Simplification of laboratory workflows for the analysis of common contaminants as required per EPA methods 8270E, 625, Appendix IX, 8081B, 8141B by the use of gas chromatography mass spectrometry

Andy Fornadel
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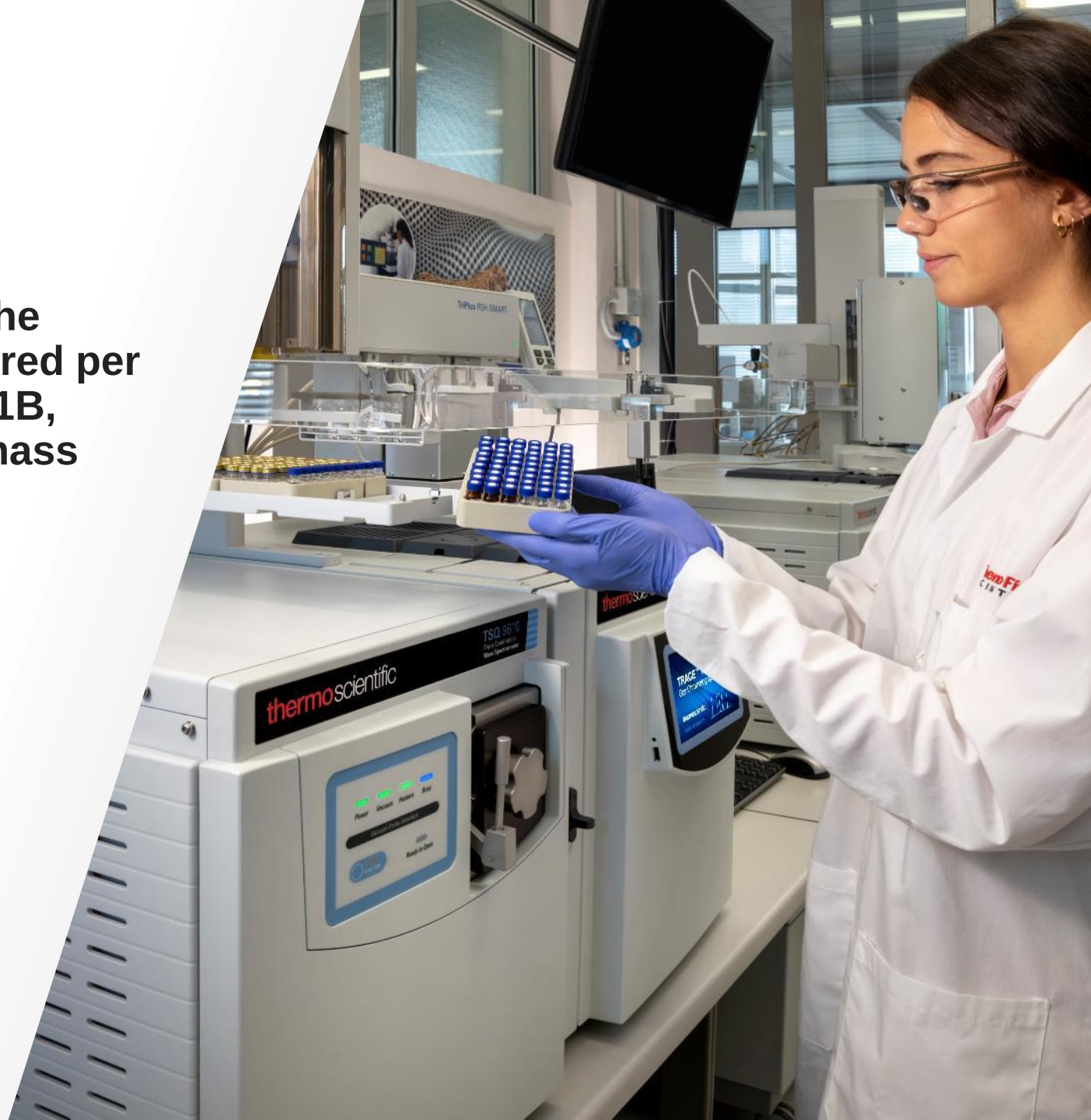
Thermo Fisher Scientific



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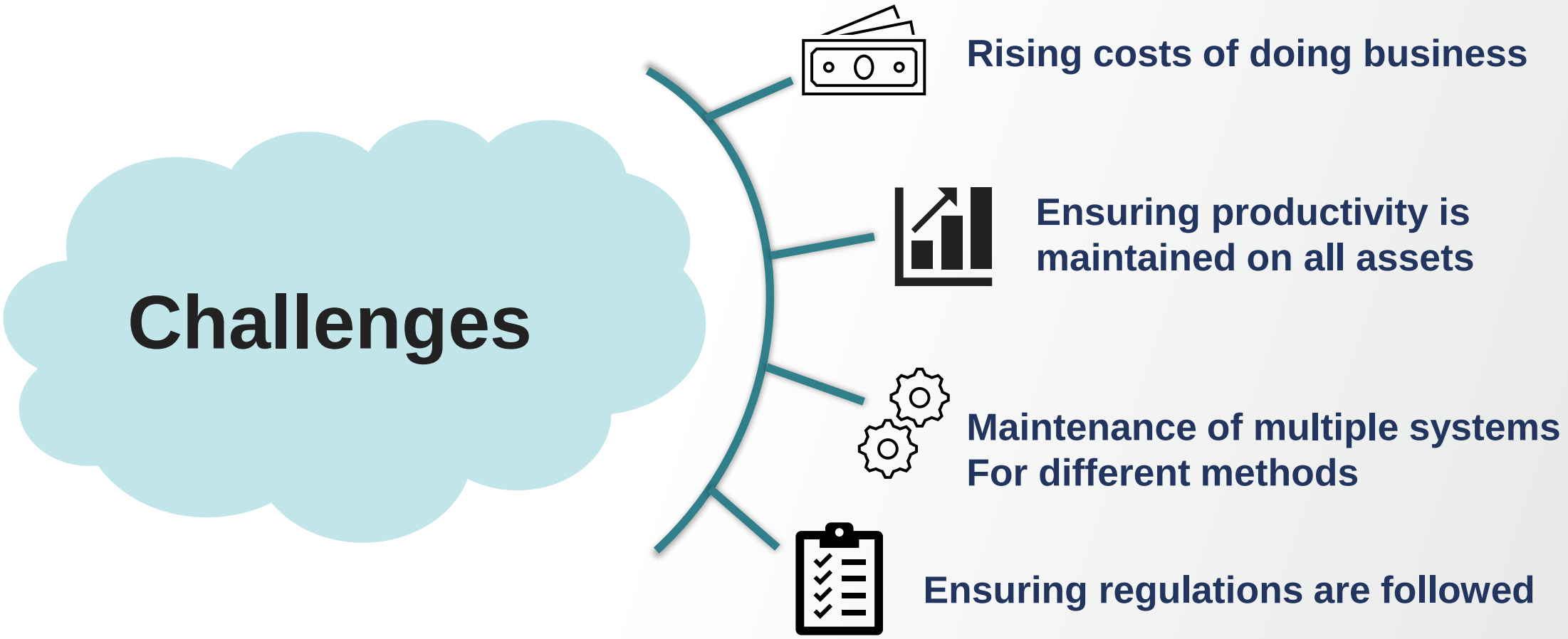
Overview

- **Challenges in the laboratory**
- **EPA 8081:**
  - Advantages of GC-MS/MS over GC ECD
  - How GC-MS/MS improves selectivity
  - Data examples including Method detection limits
- **EPA 8270E**
  - Compliant tuning
  - Critical pair separation
- **Consolidating EPA methods:**
  - Single run covering multiple methods
  - Helium saving technology
  - Single software across platforms
- **Conclusions**

Challenges faced by environmental testing laboratories



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Modernizing workflows onto a common platform

EPA 8270E (SVOC's)

EPA 625 (Base/Neutral/Acids)

Appendix IX (OP Pesticides)

EPA 8081B (OC Pesticides)

EPA 8141B (OP Pesticides)

EPA 8082A (PCB Aroclors)

EPA 8082A (PCB Congeners)

EPA 8082A (Chlordane / Toxaphene)

Single Instrument Configuration

- **Single platform for modernized workflow**

✓ **Same GC-MS/MS Platform**

TSQ9610 NV-AEI

TRACE 1610 GC w/ SSL

TriPlus RSH SMART or AS1610 Autosampler

✓ **Same Column**

TG-5SilMS 30 m × 0.25 mm ID x 0.25 µm

w/ 5 m × 0.25 mm ID GuardGold

✓ **Simplified software, operation, data processing and reporting**

Chromeleon 7.3.2

✓ **Flexibility to choose how and where to run your samples**

Single platform and configuration

- **Safer Lab Hardware**
  - No more ECDs
  - Forget about licensing fees, training, and safety concerns
- **Modern Methods**
  - Ditch the dual column methods
  - Reduce consumables cost and complexity
  - Faster calibration, analysis, and data processing
  - Optimized each SVOC method for seamless method transfer
- **Confident Results**
  - Fast, confident identification at ppb sensitivities

Improving Productivity through Data Quality

Study of **EPA 8081** on GC-ECD versus GC-MS/MS

- Dual Column
- Dual Detector

- Single Column
- MS/MS Detector

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Improving Productivity through Data Quality

Interference on GC-ECD

- Leads to systematic errors
  - Biases
- Can raise LODs
- Reduce signal/noise

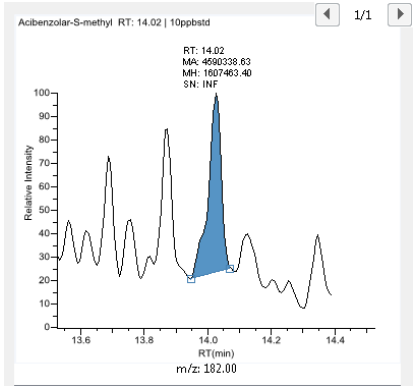
Selectivity to resolve interferences on TSQ 9610

- MS mass filtering to target specific precursor ions
- Fragmentation
- MS mass filtering of product ions
- All yields higher selectivity than ECD, reducing LODs, and increasing S/N

How does GC-MS/MS generate better data?

Single Quadrupole Detection

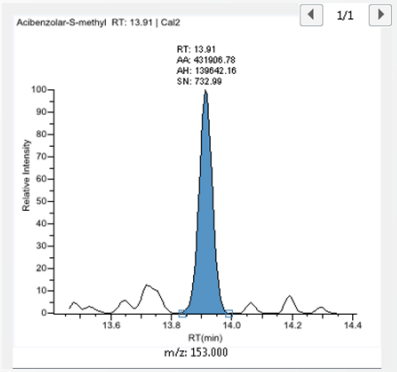
- Sensitivity in SIM (Selected Ion Monitoring)



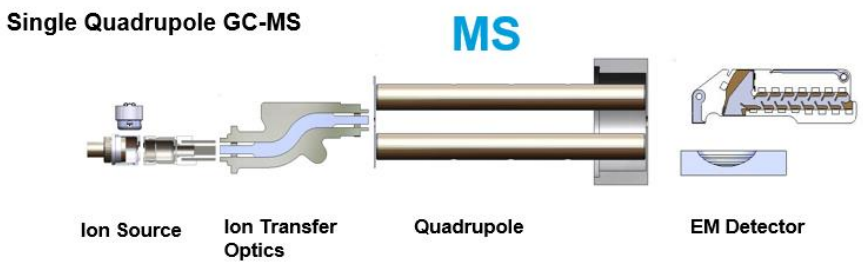
SIM on *m/z* 182

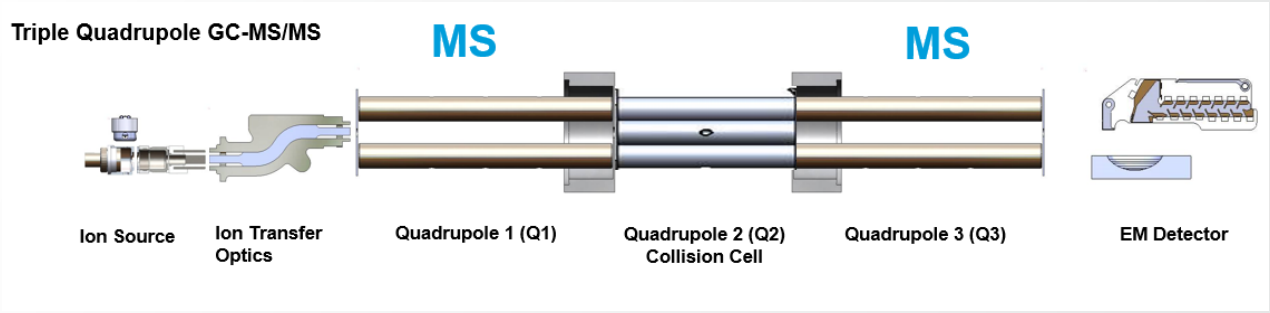
Triple Quadrupole Detection

- Sensitivity and Selectivity in SRM (Selected Reaction Monitoring)



SRM on *m/z* 182 > 153



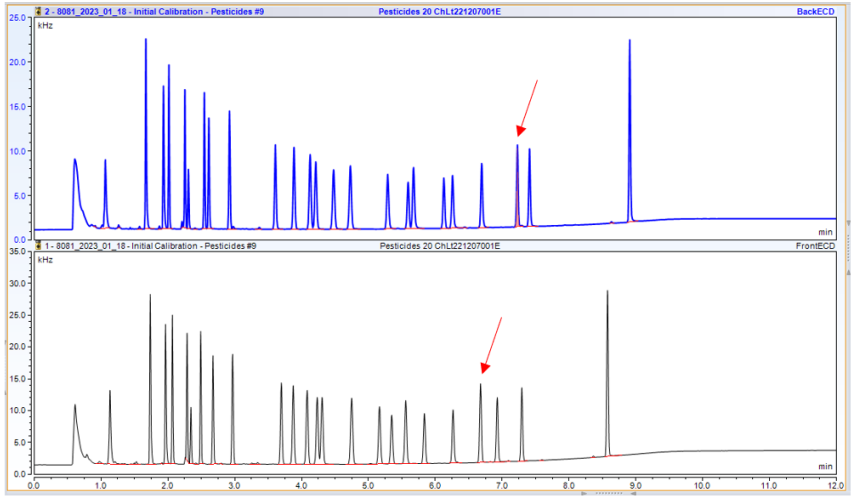


Improving Productivity through Data Quality

Conventional Detector Confirmation

- Retention Time on two columns of different phase

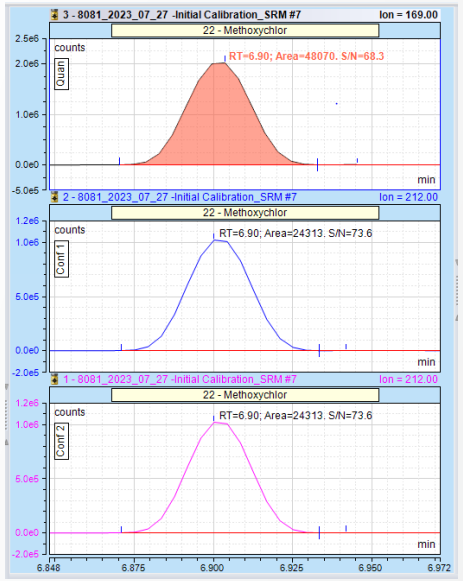
Methoxychlor

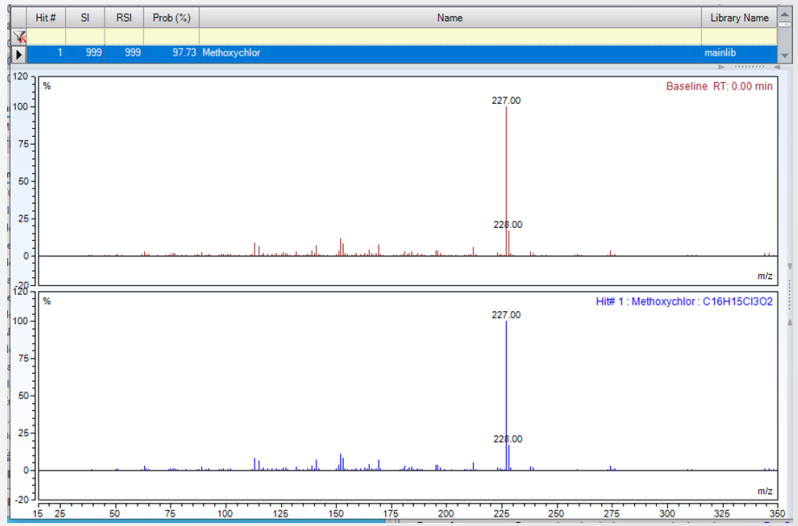


Mass Spectrometer Detector Confirmation

- Retention Time
- Unique Quantitation Ion
- Unique Confirmation ion(s)
- Ion ratio (Quantitation to Confirmation ion(s))
- Library Match in Full Scan

Methoxychlor





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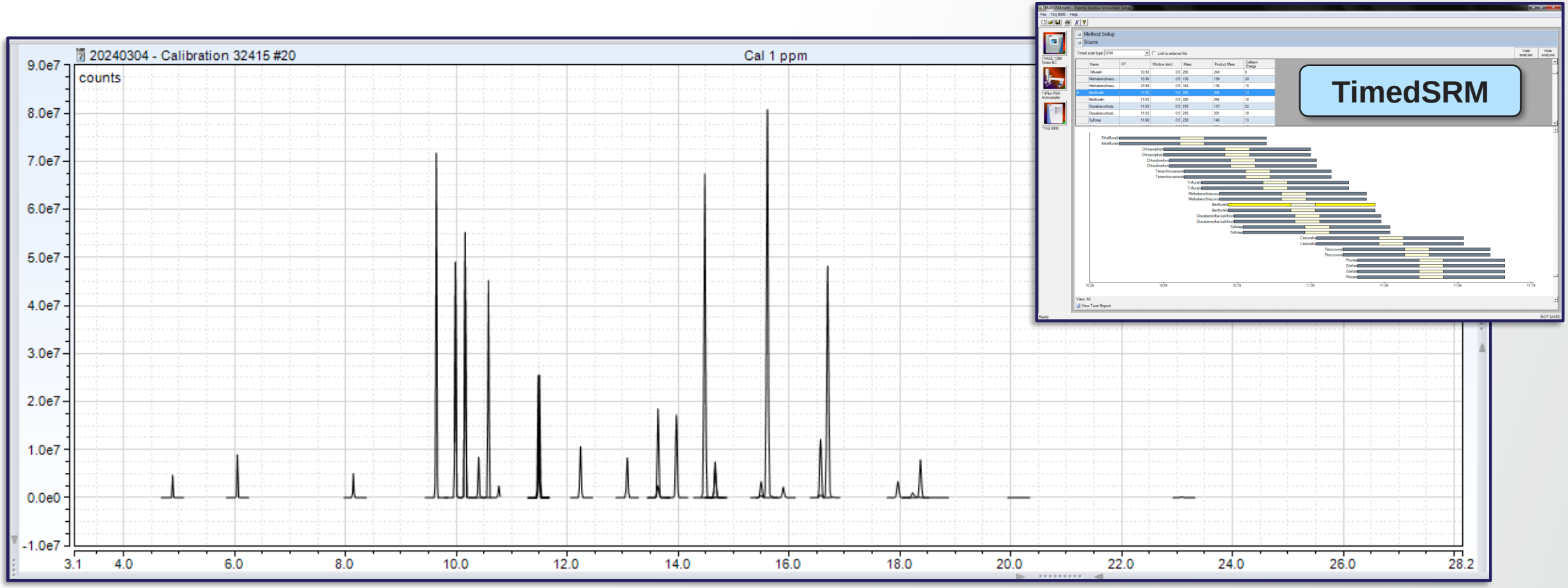
EPA 8081B Chromatography



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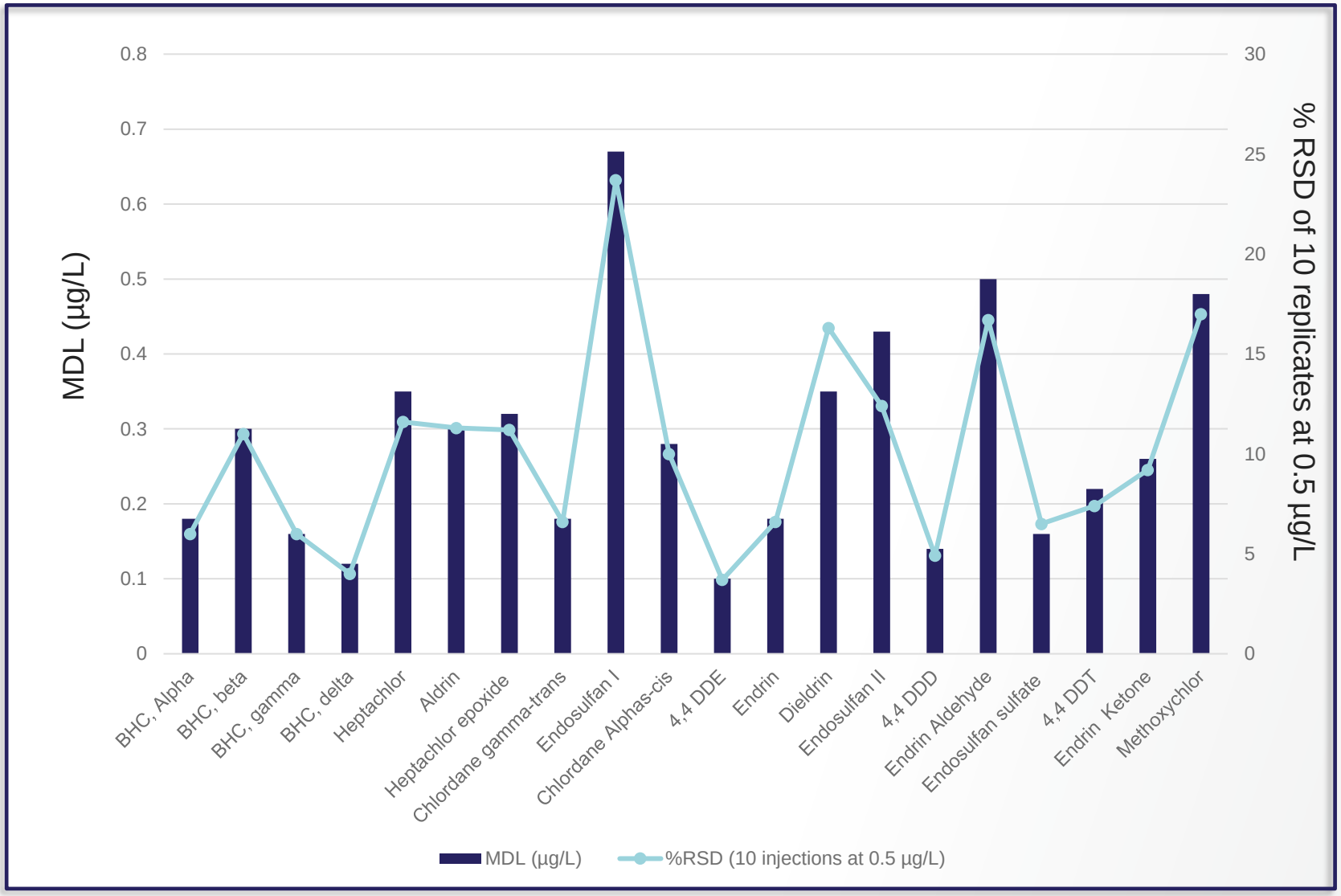
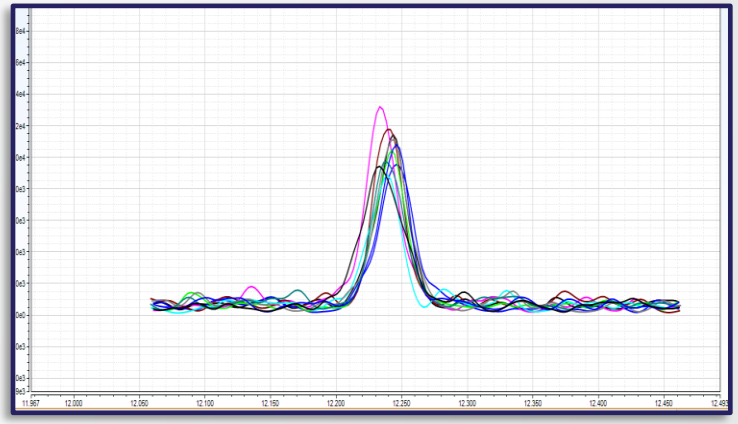
EPA 8081B Method detection limits





10 replicates for Aldrin at 0.5 µg/L

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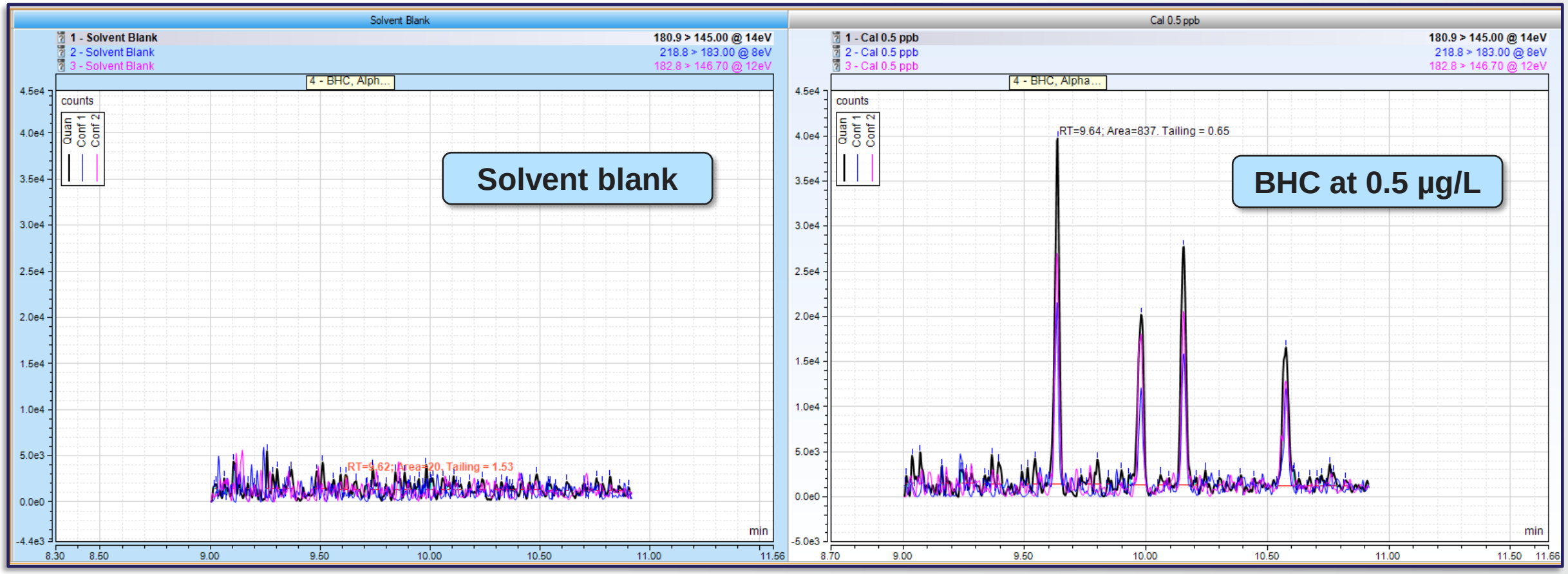
EPA 8081B Lowest calibration point

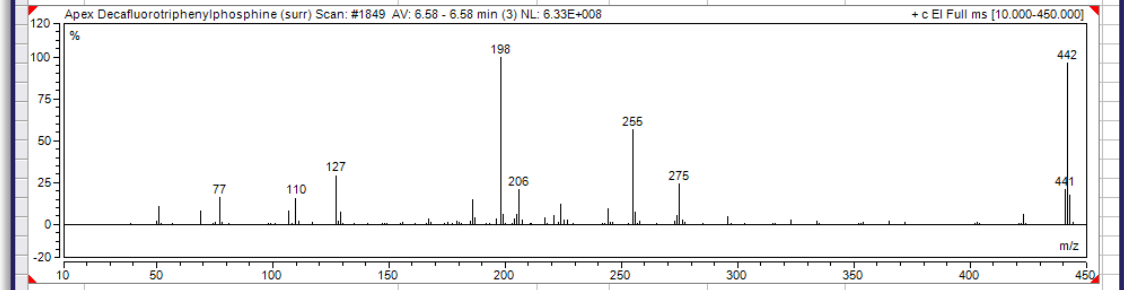


EPA 8270E: Built in tuning compliance

MS Tune Check Report (DFTPP)

Sequence Details			
Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
Directory:	App Note ENV	Created By:	Thermo
Data Vault:	2024	Updated On:	26/Mar/24 11:07:17
No. of Injections:	35	Updated By:	sarah.crumlett
Calibration Source:	20240221 - A Tune and ISTD Replicates		

Injection Details			
Injection Name:	Tune Check	Injection Volume:	1.00
Analysis Type:	MS Tune Check	Dilution Factor:	1.0000
Injection Type:	Unknown	Vial Number:	2
Injection Date/Time:	21/Feb/24 18:50	Processing Method:	EPA 8270E - DFTPP & Tailing V30
		Instrument Method:	DFTPP



No.	Name	Eval. Result	Operator	Ref. Value 1	Ref. Value 2	Result
1	m/z 68 - Less than 2% of m/z 69	0.3	<	2		Passed
2	m/z 69 - Present	51184357.7	>	0		Passed
3	m/z 70 - Less than 2% of m/z 69	0.6	<	2		Passed
4	m/z 197 - Less than 2% of m/z 198	0.0	<	2		Passed
5	m/z 198 - Base Peak or Present	632495297.7	>	0		Passed
6	m/z 199 - 5 to 9% of m/z 198	5.7	between	5	9	Passed
7	m/z 365 - Greater than 1% of m/z 198	2.1	>	1		Passed
8	m/z 441 - Less than 150% of m/z 443	121.5	<	150		Passed
9	m/z 442 - Base Peak or Present	611513468.7	>	0		Passed
10	m/z 443 - 15 to 24% of m/z 442	18.1	between	15	24	Passed
					Overall Result:	Passed

EPA 8270E: Built in tuning compliance

MS Tune Check Report (DFTPP)

Sequence Details

Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
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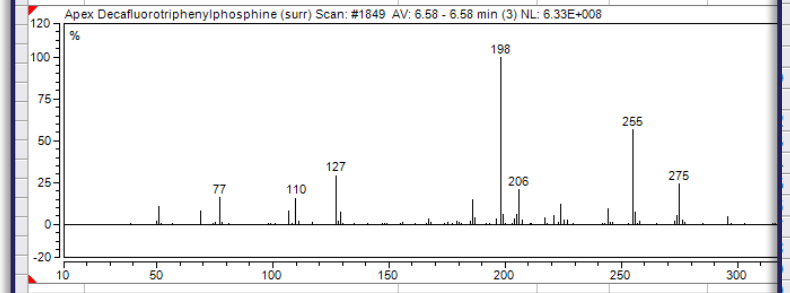
No. of Injections:	35	Updated By:	sarah.crumlett
Calibration Source:	20240221 - A Tune and ISTD Replicates		

Injection Details

Injection Name:	Tune Check	Injection V	
Analysis Type:	MS Tune Check	Dilution Fa	
Injection Type:	Unknown	Vial Numb	
Injection Date/Time:	21/Feb/24 18:50	Processin	
		Instrumen	



No.	Name	Eval. Result	Operator	Ref. Value 1	Ref. V
1	m/z 68 - Less than 2% of m/z 69	0.3	<	2	
2	m/z 69 - Present	51184357.7	>	0	
3	m/z 70 - Less than 2% of m/z 69	0.6	<	2	
4	m/z 197 - Less than 2% of m/z 198	0.0	<	2	
5	m/z 198 - Base Peak or Present	632495297.7	>	0	
6	m/z 199 - 5 to 9% of m/z 198	5.7	between	5	9
7	m/z 365 - Greater than 1% of m/z 198	2.1	>	1	
8	m/z 441 - Less than 150% of m/z 443	121.5	<	150	
9	m/z 442 - Base Peak or Present	611513468.7	>	0	
10	m/z 443 - 15 to 24% of m/z 442	18.1	between	15	2
					Overall R

Breakdown Report

Sequence Details

Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
Directory:	App Note ENV	Created By:	Thermo
Data Vault:	2024	Updated On:	24/Feb/24 09:14:25
No. of Injections:	35	Updated By:	sarah.crumlett
Calibration Source:	20240221 - A Tune and ISTD Replicates		

Injection Details

Injection Name:	Tune Check	Injection Volume:	1.00
Analysis Type:	n.a.	Dilution Factor:	1.0000
Injection Type:	Unknown	Vial Number:	2
Injection Date/Time:	21/Feb/24 18:50	Processing Method:	EPA 8270E - DFTPP & Tailing V30
		Instrument Method:	DFTPP

DDT Breakdown

Compound	Type	Response	%Breakdown	Max %Breakdown	Result
4,4'-DDT	Native	81546587	3.345	20	Pass
4,4'-DDE	Breakdown	130821			
4,4'-DDD	Breakdown	2691157			

Endrin Breakdown

Compound	Type	Response	%Breakdown	Max %Breakdown	Result
Endrin	Native	72779601	5.933	20	Pass
Endrin aldehyde	Breakdown	509389			
Endrin ketone	Breakdown	4080643			

EPA 8270E: Built in tuning compliance

MS Tune Check Report (DFTPP)

Sequence Details

Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
Directory:	App Note ENV	Created By:	Thermo
Data Vault:	2024	Updated On:	26/Mar/24 11:07:17
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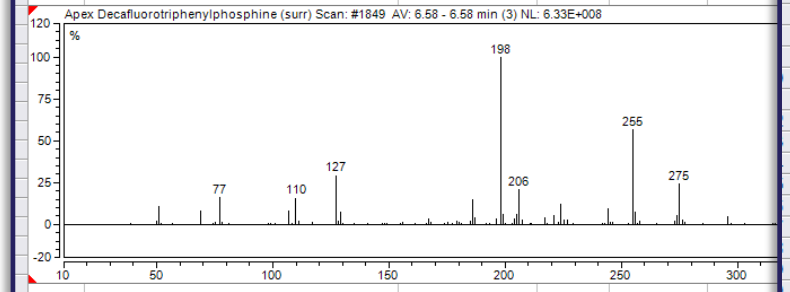
Injection Details

Injection Name:	Tune Check	Injection V	
Analysis Type:	MS Tune Check	Dilution Fa	
Injection Type:	Unknown	Vial Numb	
Injection Date/Time:	21/Feb/24 18:50	Processin	
		Instrumen	



No.	Name	Eval. Result	Operator	Ref. Value 1	Ref. V
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2	m/z 69 - Present	51184357.7	>	0	
3	m/z 70 - Less than 2% of m/z 69	0.6	<	2	
4	m/z 197 - Less than 2% of m/z 198	0.0	<	2	
5	m/z 198 - Base Peak or Present	632495297.7	>	0	
6	m/z 199 - 5 to 9% of m/z 198	5.7	between	5	9
7	m/z 365 - Greater than 1% of m/z 198	2.1	>	1	
8	m/z 441 - Less than 150% of m/z 443	121.5	<	150	
9	m/z 442 - Base Peak or Present	611513468.7	>	0	
10	m/z 443 - 15 to 24% of m/z 442	18.1	between	15	2
					Overall R

Breakdown Report

Sequence Details

Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
Directory:	App Note ENV	Created By:	Thermo
Data Vault:	2024	Updated On:	24/Feb/24 09:14:25
No. of Injections:	35	Updated By:	sarah.crumlett
Calibration Source:	20240221 - A Tune and ISTD Replicates		

Injection Details

Injection Name:	Tune Check
Analysis Type:	n.a.
Injection Type:	Unknown
Injection Date/Time:	21/Feb/24 18:50

Compound	Type
4,4'-DDT	Native
4,4'-DDE	Breakdown
4,4'-DDD	Breakdown

Compound	Type
Endrin	Native
Endrin aldehyde	Breakdown
Endrin ketone	Breakdown

Peak Tailing Report

Sequence Details

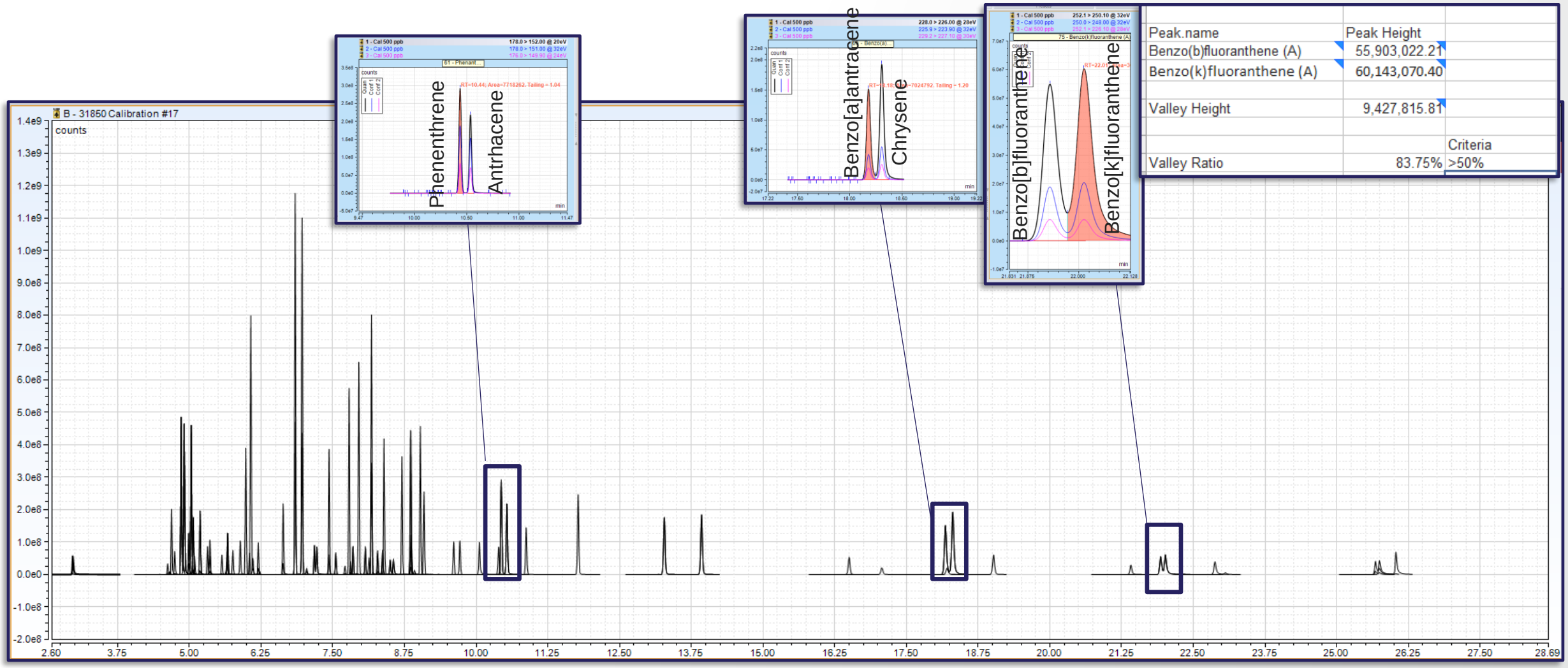
Sequence Name:	20240221 - A Tune and ISTD Replicates	Created On:	21/Feb/24 17:53:39
Directory:	App Note ENV	Created By:	Thermo
Data Vault:	2024	Updated On:	24/Feb/24 09:14:25
No. of Injections:	35	Updated By:	sarah.crumlett
Calibration Source:	20240221 - A Tune and ISTD Replicates		

Injection Details

Injection Name:	Tune Check	Injection Volume:	1.00
Analysis Type:	n.a.	Dilution Factor:	1.0000
Injection Type:	Unknown	Vial Number:	2
Injection Date/Time:	21/Feb/24 18:50	Processing Method:	EPA 8270E - DFTPP & Tailing V30
		Instrument Method:	DFTPP

Compound TIC	Ret Time min TIC	Asymmetry (AIA) TIC	Limit TIC	Outside Limit?
Pentachlorophenol	6.201	0.97	2.00	
Benzidine	7.466	1.00	2.00	

EPA 8270E separation of structural isomers



Peak.name	Peak Height	
Benzo(b)fluoranthene (A)	55,903,022.21	
Benzo(k)fluoranthene (A)	60,143,070.40	
Valley Height	9,427,815.81	
		Criteria
Valley Ratio	83.75%	>50%

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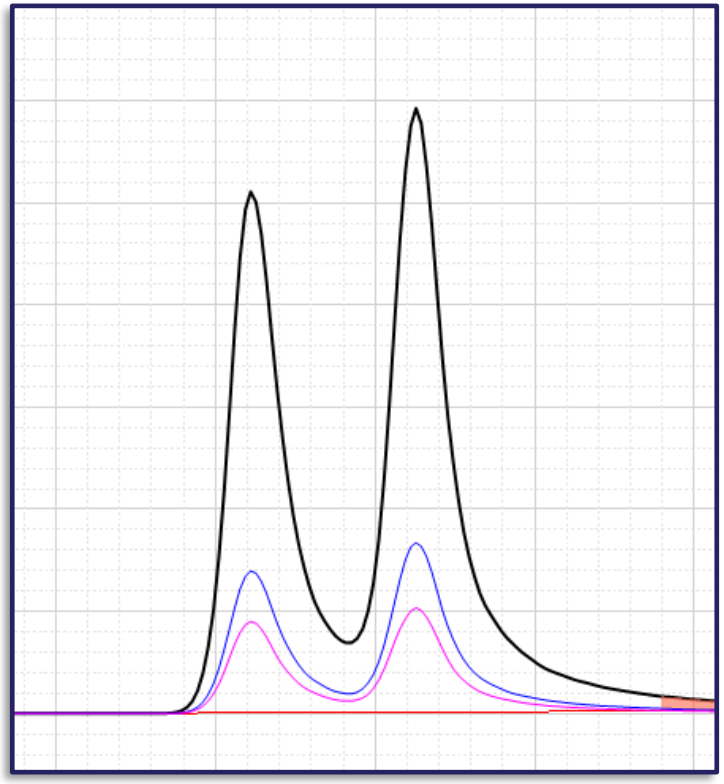
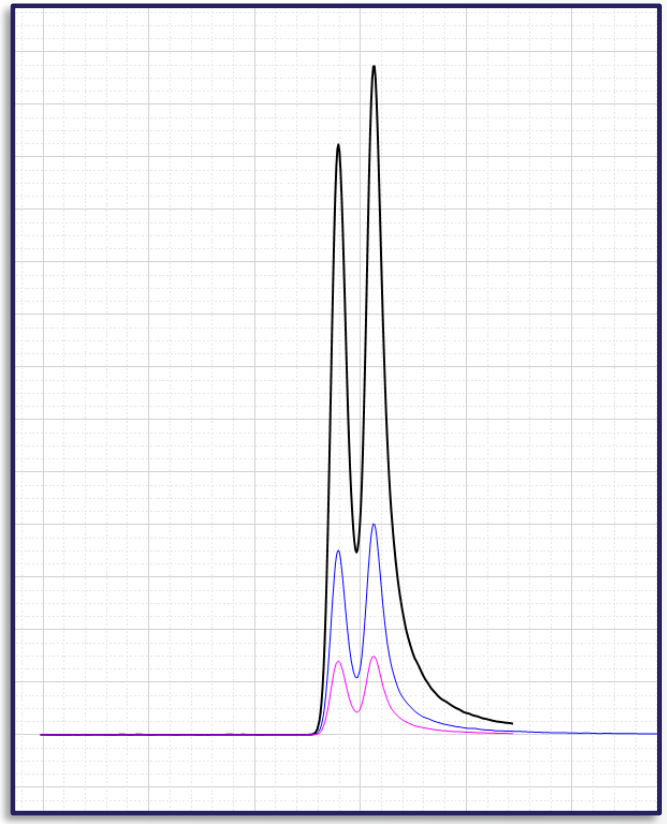
EPA 8270E Critical Pair Resolution

Isomers must be at least 50% resolved on the mid-point of the ICAL

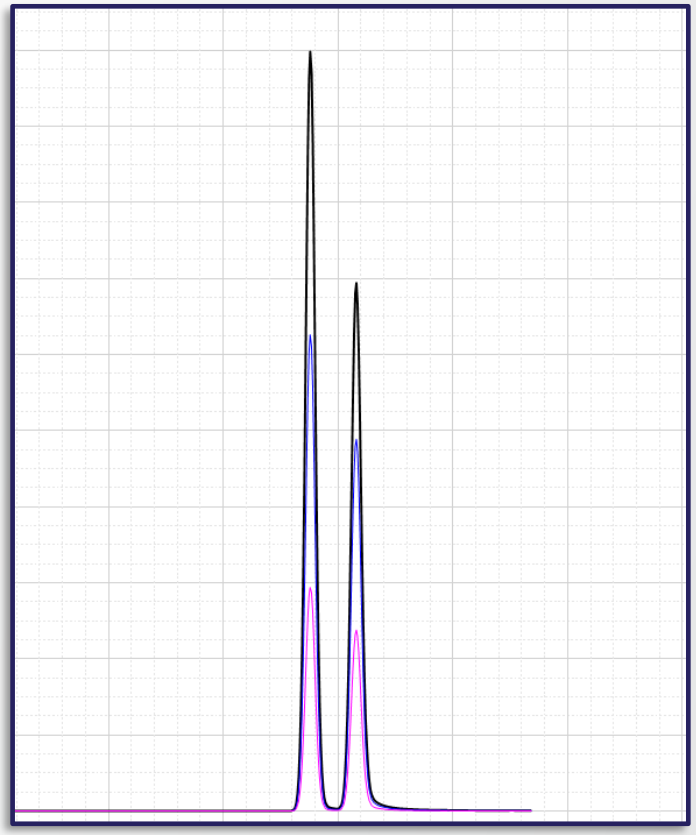
- Benzo(a)anthracene and Chrysene



- Benzo(b)fluoranthene and Benzo(k)fluoranthene



- Phenanthrene and Anthracene



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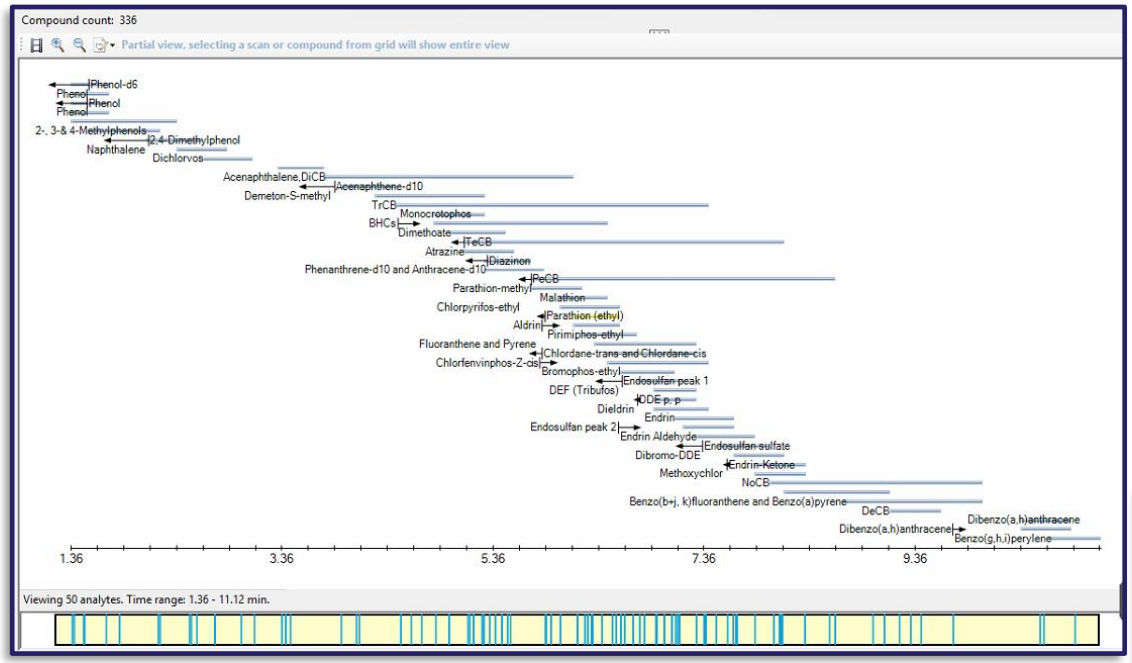
Consolidating methods Improving Productivity

Better results, less time



Timed SRM

- Consolidate methods into single injection
  - In some cases, common SVOC workflows can be consolidated into a single injection
  - 336 SVOCs analyzed in a single run
  - PCBs, pesticides, and PAHs/Phenols

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Combining EPA methods Improving Productivity

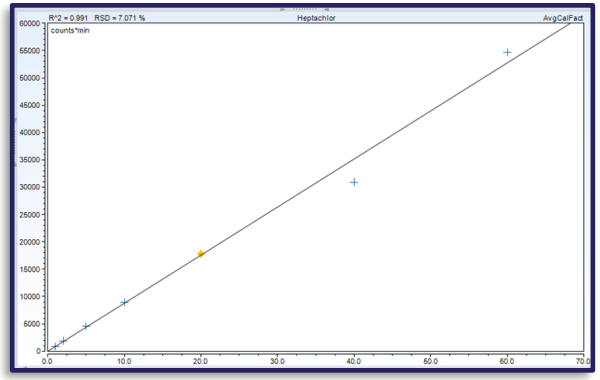
Better results, less time

Peak Name	Cal.Type	R^2 RSD (%)	R^2 / %RSD Failed?	Manually Integrated?
MS Quantitation	MS Quantitation	MS Quantitation Peak	MS Quantitation	
Hexachlorocyclopentadiene	AvgCalFact	5.85 (%RSD)		
TCMX (S)	AvgCalFact	7.55 (%RSD)		
alpha-BHC	AvgCalFact	7.21 (%RSD)		
Hexachlorobenzene	AvgCalFact	6.74 (%RSD)		
beta-BHC	AvgCalFact	6.68 (%RSD)		
gamma-BHC	AvgCalFact	6.73 (%RSD)		
delta-BHC	AvgCalFact	5.82 (%RSD)		
Heptachlor	AvgCalFact	7.07 (%RSD)		
Aldrin	AvgCalFact	8.75 (%RSD)		
Heptachlor Epoxide	AvgCalFact	7.71 (%RSD)		
gamma-Chlordane	AvgCalFact	8.72 (%RSD)		
alpha-Chlordane	AvgCalFact	6.31 (%RSD)		
Endosulfan I	AvgCalFact	4.58 (%RSD)		
4,4-DDE	AvgCalFact	5.15 (%RSD)		
Dieldrin	AvgCalFact	8.38 (%RSD)		
Endrin	AvgCalFact	5.22 (%RSD)		
4,4-DDD	AvgCalFact	5.55 (%RSD)		
Endosulfan II	AvgCalFact	10.35 (%RSD)		
Endrin Aldehyde	AvgCalFact	12.57 (%RSD)		
4,4-DDT	AvgCalFact	8.57 (%RSD)		
Endosulfan Sulfate	AvgCalFact	8.22 (%RSD)		
Methoxychlor	AvgCalFact	9.99 (%RSD)		
Endrin Ketone	AvgCalFact	12.39 (%RSD)		
DCBP (S)	AvgCalFact	16.38 (%RSD)		
		Number of failed tests:	0	0

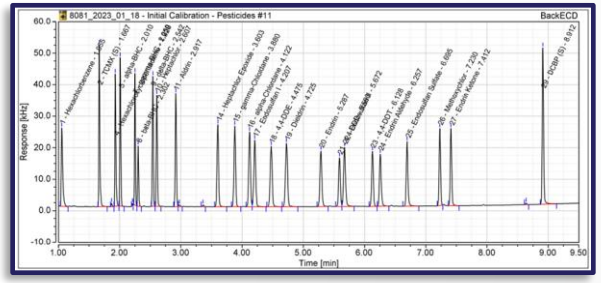
Compound	Ret.Time min	Quantitation Peak (m/z)	Response	Cal Amount	Amount
MS Quantitation	MS Quantitation	MS Quantitation	MS Quantitation	MS Quantitation Pea	MS Quantitation
Hexachlorocyclopentadiene	2.828	143.000	519	1.00	0.97
alpha-BHC	4.138	183.000	513	1.00	0.93
Hexachlorobenzene	4.189	248.800	1450	1.00	1.02
beta-BHC	4.299	145.000	741	1.00	1.07
gamma-BHC	4.394	145.000	773	1.00	0.94
delta-BHC	4.576	147.000	346	1.00	0.99
Heptachlor	5.003	237.000	817	1.00	0.93
Aldrin	5.316	191.000	239	1.00	0.94
Heptachlor Epoxide	5.601	263.000	195	1.00	0.88
gamma-Chlordane	5.778	237.000	270	1.00	0.90
alpha-Chlordane	5.875	266.000	272	1.00	0.97
Endosulfan I	5.888	206.000	104	1.00	0.99
4,4-DDE	5.977	248.000	596	1.00	0.98
Dieldrin	6.077	193.000	212	1.00	1.13
Endrin	6.228	173.000	63	1.00	1.07
4,4-DDD	6.266	199.000	514	1.00	1.04
Endosulfan II	6.288	123.000	58	1.00	1.01
Endrin Aldehyde	6.385	138.000	68	1.00	1.24
4,4-DDT	6.534	165.000	1837	1.00	1.14
Endosulfan Sulfate	6.545	237.000	478	1.00	1.02
Methoxychlor	6.903	169.000	2867	3.00	3.25
Endrin Ketone	6.922	281.000	50	1.00	1.13

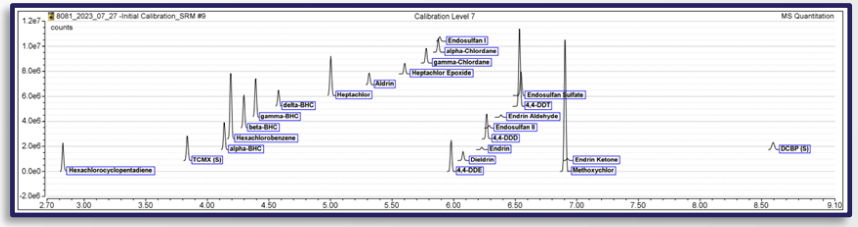
Calibration results from 1-60 ppb using TSQ 9610 (left) and 1 ppb check (right)

- Unambiguous results for known and unknowns
- GC-MS/MS provides equivalent or better data than GC-ECD without the challenges
- Positive identification in a single run
- Better data reduces time spent on data review



Calibration curve for heptachlor





Comparison of 60 ppb analytes using ECD (left) and MS (right)

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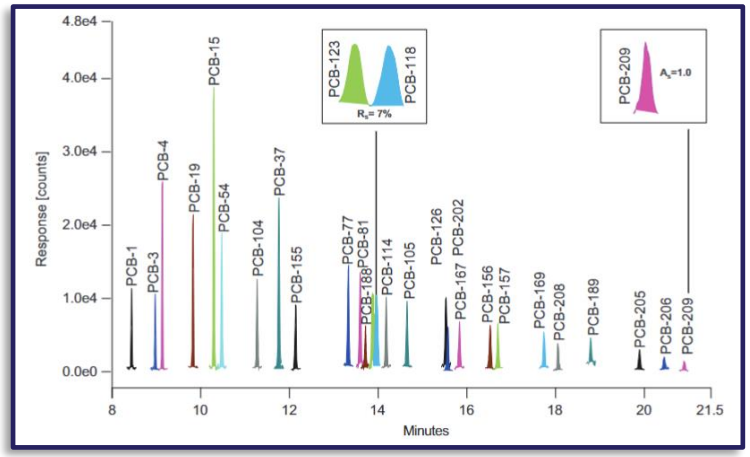
Sustainable operation

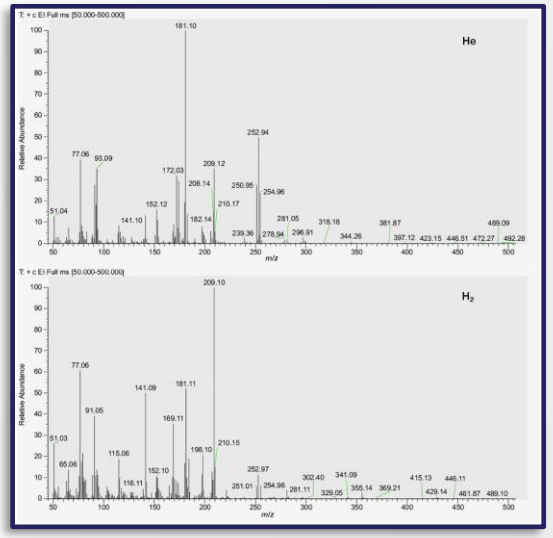


PCBs analyzed using TSQ 9610 with HeSaver/H2Safer at 0.2 ng/mL, extending lifetime of He cylinder by ~4.5x

Reduce carrier gas usage

- A unique solution for reducing helium usage, conversion to hydrogen
  - Conserve carrier gas during analysis and standby
  - Reduce helium consumption by up to 70% without revalidating methods
  - Safely convert to hydrogen

	Helium consumption with standard SSL	Helium consumption with HeSaver SSL	Gas savings
Daily He usage	44.0 L	12.5 L	-72%
He cylinder life	~7.5 months	~2 years	



Deltamethrin full scan spectra using He (top) and H_2 (bottom)

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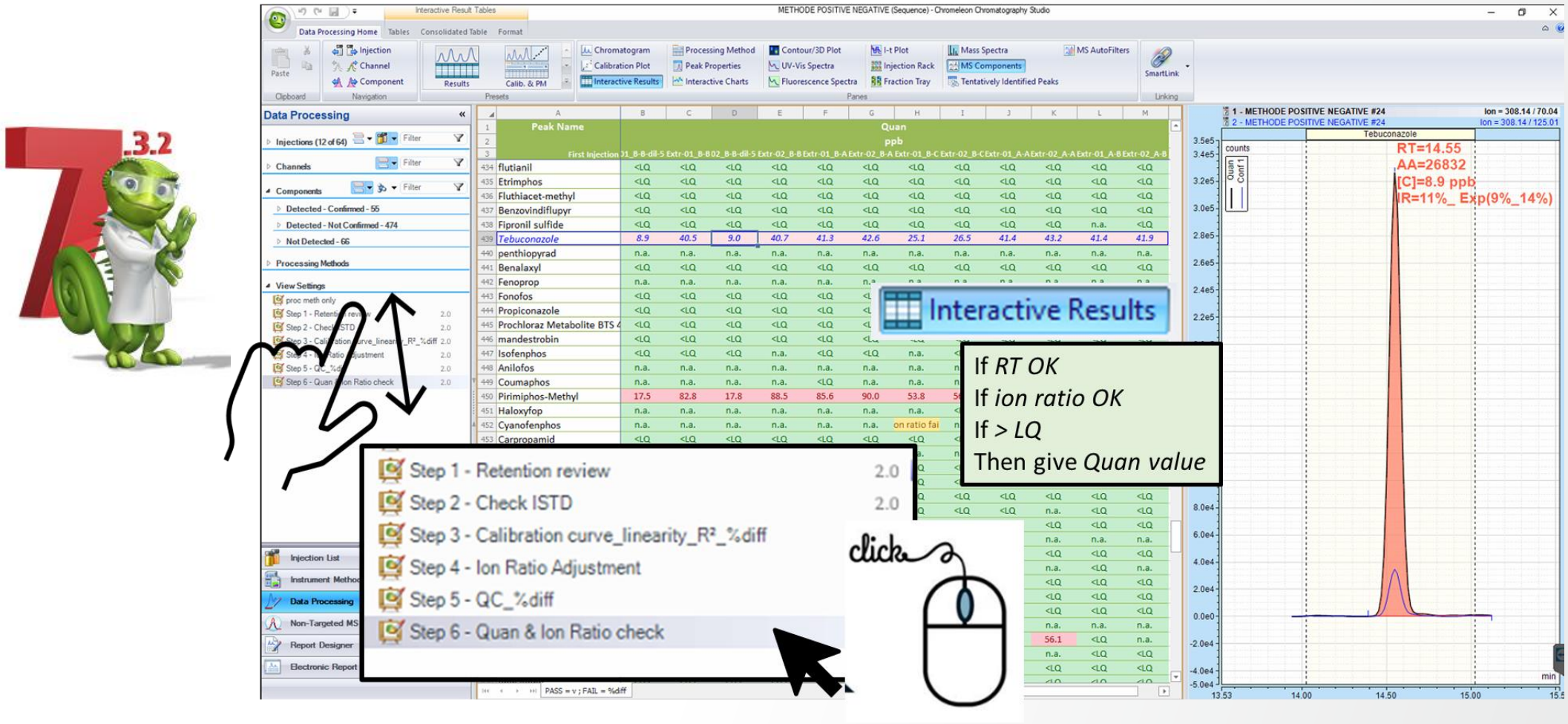
Simplified data review: Single software

Same Data Management Worflows for IC, LC, GC, and related MS workflows



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Moving environmental methods to GC-MS/MS

NeverVent technology

- Increasing instrument uptime

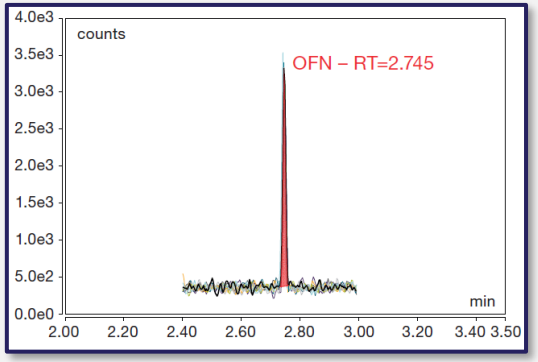
Off–axis ion guide pre-filter

- Eliminates the neutral noise

XLXR detector

- Extended dynamic range and lifetime

Excellent sensitivity



8 x 1 fg on-column OFN injections with %RSD of 4.1%. IDL is 0.12 fg

900 continuous injection no maintenance

Modern GC design

- Unique modular injector and detector design
- Easy to use touch screen
- Real time instrument monitoring and video guides

Software productivity tools

- Compliant ready software
- SmartStatus and SmartTune

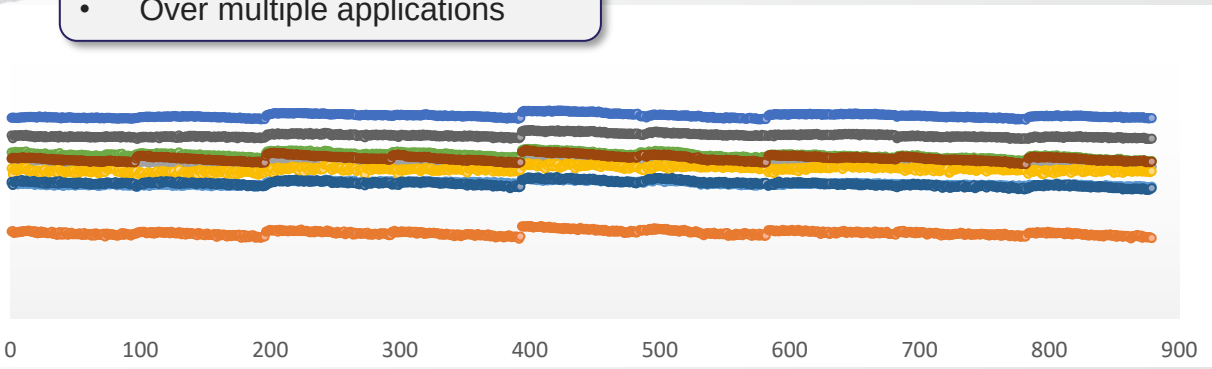
Sustainable operation

- Partnership with MyGreenLab
- Helium saving capabilities

ACT.
The Environmental Impact Factor Label

Proven robustness

- Over multiple applications



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Conclusions

- **The modern laboratory demands the next generation of analytical capability**

- **Modernizing common SVOC workflows can be realized using the TSQ 9610 GC-MS/MS**
  - EPA 8270E, EPA 625, Appendix IX, EPA 8081B, EPA 8141B,
  - Covering SVOCs, organochlorine pesticides, organophosphorus compounds, PCBs

- **Simplifies and accelerates routine operation for an increasingly stringent market**
  - Generate better data to reduce common challenges
  - Improve sample throughput
    - Automated sample preparation, faster calibrations and chromatography, single-run confirmation, unique features of TSQ 9610
  - Streamline operations
    - Simpler training, fewer specialized operators, harmonized consumables and laboratory supplies, unified chromatography software (IC, GC, GCMS, LC, LCMS)
  - Potential to consolidate of methods for multiple assays in a single injection
  - Peace-of-mind for future operations as methods become more stringent

Thank you