

Innovations in GC Inlet Liner Technology

Matrix Trapping Without the Formation of New Active Sites

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EPA 8270D/E: Semi-volatile compounds analysis by GC/MS

Requirements of the method

- **Instrument Performance Checkout**
 - Pass DFTPP tuning check
 - Pass DDT breakdown percentage (<20% total)
 - Pentachlorophenol and benzidine tailing factors < 2.0
 - Isomer pair resolution
- **Calibration curve: 0.1 to 100ppm**
 - Achieve linearity and verify responses $\pm 20\%$
 - Use average response factors, or linear regression fit, where necessary
 - Continuing Calibration Verification: Cal drift within $\pm 20\%$ for >80%* compounds
 - Study used >90% compounds
 - Internal Standards: Area of IS peak area drift within $\pm 50\%$

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Ability of new fritted liner to achieve 8270 requirements

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Method parameters

Parameter	Value
Injection volume	1 µL
Inlet	Split/splitless 280 °C; Pulsed splitless 30 psi until 0.6 minutes; Purge 50 mL/min at 0.6 minutes; Switched septum purge 3 mL/min
Inlet liner	Agilent Ultra Inert splitless single-taper liner with frit (p/n 5190-5112)
Column temperature program	40 °C (hold for 0.5 minutes), 10 °C/min to 100 °C, 25 °C/min to 260 °C, 5 °C/min to 280 °C, 15 °C/min to 320 °C (hold 2 min)
Column	J&W DB-8270D 30 m × 0.25 mm × 0.25 µm column
Carrier gas and flow rate	Helium at 1.3 mL/min, constant flow
MS parameters	
Transfer line temperature	320 °C
Ion source temperature	300 °C
Quadrupole temperature	150 °C
Drawout lens	9mm

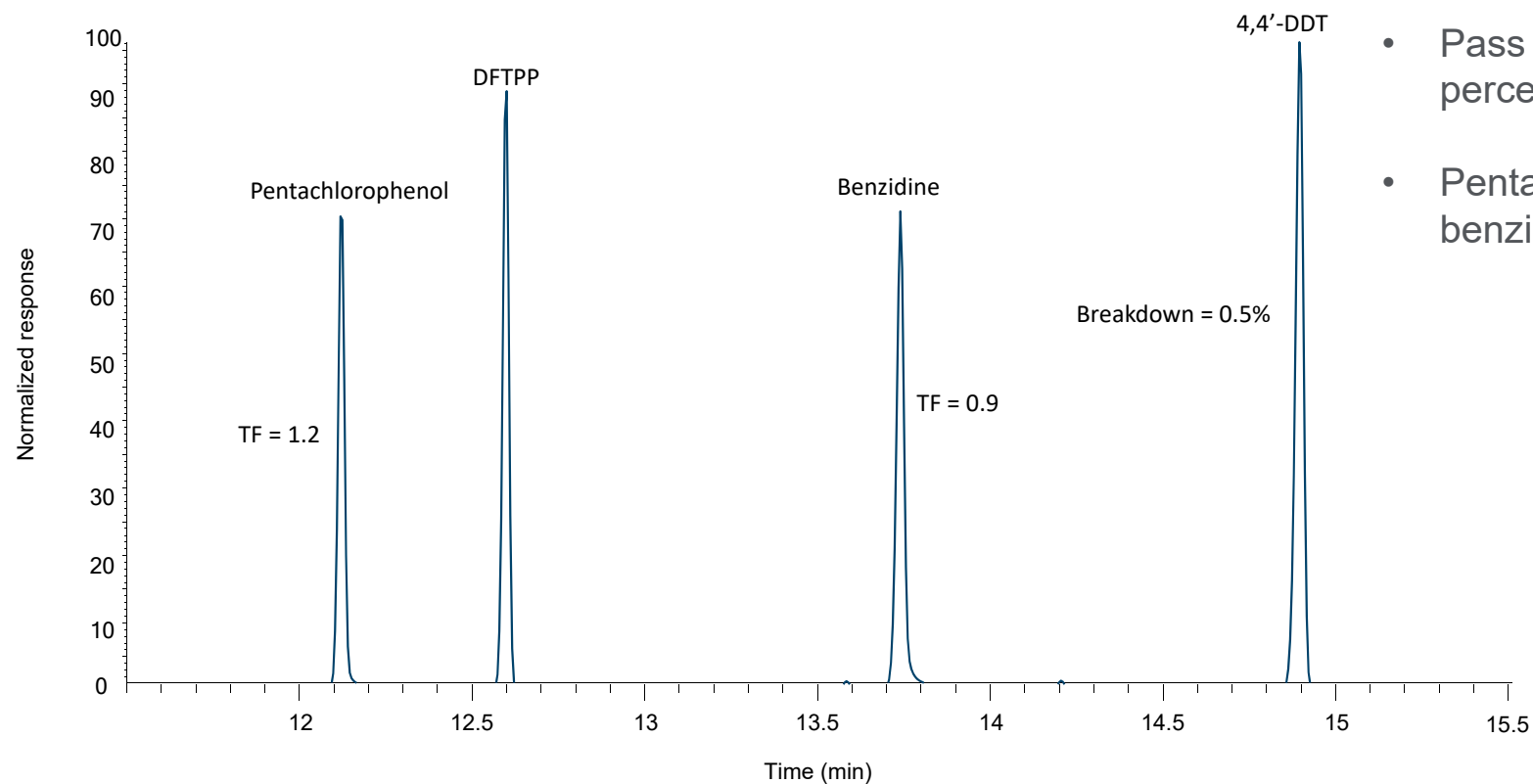


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8270D/E Initial Qualification: 25ppm DFTPP tuning standard



- Pass DFTPP tuning check

- Pass DDT breakdown percentage (< 20% total) ✓

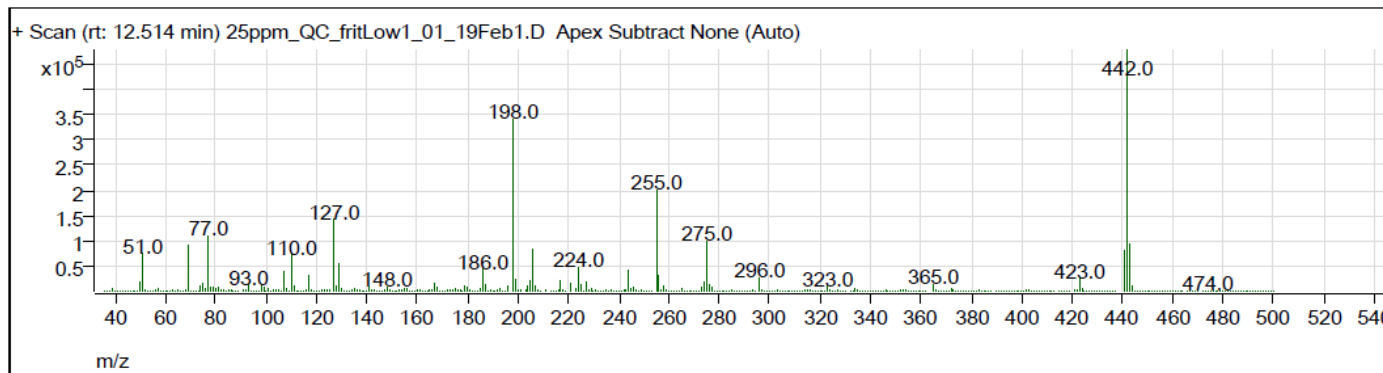
- Pentachlorophenol and benzidine tailing factors < 2.0 ✓

DFTPP ion ratios: Initial Qualification

Pass DFTPP tuning check

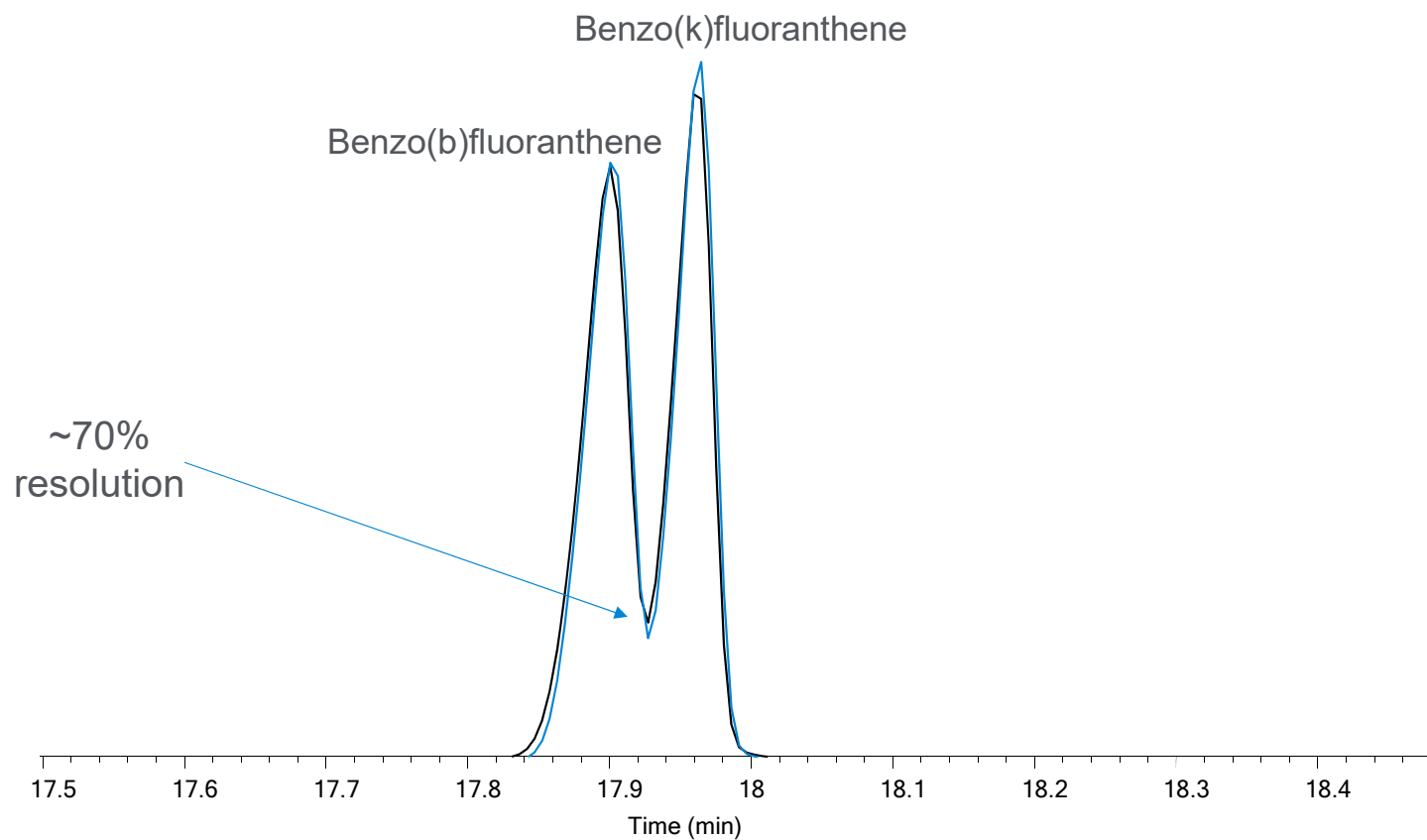


Target mass	Relative to mass	Lower limit %	Upper limit %	Relative abundance %	Pass/fail
51	198	10	80	27.4	Pass
68	69	0	2	1.7	Pass
70	69	0	2	0.5	Pass
127	442	40	60	41.0	Pass
197	442	0	1	0.7	Pass
198	442	50	100	74.7	Pass
199	198	5	9	6.8	Pass
275	442	10	30	28.8	Pass
365	198	1	100	4.1	Pass
441	442	1	100	84.9	Pass
442	442	100	100	100	Pass
443	442	17	23	19.4	Pass

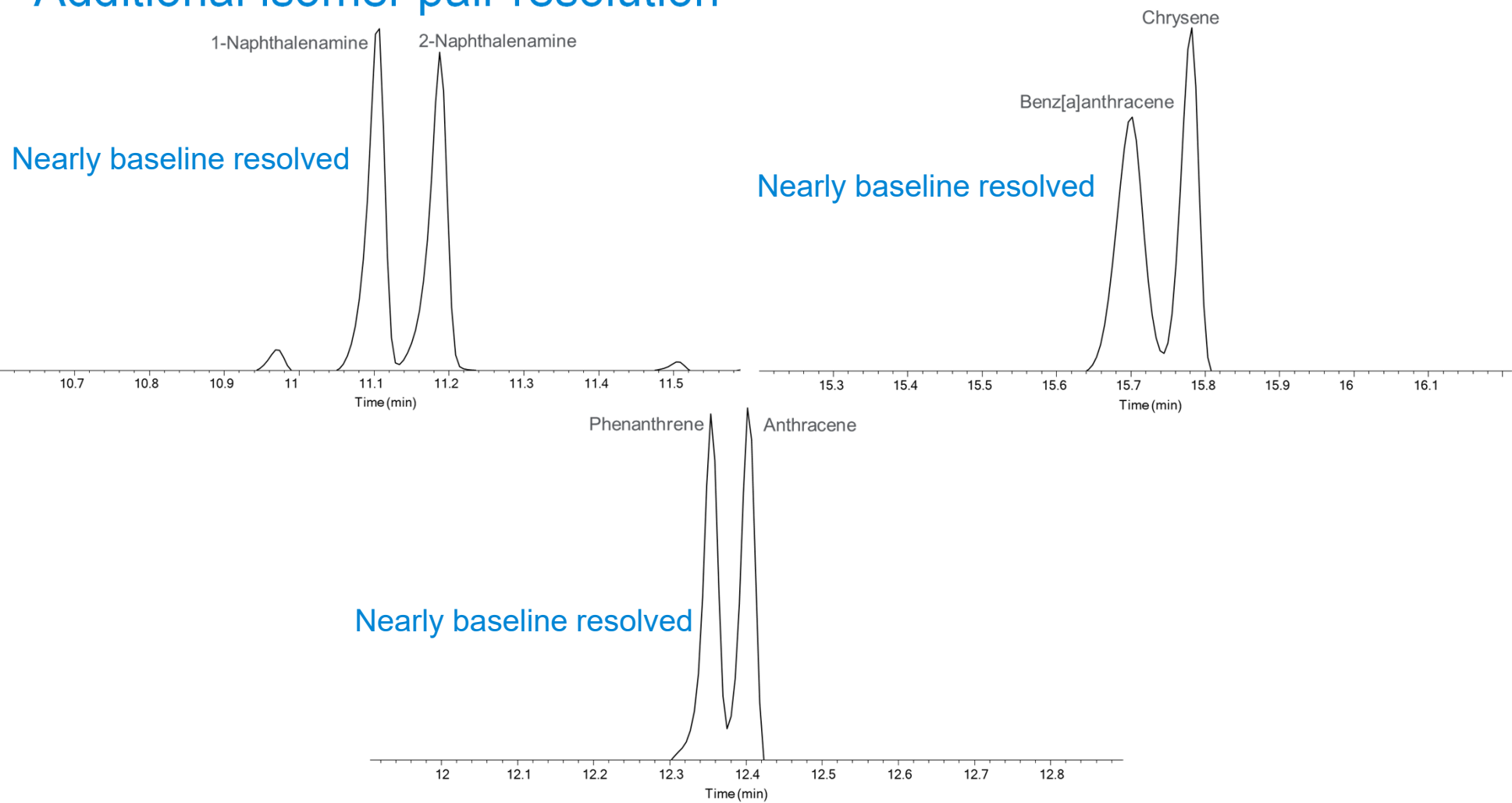


8270 Qualification: Benzo(b)fluoranthene and benzo(k)fluoranthene separation

Isomer pair resolution >50% ✓




Additional isomer pair resolution



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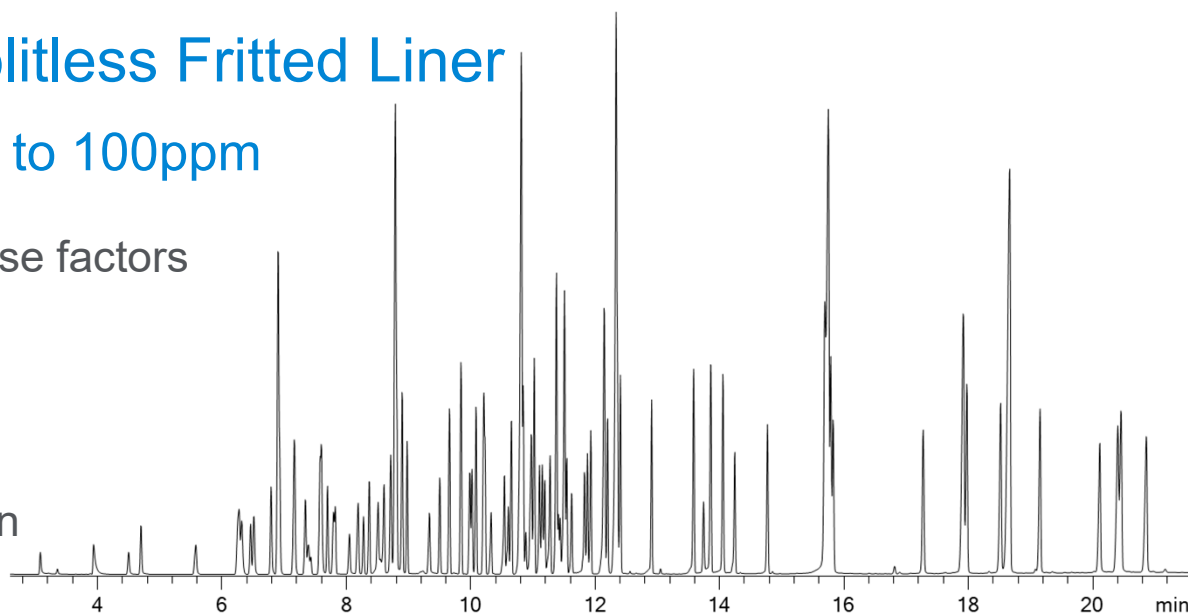
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Achieving Calibration with Splitless Fritted Liner

Calibration curve linearity from 0.1 to 100ppm

- Achieved linearity with average response factors (RFs) for 93 compounds
- Average RSD in RFs: 10.25 %
- 4 compounds required linear regression

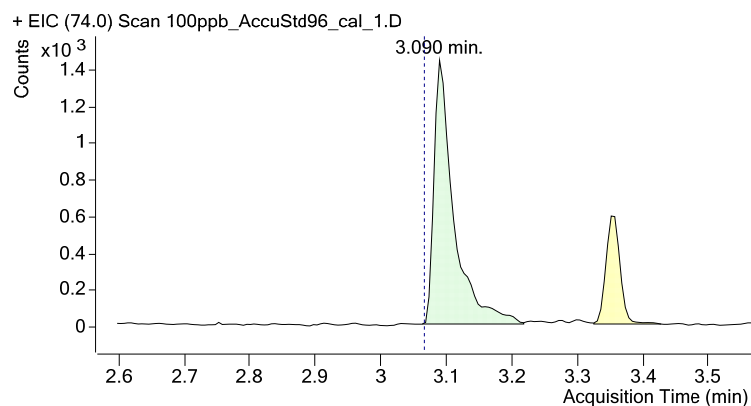


Full TIC of 10ppm target analytes and surrogates and 40ppm ISTDs

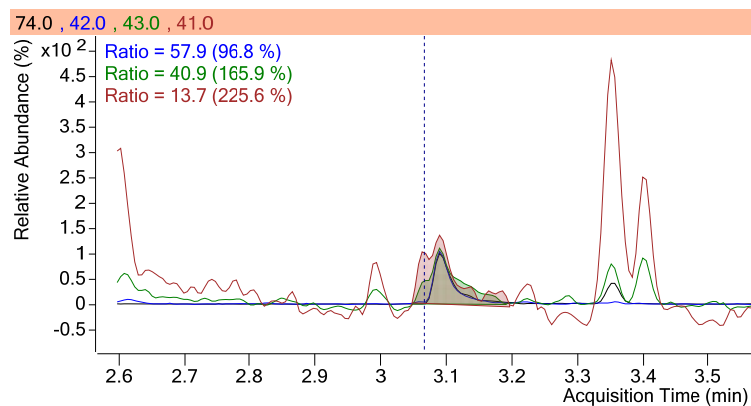
Compound No.	Compound	R ²	Calibration range (µg/mL)	Percent difference of lowest level standard (±30 % required)
27	Benzoic acid	0.9983	0.5 – 100	20.0
51	2,4-Dinitrophenol	0.9989	0.5 – 100	8.0
52	4-Nitrophenol	0.9958	0.2 – 100	-5.0
63	2-Methyl-4,6-dinitrophenol	0.9964	0.5 – 100	-14.0

Calibration Curve for N-nitrosodimethylamine (NDMA)

Quant ion: 0.1ppm



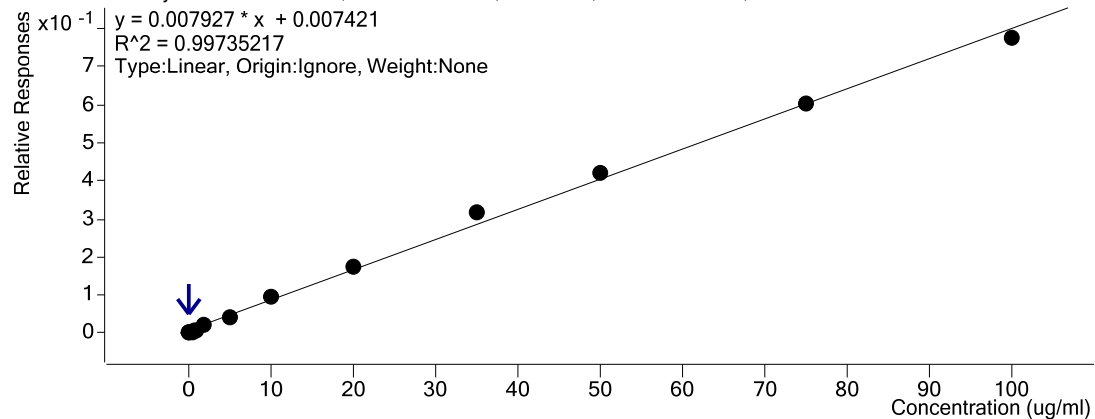
Qualifier ions: 0.1 ppm



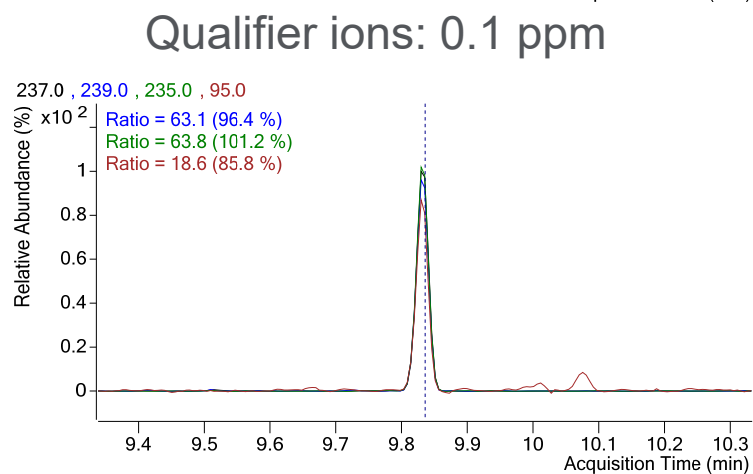
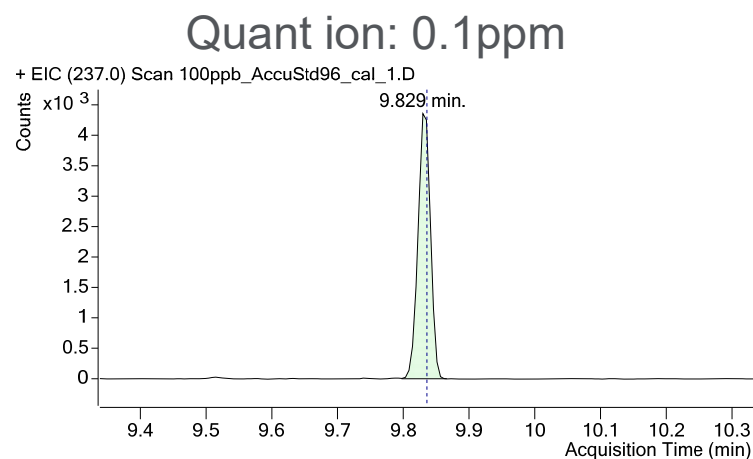
0.1 to 100 ppm

Avg RF %RSD = 7.41 %

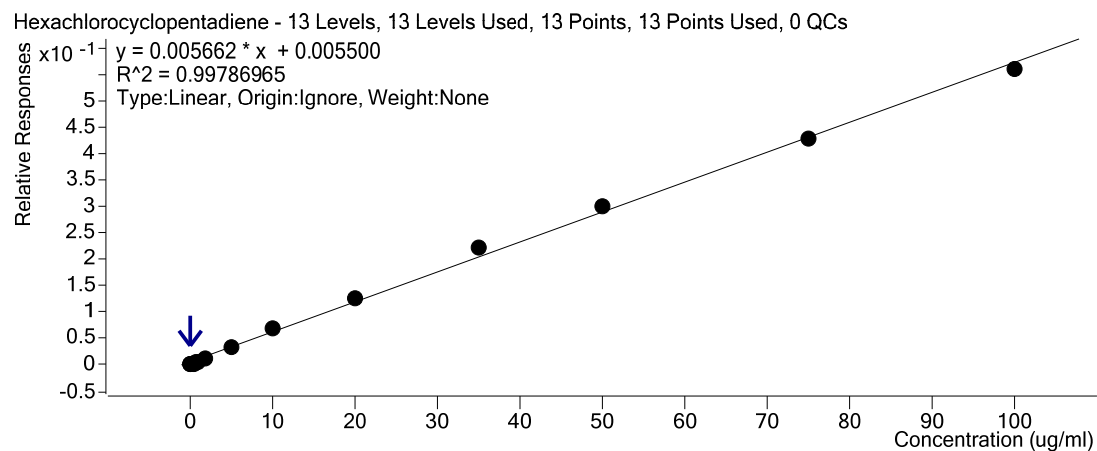
N-Nitrosodimethylamine - 13 Levels, 13 Levels Used, 13 Points, 13 Points Used, 0 QCs



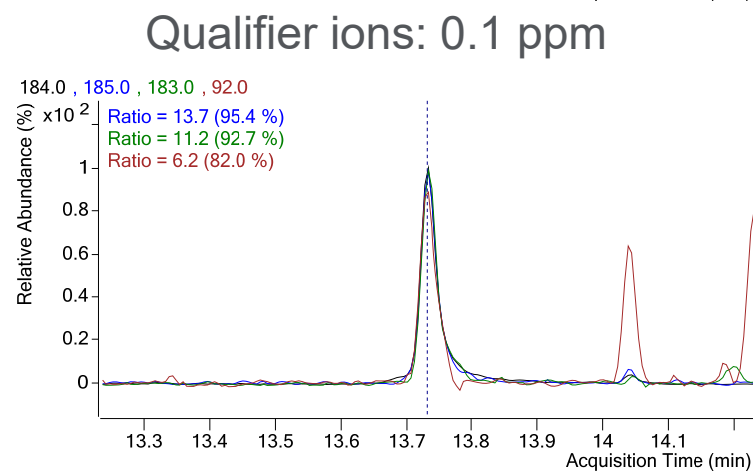
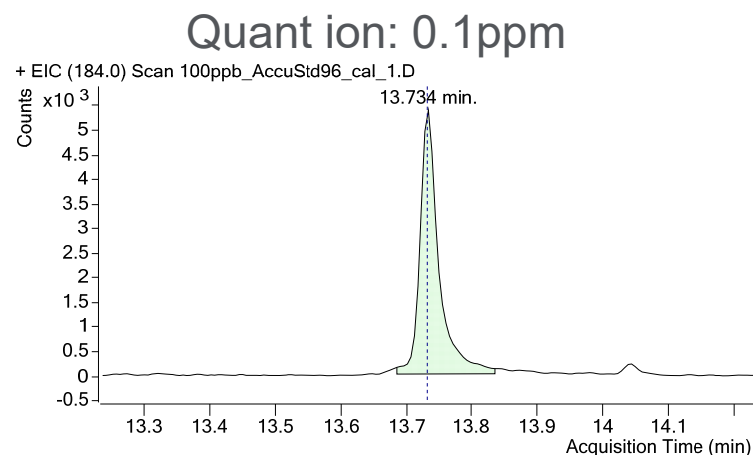
Calibration Curve for hexachlorocyclopentadiene



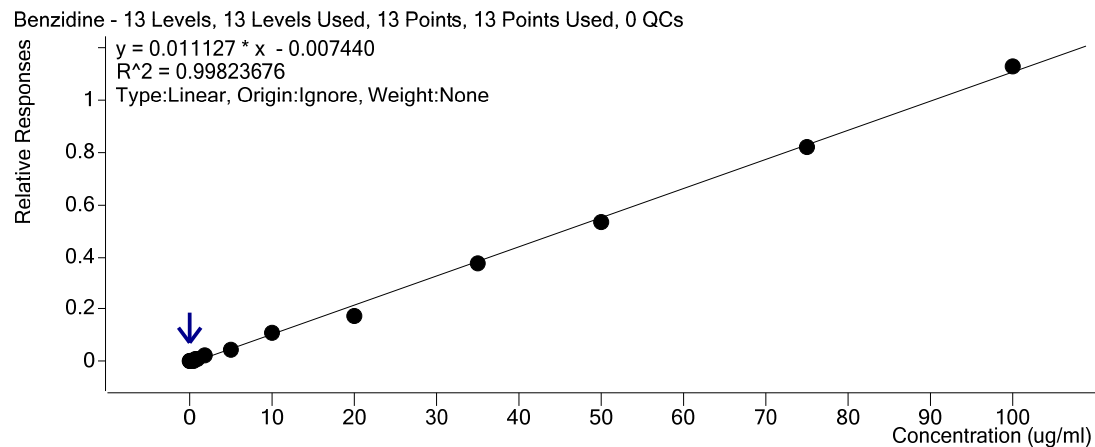
0.1 to 100 ppm
Avg RF %RSD = 8.21 %



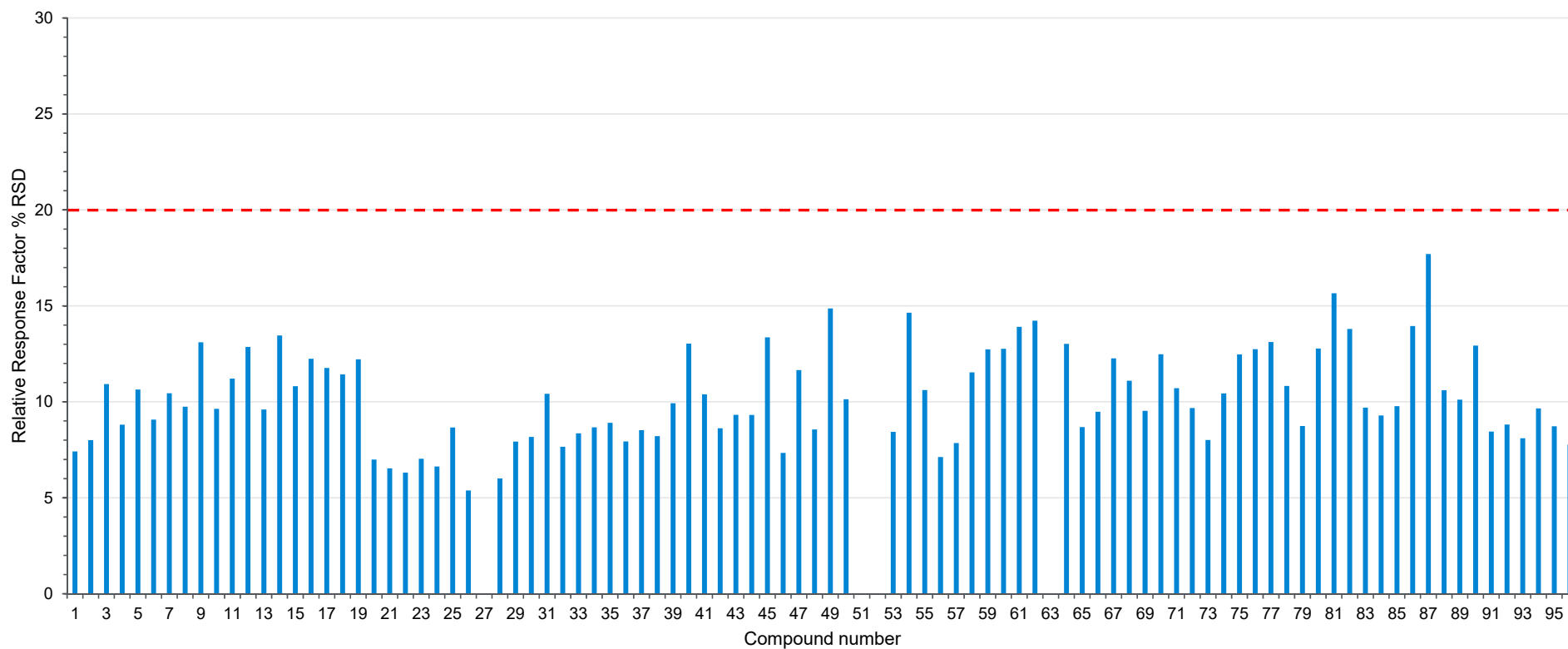
Calibration Curve for benzidine



0.1 to 100 ppm
Avg RF %RSD = 8.74 %





Calibration curve linearity from 0.1 to 100ppm: Achieved for 93 of 97 compounds with average response factors



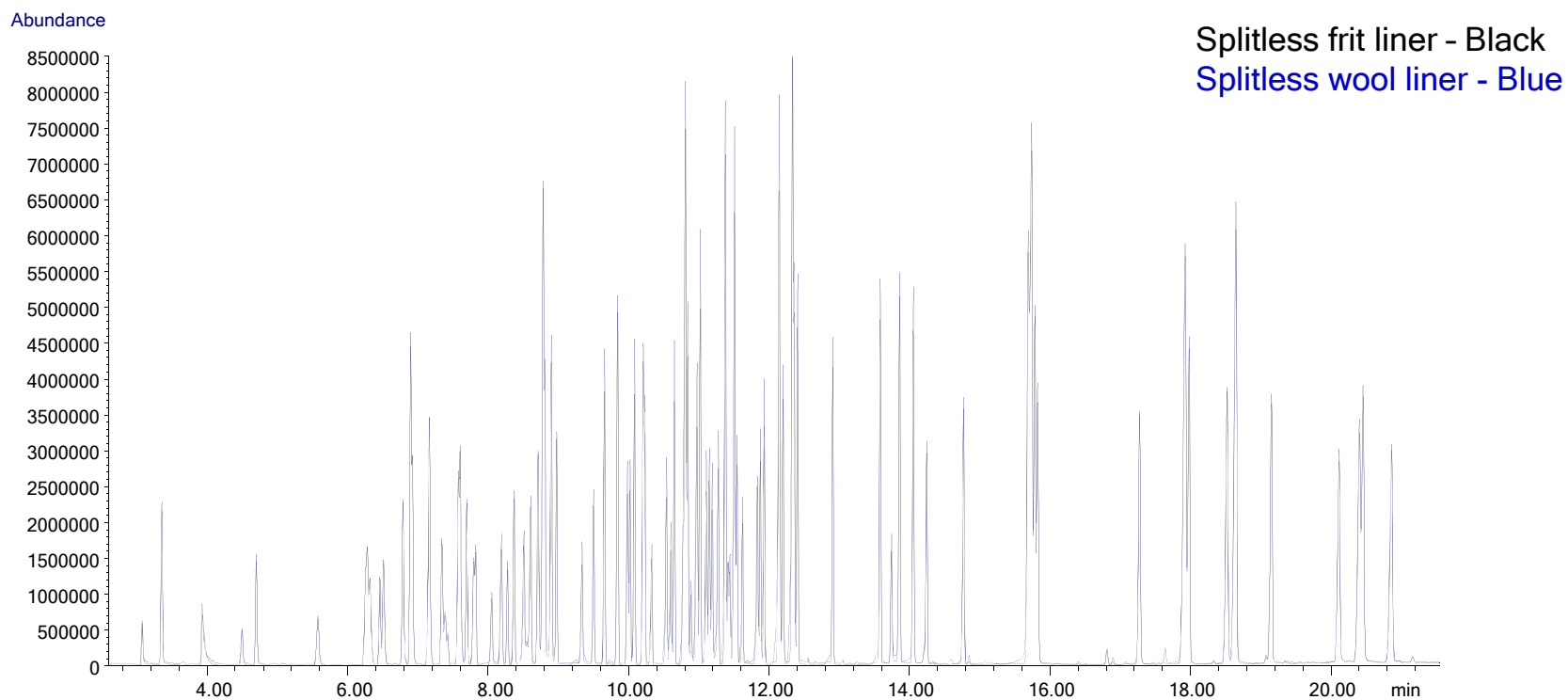
Comparison of percent RSDs of the target analytes for calibration ranges from 0.1 to 100 ng/μL

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Ability of new fritted liner to achieve 8270 requirements

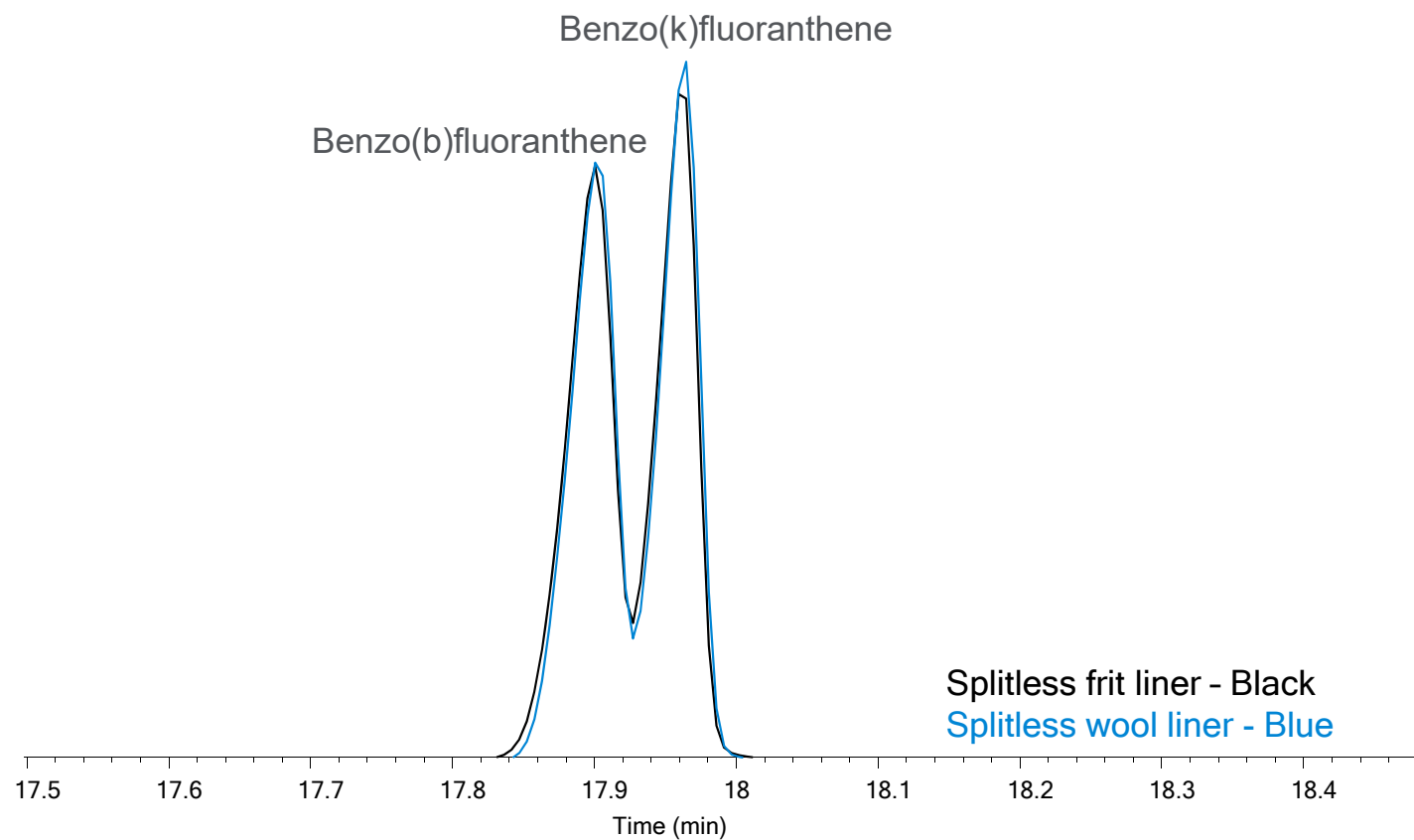
- Instrument Performance Checkout 
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 - Pass DDT breakdown percentage (<20% total)
 - Pentachlorophenol and benzdine tailing factors < 2.0
 - Isomer pair resolution
- Calibration curve: 100ppb to 100ppm 
 - Achieve linearity and verify responses $\pm 20\%$
 - Use average response factors, or linear regression fit, where necessary
- Ability to use fritted and wool liners with same calibration curve
- Lifetime comparison to wool liners
 - Continuing Calibration Verification: Cal drift within $\pm 20\%$ for >80%* compounds
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Overlay of splitless frit and wool liners



TIC overlays of 20ppm target analytes and surrogates and 40ppm ISTDs

Isomer Resolution: Compare Frit and Wool EIC Overlays



Lifetime testing: Soil matrix

Run	Sample type	Name
1	DCM blank	DCM blank
2	DFTPP tuning mix 25ppm	QC check
3	8270 short mix 20ppm	Mini-CCV
4	AccuStd 96 comp mix 20ppm	CCV
5-14	8x diluted soil matrix (10runs)	Soil matrix
15	DCM blank	DCM blank
16	DFTPP tuning mix 25ppm	QC check
17	8270 short mix 20ppm	Mini-CCV
18	AccuStd 96 comp mix 20ppm	CCV
19-28	8x diluted soil matrix (10runs)	Soil matrix
29	DCM blank	DCM blank
30	DFTPP tuning mix 25ppm	QC check
31	8270 short mix 20ppm	Mini-CCV
32	AccuStd 96 comp mix 20ppm	CCV

Criteria:

DDT % breakdown <20%

Pentachlorophenol, benzidine tailing factor <2.0

DFTPP Ion ratios pass

CCV: Cal drift within $\pm 20\%$ for >90%* compounds

ISTD: Area of IS peak area drift within $\pm 50\%$

Liner types

Agilent Ultra Inert frit

Splitless single taper with wool A

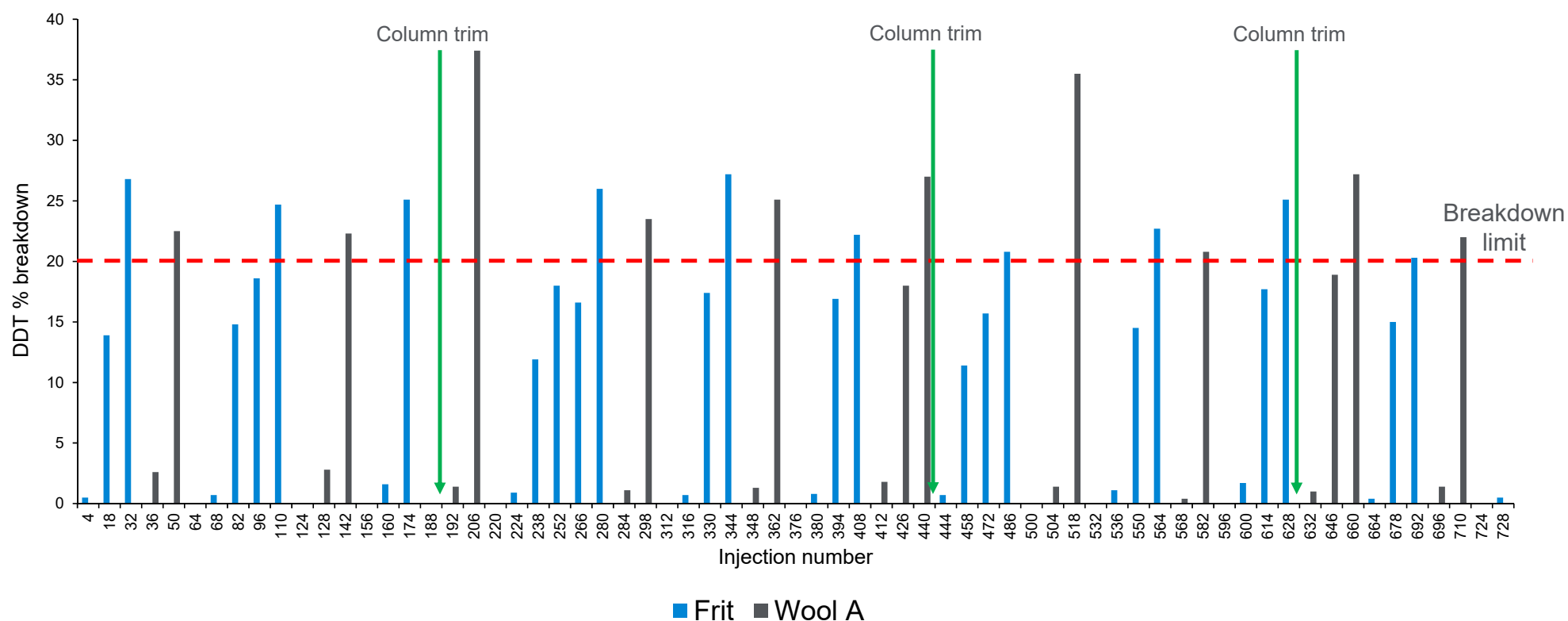
Splitless single taper with wool B

Splitless single taper with wool C

Lifetime Comparison: Frit vs. Wool A

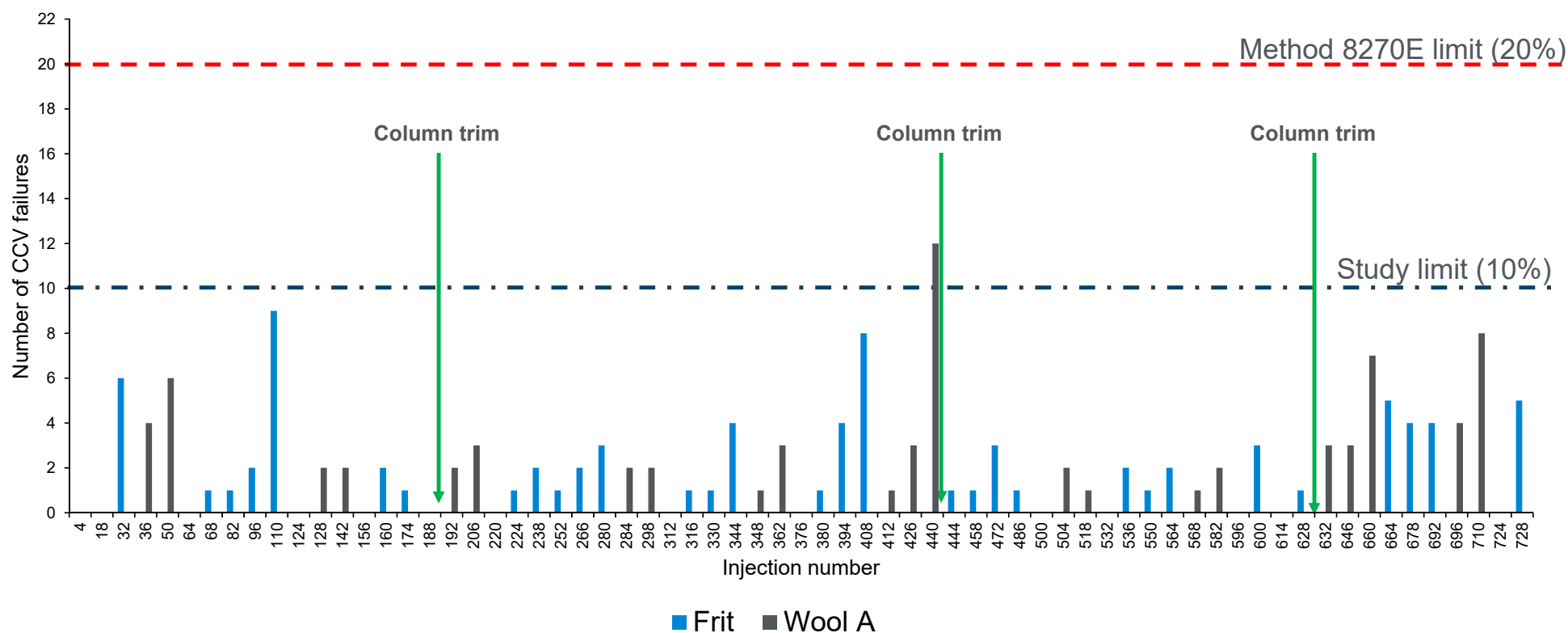
DDT % breakdown

Average Frit lifetime: 23 matrix injections
Average Wool A lifetime: 12 matrix injections



Lifetime Comparison: Frit vs. Wool A

CCV compound failure rate



Soil matrix lifetime testing: Frit vs. wool liners

Liner type	Average initial DDT % breakdown (QC1)	Average lifetime (number of matrix injections)	Average final DDT % breakdown
Agilent Ultra Inert frit	0.91 %	23 matrix injections	24.1 %
Splitless single taper with wool A	1.56 %	12 matrix injections	26.4 %
Splitless single taper with wool B	1.06 %	10 matrix injections	25.0 %
Splitless single taper with wool C	1.02 %	10 matrix injections	30.1 %

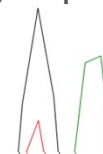


Peak recovery after liner replacement

2,4-dinitrophenol



hexachlorocyclopentadiene

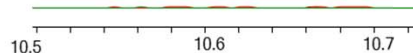


hexachlorocyclopentadiene

Liner 1: Run 1

Liner 1: After 20 matrix

Liner 2: New liner



Liner 1: Run 1

Liner 1: After 20 matrix injections

Liner 2: New liner



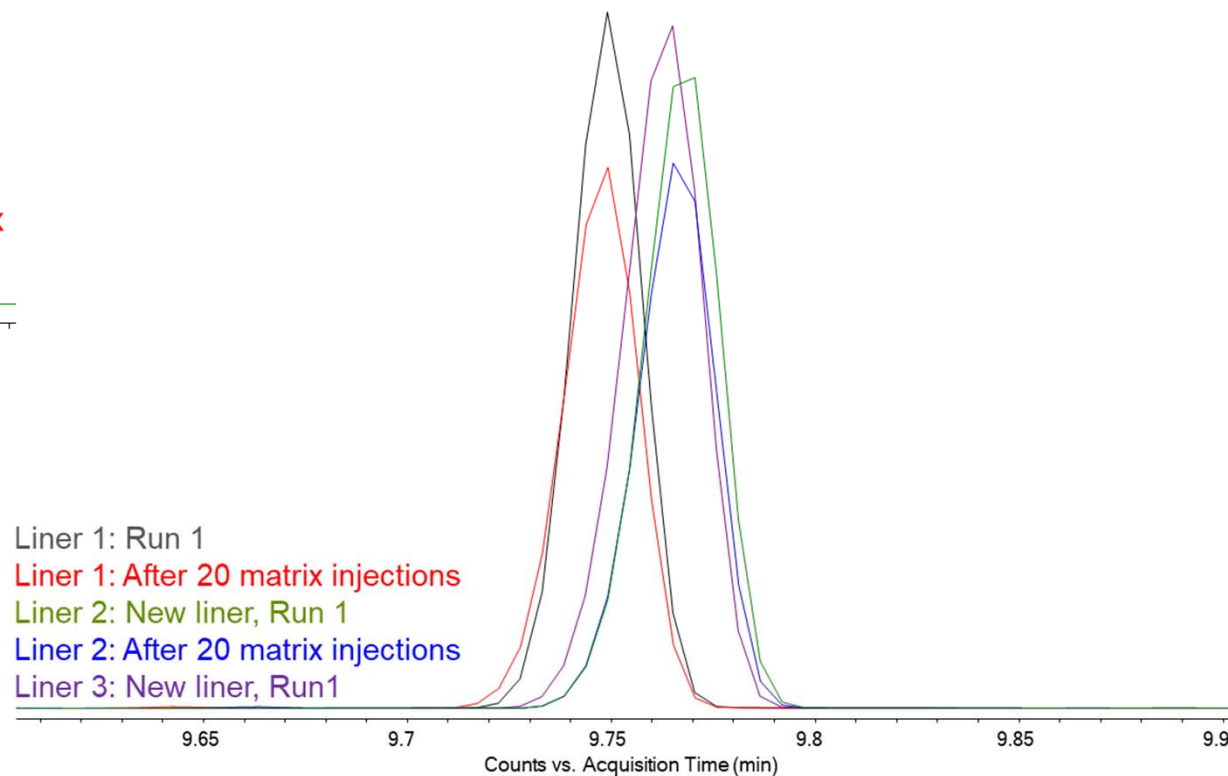
Liner 1: Run 1

Liner 1: After 20 matrix injections

Liner 2: New liner, Run 1

Liner 2: After 20 matrix injections

Liner 3: New liner, Run1



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- Lifetime comparison to wool liners ✓
 - Splitless fritted liners have ~2x longer lifetime than splitless wool liners

Thank you for your attention!

