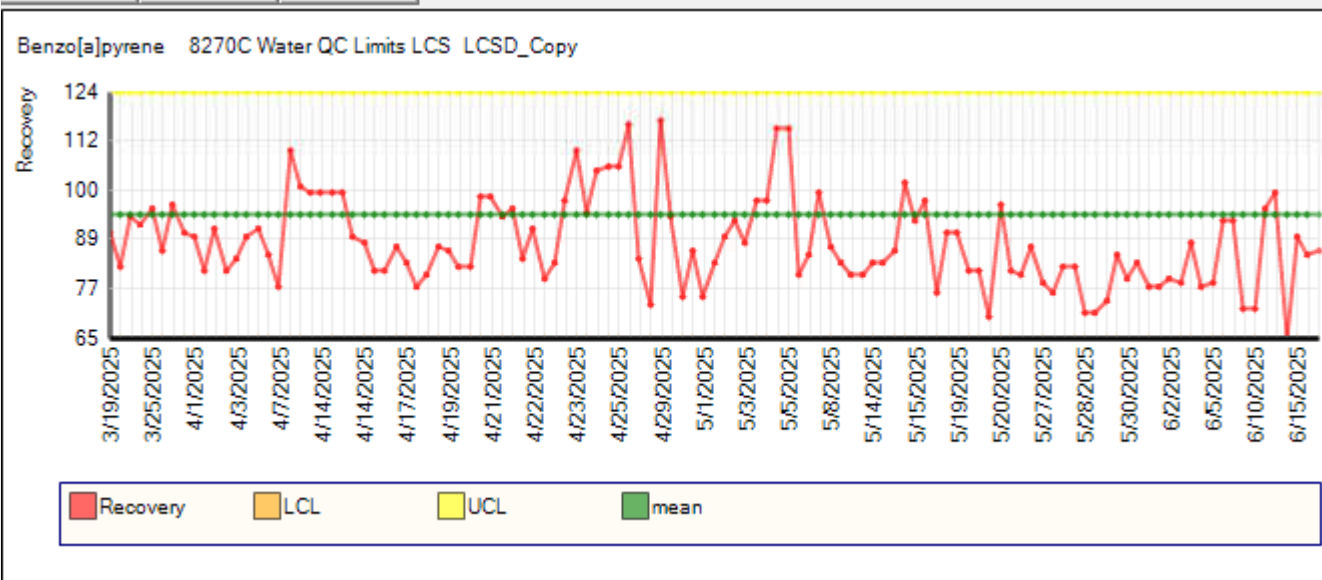
Limit of Quantitation

Compound Class	GC-SIM LOQ	GC-QQQ LOQ	3510/GC-MS/SIM Limitations	3511/GC-QQQ Advantages
PAHs	2-5 µg/L (Full Scan) 0.1-0.2 µg/L (SIM)	0.2-0.5 µg/L	Matrix Interference Evaporative losses	Lower LOQs Mass separation
Phthalate esters	5-10 µg/L (Full Scan)	1 µg/L	Background contamination	Reduced background
Phenols (Acidic SVOC)	10-50 µg/L	0.5-5 µg/L	Poor chromatography, Thermal/pH degradation	Specific transitions No concentration
Nitrosamines	10 µg/L	0.5-1 µg/L	Background contamination Evaporative losses	Lower LOQs Optimized detection

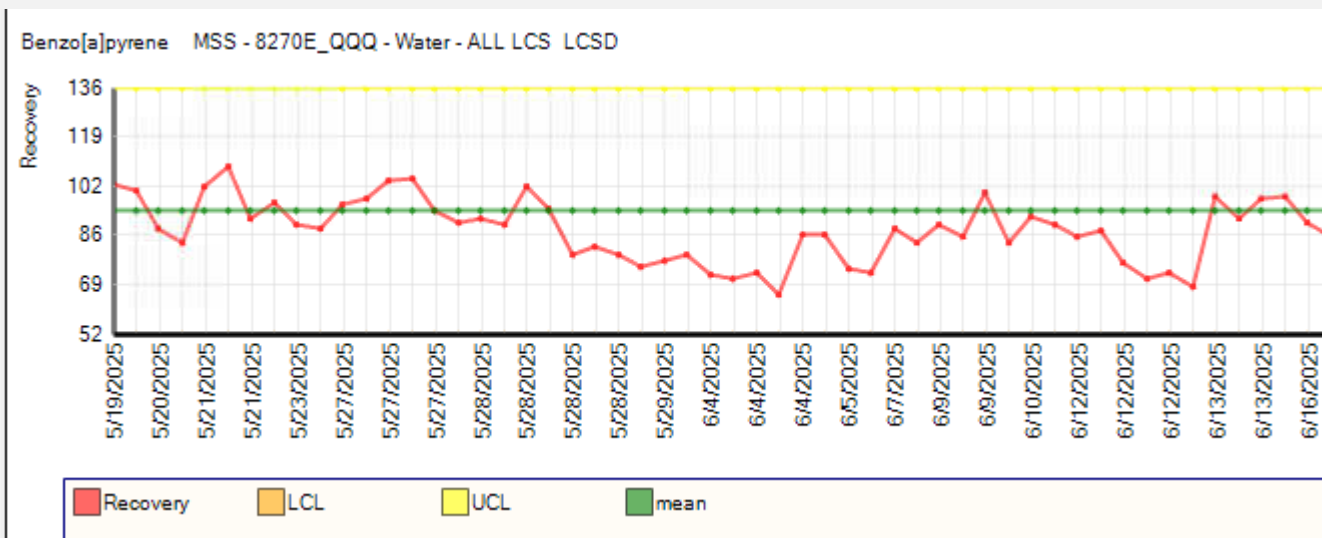
- Peak Identification
- Performance Check
- Calibration Curve
- MDL/LOD/LOQ
- %R of LCS, MS/D

Reading the Tea Leaves: Benzo[a]pyrene

Control Limits: 54-128%



LCS/LCSD
%R: 89%
%RSD: 8.0%

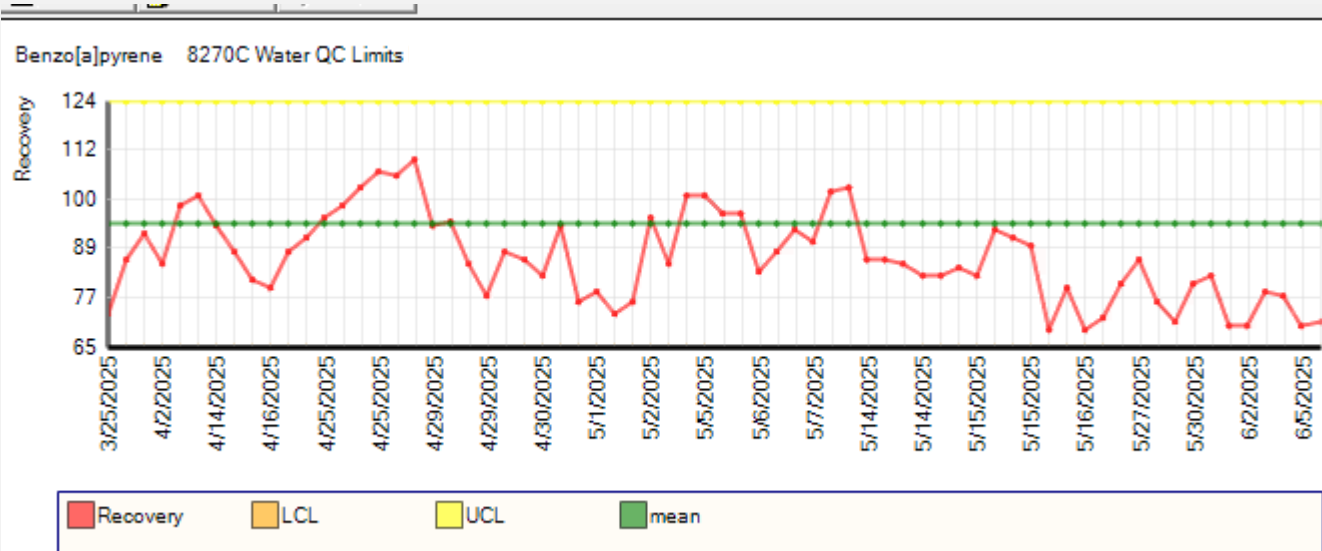


LCS/LCSD
%R: 87%
%RSD: 3.8%

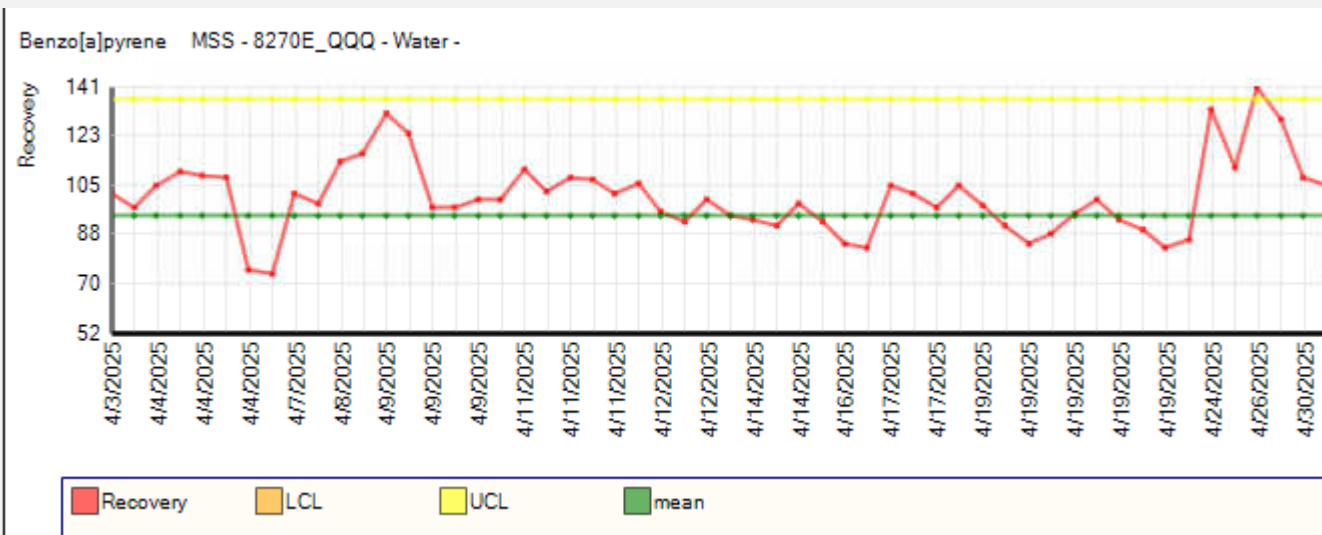
- Peak Identification
- Performance Check
- Calibration Curve
- MDL/LOD/LOQ
- %R of LCS, MS/D

Reading the Tea Leaves: Benzo[a]pyrene

Control Limits: 54-128%



MS/MSD
%R: 86%
%RSD: 4.0%

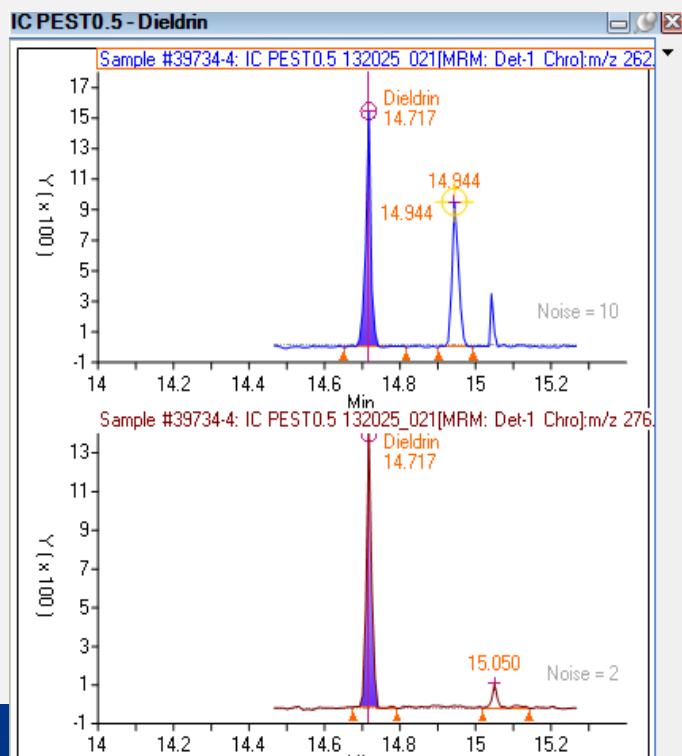


MS/MSD
%R: 101%
%RSD: 3.3%

But wait! There's MORE!

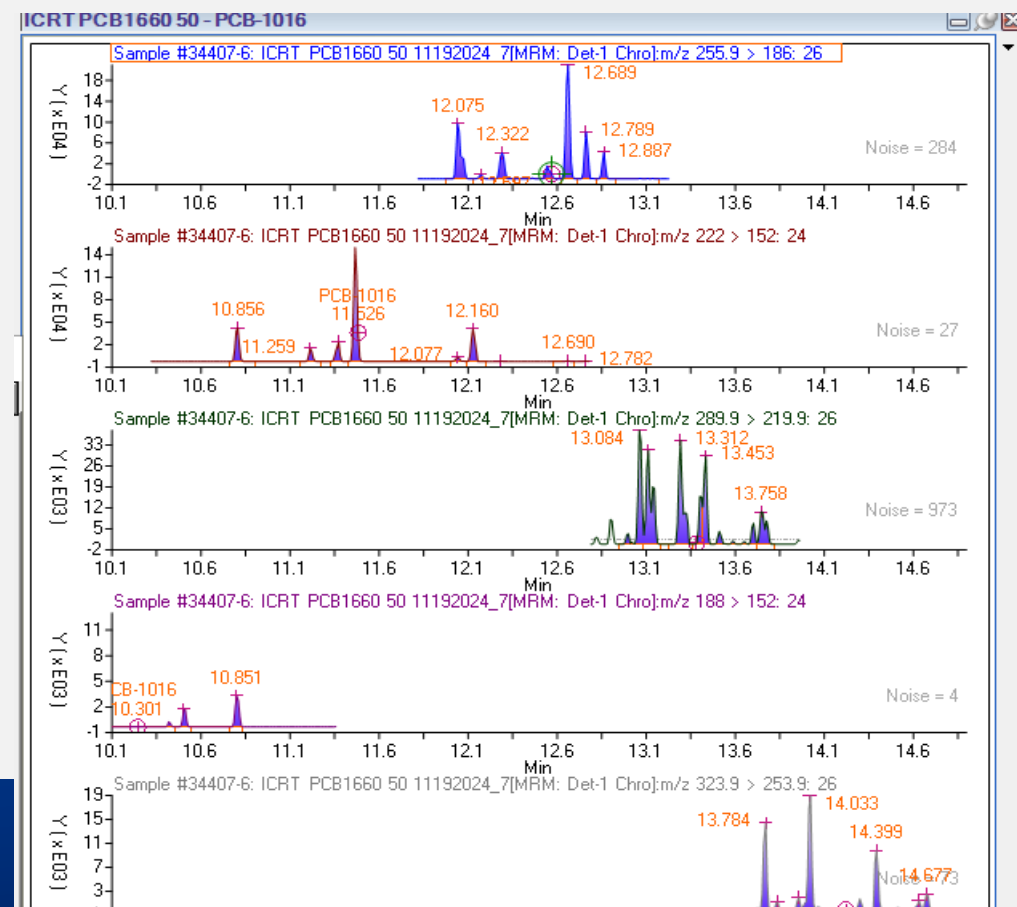
- Better resolution and lower MDLs = More analytes!

EPA 8011, 8081, 8082, 8141



GC/MSMS (EPA Method 8081)
Dieldrin @ 0.5 PPB
Calibration range 0.5-100 ppb
%RSD 6.3

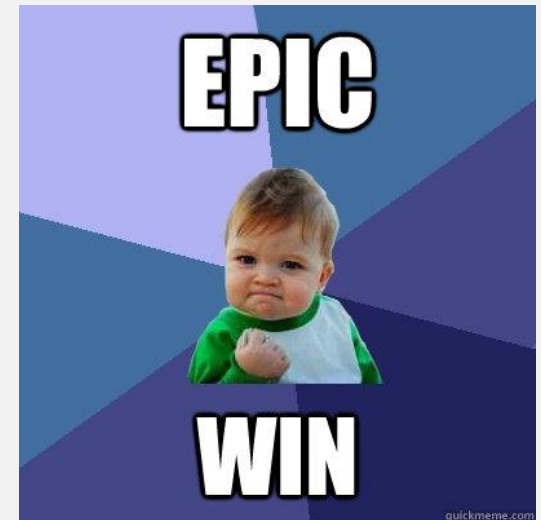
GC/MSMS (EPA Method 8082)
AR 1016 @ 50 PPB
Calibration range 1.0-1000 ppb
%RSD 6.6



Testing

What Changes?

- Less sample volume
 - 1 L or 250 mL > 40 or 60 mL
- Less Shipping & Broken Containers
- Less matrix interference
- More robust analysis
- Expanded Analyte List for EPA 8270E
- Faster & reliable results
- Compliant with 2024 EPA DCM Rule



Julian Martinez
Eurofins Environment Testing

Julian.Martinez@ET.EurofinsUS.com

THANK YOU



Environment Testing