Reduced EI Source Contamination and Improved System Longevity with the Intuvo 9000 GC for EPA 8270 Environmental Analysis

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Agilent
Trusted Answers

How is the Intuvo Different?
A whole new way to GC

Disposable
Guard chip

Modular
Intuvo flow chips

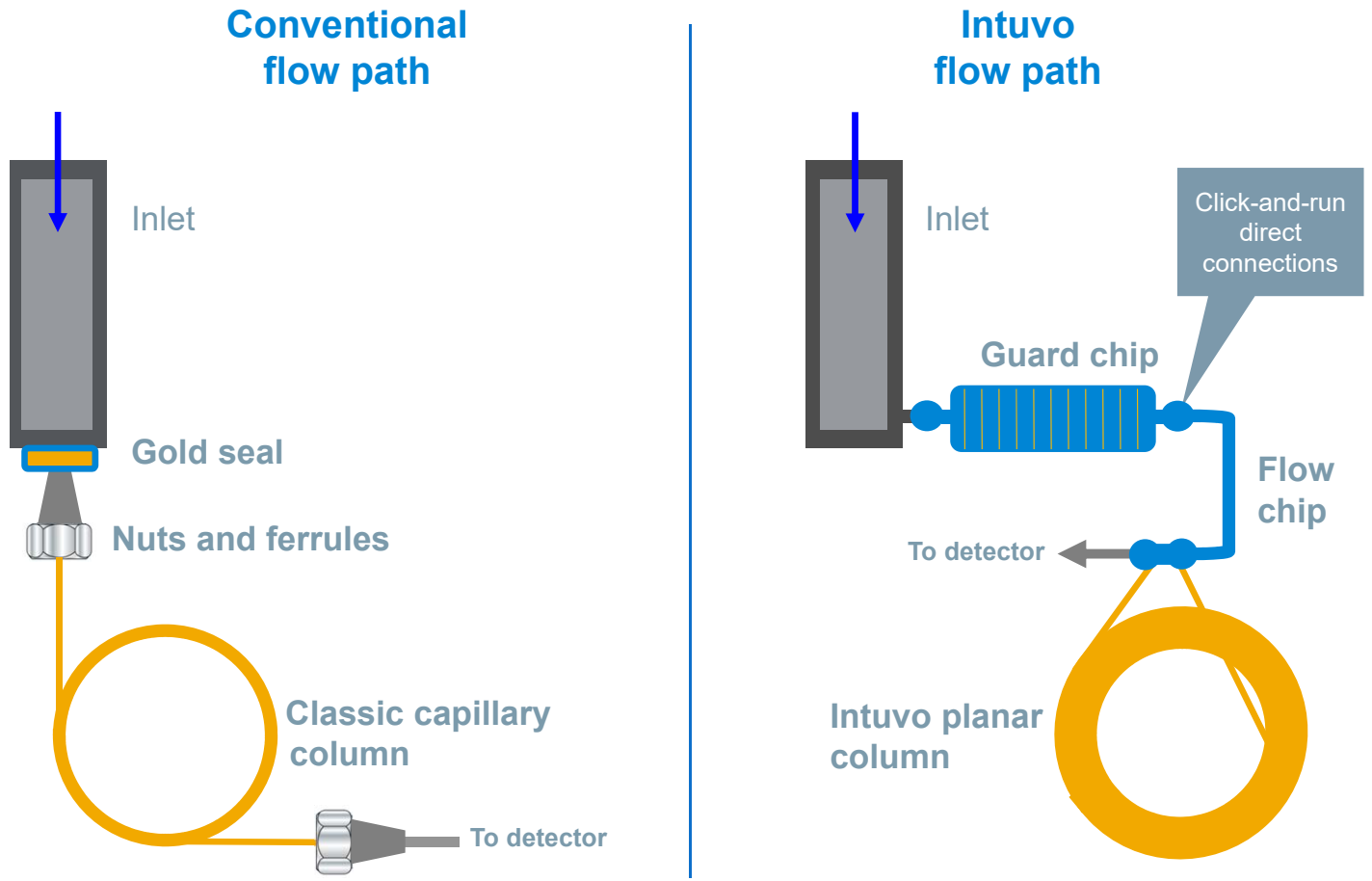
Ferrule-free click-and-run
connections

No-trim
column

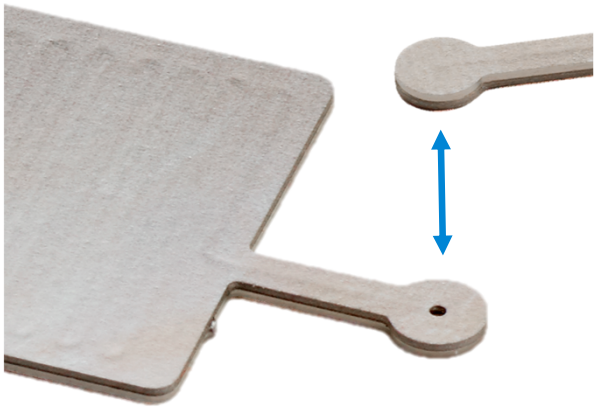
Direct
heating

Smart Key

Innovating the GC Flow Path



Click-and-Run Direct Connections
Eliminating connection uncertainty



- No more ferrules
- Direct face seal connections
- Audible and tactile click lets you know connection is made

- Easier to train
- Less unplanned downtime
- Fewer batch reruns and precious samples lost

Why are Semivolatile Organic Compounds Important?

Certain semivolatile organic compounds (SVOCs) are considered environmental contaminants.

Many world regulatory agencies have established methods and set performance criteria for measurement of SVOCs in a variety of matrices.

For example, United States Environmental Protection Agency (USEPA) method 8270D contains a list of 243 compounds that are suitable for analysis by GC/MS in solid waste, soil, air and water extracts.

In practice, the analysis method, compounds analyzed and calibration range can vary quite a bit from lab to lab.

Why Intuvo for SVOCs?

Typically, a high-volume analysis for an environmental testing lab.

Some difficult matrices with active compounds require frequent maintenance. Intuvo chip technology makes maintenance easy.

Small footprint, low power requirements make Intuvo attractive for a contract lab.

EPA 8270D Requirements

- **Performance based method**

- **Performance test**
  - Initial runs to validate mass spec tuning, system activity and system reactivity (i.e. *tuning standard*).
  - Resolution of isomers

- **Initial calibration**
  - Average response factor, curve fitting, curve weighting.
  - Validation of calibration required.

- **Periodic checks (~12 hours)**
  - Check system performance
  - Check calibration
  - Check ISTD response, retention time shift

Selection of Analytes

8270 Calibration Standard – 77 Acids/Bases/Neutrals (PAHs), 6 ISTDs

Compound Number	Compound
1	N-Nitrosodimethylamine
2	Pyridine
3	2-Fluorophenol (surrogate)
4	Phenol-d_5 (surrogate)
5	Phenol
6	Aniline
7	Bis(2-chloroethyl) ether
8	2-Chlorophenol
9	1,3-Dichlorobenzene
10	1,4-Dichlorobenzene
11	Benzyl alcohol
12	1,2-Dichlorobenzene
13	2-Methylphenol
14	Bis(2-chloroisopropyl) ether
15	4-Methylphenol
16	N-Nitrosodi-n-propylamine
17	Hexachloroethane
18	Nitrobenzene-d_5 (surrogate)
19	Nitrobenzene
20	Isophorone
21	2-Nitrophenol
22	2,4-Dimethylphenol
23	Benzoic acid
24	Bis(2-chloroethoxy)methane
25	2,4-Dichlorophenol
26	1,2,4-Trichlorobenzene
27	Naphthalene
28	4-Chloroaniline
29	Hexachlorobutadiene
30	4-Chloro-3-methylphenol
31	2-Methylnaphthalene
32	Hexachlorocyclopentadiene
33	2,4,6-Trichlorophenol
34	2,4,5-Trichlorophenol
35	2-Fluorobiphenyl (surrogate)

Compound Number	Compound
36	2-Chloronaphthalene
37	2-Nitroaniline
38	Dimethyl phthalate
39	2,6-Dinitrotoluene
40	Acenaphthylene
41	3-Nitroaniline
42	Acenaphthene
43	2,4-Dinitrophenol
44	4-Nitrophenol
45	2,4-Dinitrotoluene
46	Dibenzofuran
47	Diethyl phthalate
48	4-Chlorophenyl-phenyl ether
49	Fluorene
50	4-Nitroaniline
51	4,6-Dinitro-2-methylphenol
52	N-Nitrosodiphenylamine
53	Azobenzene
54	2,4,6-Tribromophenol (surrogate)
55	4-Bromophenyl phenyl ether
56	Hexachlorobenzene
57	Pentachlorophenol
58	Phenanthrene
59	Anthracene
60	Carbazole
61	Di-n-butylphthalate
62	Fluoranthene
63	Benzidine
64	Pyrene
65	p-Terphenyl-d_{14}
66	Butylbenzylphthalate
67	3,3'-Dichlorobenzidine
68	Benzo[a]anthracene
69	Bis(2-ethylhexyl)phthalate
70	Chrysene

Compound Number	Compound
71	Di-n-octyl phthalate
72	Benzo[b]fluoranthene
73	Benzo[k]fluoranthene
74	Benzo[a]pyrene
75	Indeno[1,2,3-cd]pyrene
76	Dibenzo[a,h]anthracene
77	Benzo[g,h,i]perylene
78	1,4-Dichlorobenzene-d_4 (internal standard)
79	Naphthalene-d_8 (internal standard)
80	Acenaphthalene-d_{10} (internal standard)
81	Phenanthrene-d_{10} (internal standard)
82	Chrysene-d_{12} (internal standard)
83	Perylene-d_{12} (internal standard)

GC Methods

GC (7890 & Intuvo)

Injection volume	1 µL
Inlet	Split/Splitless 300 °C Pulsed splitless 60 psi until 0.5 min Purge 50 mL/min at 0.5 min Septum purge switched flow mode 3mL/min
Liner	UI Splitless liner with a single taper and glass wool at the bottom
Guard chip (*Intuvo only*)	60 °C for 2 min, 20 °C/min to 260 °C, 6 °C/min to 330 °C hold
Column	Agilent DB-5ms UI 30 m x 0.25 mm x 0.5 µm
Flow	2 mL/min constant flow
Column temperature	40 °C for 2 min, 20 °C/min to 260 °C, 6 °C/min to 330 °C hold 1.333 min (standards) or 10.333 min (soil extract)

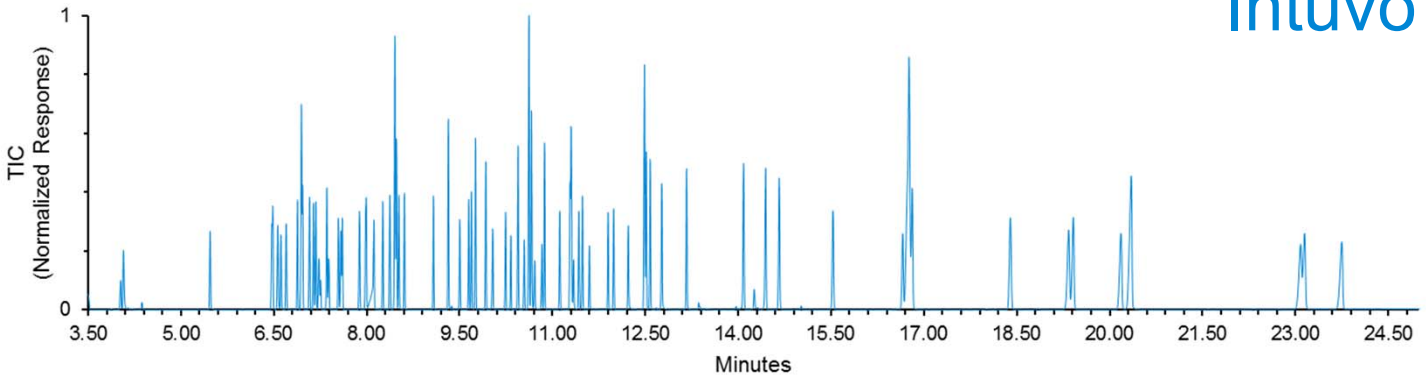
MSD (5977)

Transferline temperature	330 °C
Drawout plate	6 mm
Ion source temperature	330 °C
Quadrupole temperature	200 °C
Scan	35 to 550 m/z
Gain factor	1
Threshold	50
A/D samples	2

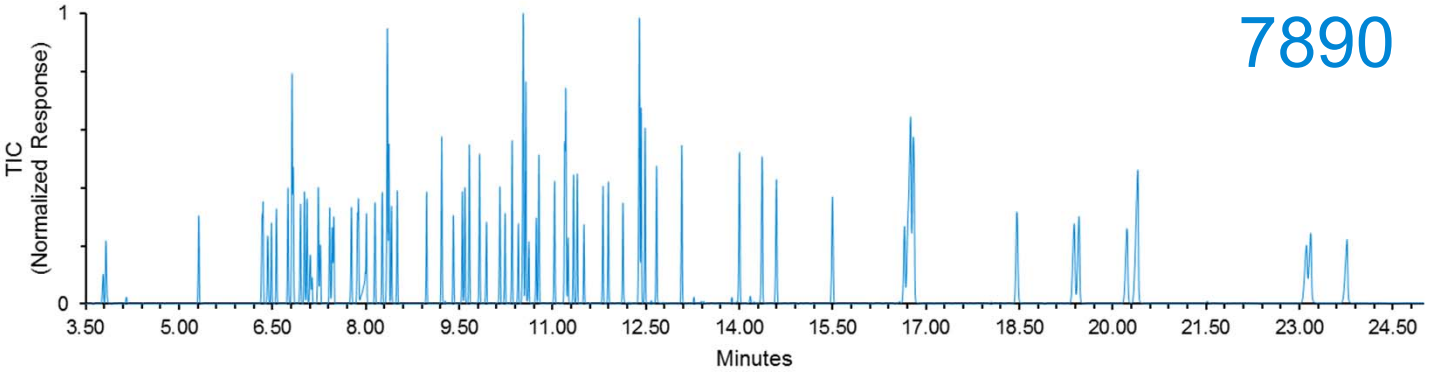
Chromatographic Comparison

8270 Calibration Standard – 77 Acids/Bases/Neutrals (PAHs), 6 ISTDs

Intuvo

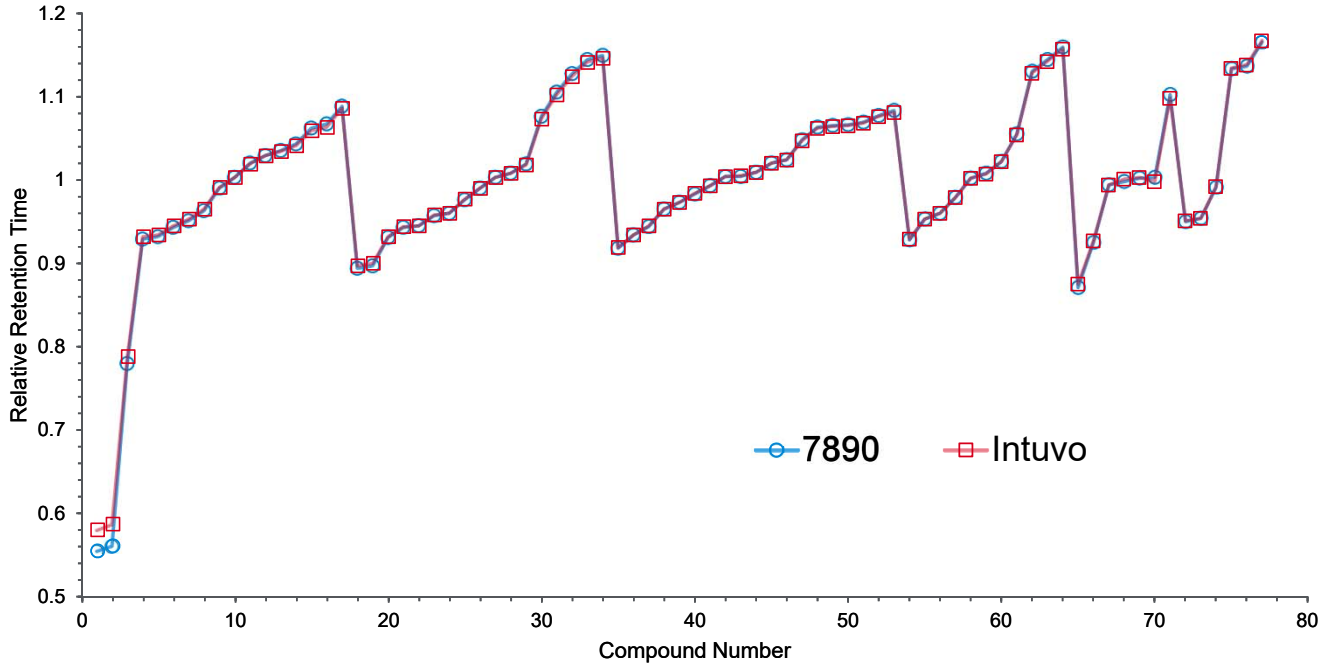


7890



Retention Time Comparison

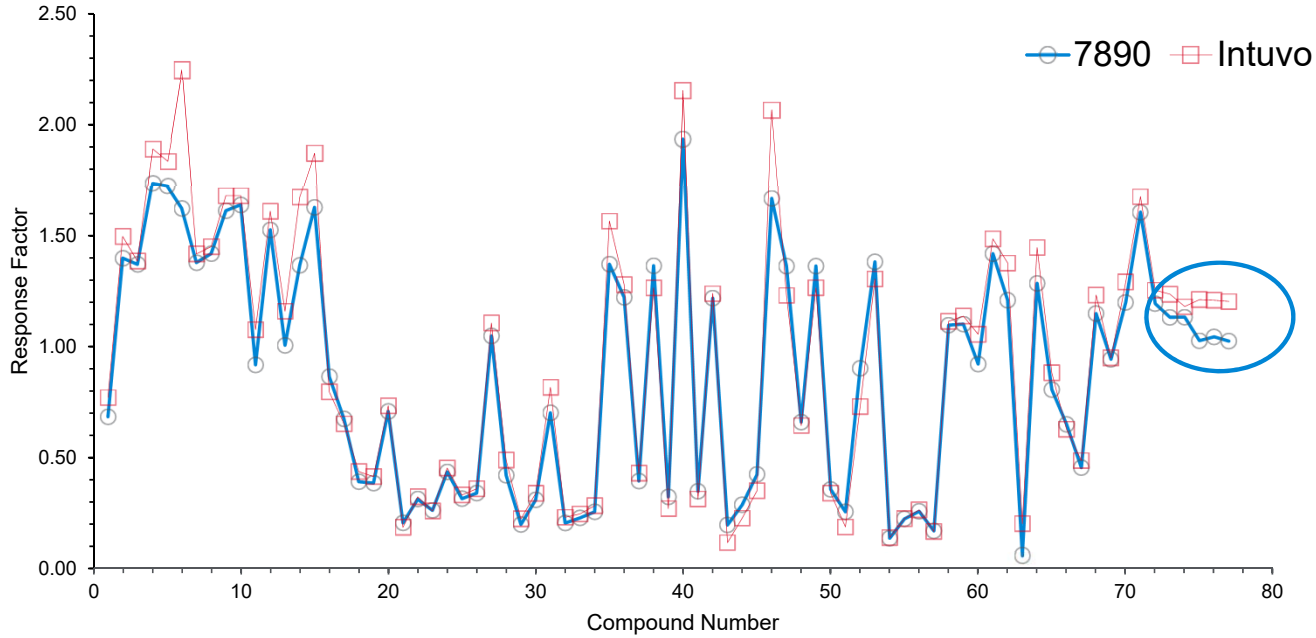
Relative Retention Time = RT_{target} / RT_{ISTD}



Average relative retention time difference = 0.0006

Response Comparison

$$Response\ Factor = (Area_{target} * Concentration_{ISTD})/(Area_{ISTD} * Concentration_{target})$$

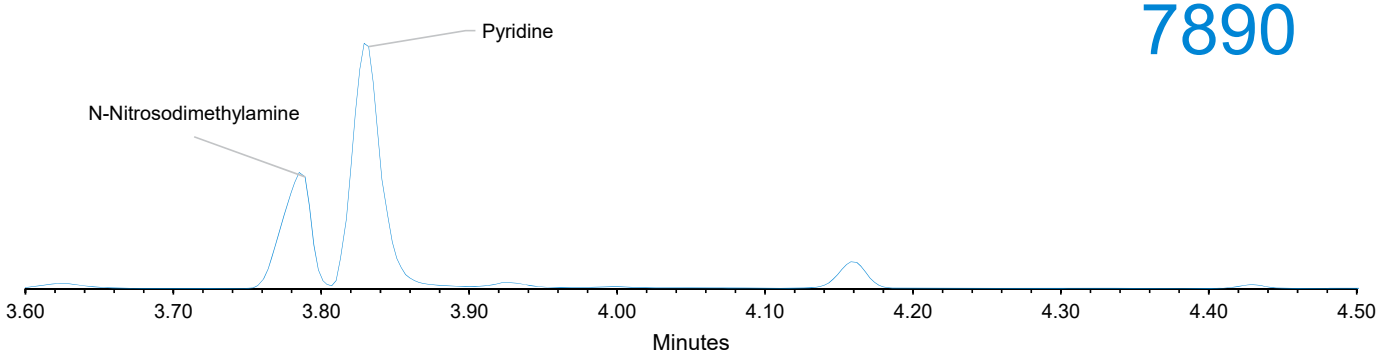


Average response factor difference = 4.6%

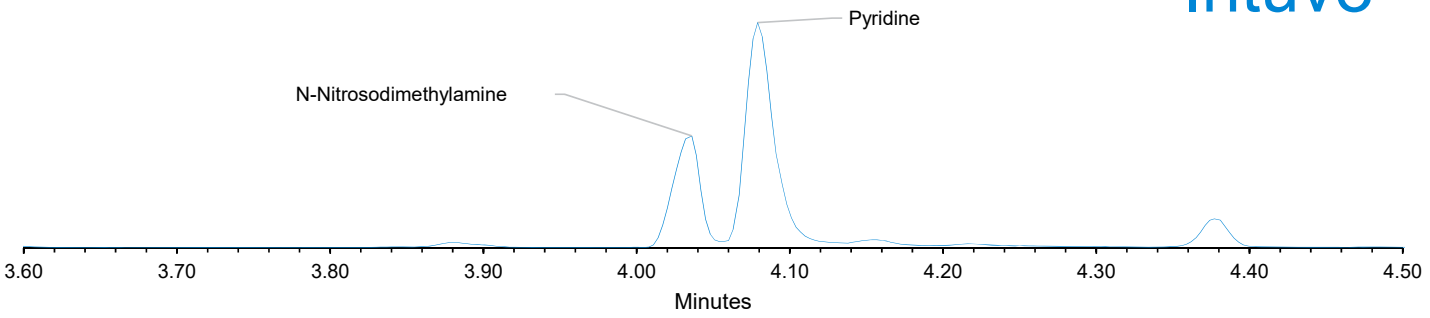
Resolution Comparison

First eluters

7890



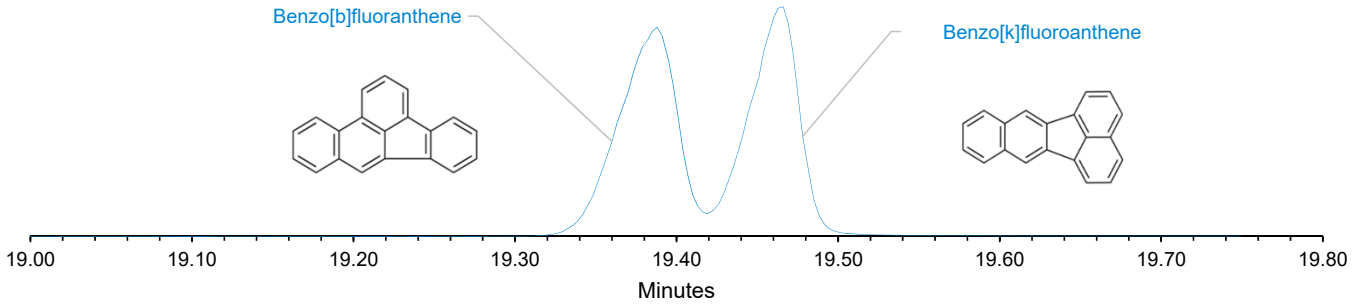
Intuvo



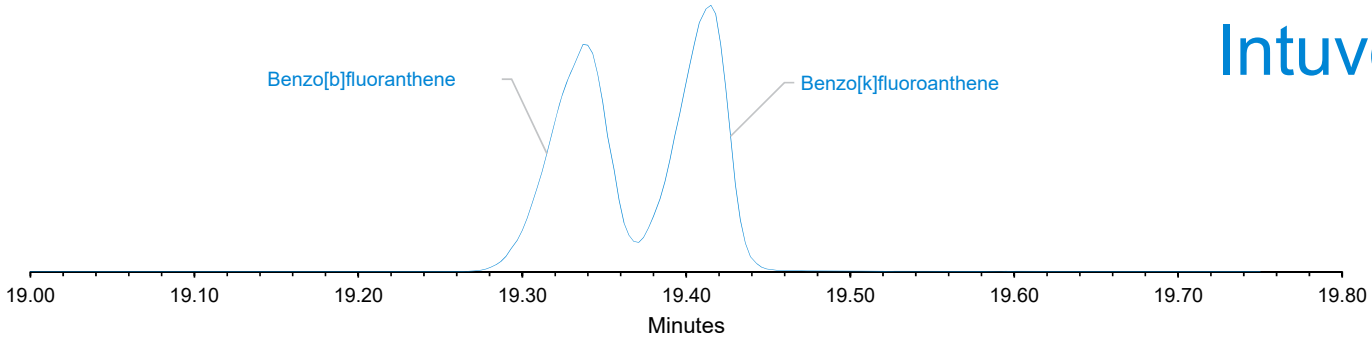
Resolution Comparison

Critical pair requirement – greater than 50% resolved

7890



Intuvo

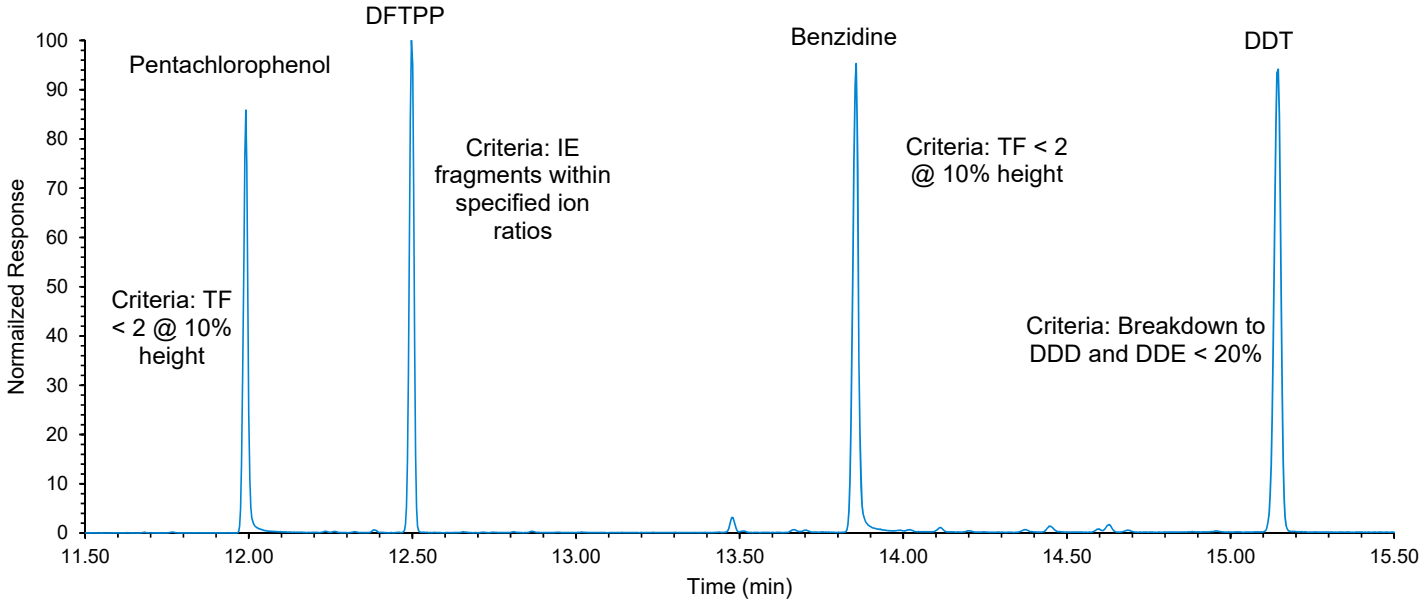


Benzo b & k 90% resolved

8270D Performance Check

8270D Performance Mixture

Test system acid/base activity, inertness and MSD tuning ratios (25 µg/mL)



8270D Performance Mixture Results

Test system acid/base activity, inertness and MSD tuning ratios

Acid/Base Activity

Compound Name	Expected RT	Observed RT	Tailing Factor	Pass/Fail
Pentachlorophenol	11.970	11.991	1.0	Pass
Benzidine	13.830	13.856	0.8	Pass

Inertness

Compound Name	Expected RT	Observed RT	TIC Area	Breakdown %	Pass/Fail
4,4'-DDT	15.146	15.146	7702869	1.4	Pass
4,4'-DDD	14.600	14.630	112457		
4,4'-DDE	15.100	0.000	0		

Tuning Ratios

Target Mass	Rel. To Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Pass/Fail
51	442	10	80	31.1	214582	Pass
68	69	0	2	0.0	0	Pass
70	69	0	2	0.4	990	Pass
127	442	10	80	39.4	271846	Pass
197	442	0	2	0.0	0	Pass
198	442	50	100	84.0	579656	Pass
199	198	5	9	6.1	35538	Pass
275	442	10	60	22.4	154842	Pass
365	198	1	100	4.2	24635	Pass
441	442	0	24	15.4	106169	Pass
442	442	100	100	100.0	689875	Pass
443	442	15	24	18.7	129287	Pass

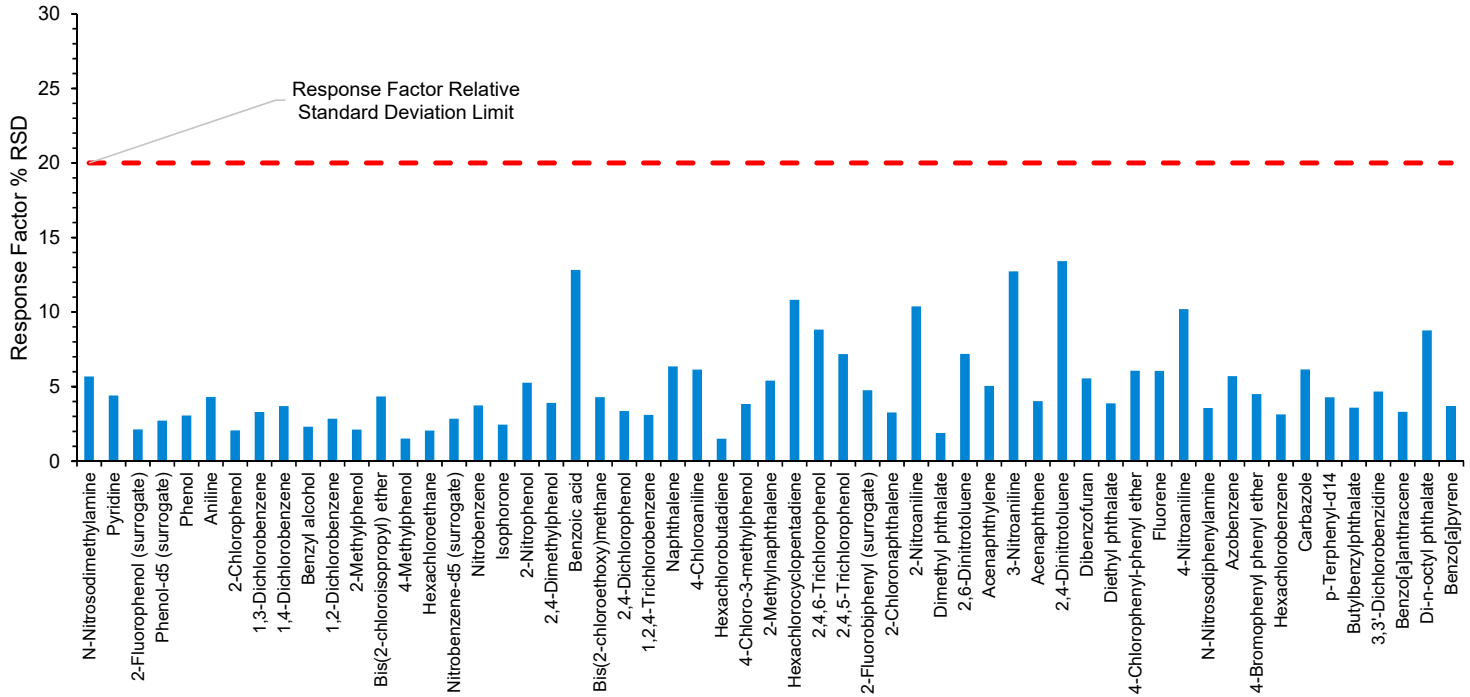
8270D Calibration Performance Specifications

Calibration - Group I

Calibration: Average response factor

Range: 0.1 to 100 µg/mL for 57 of the 77 compounds

Average RSD: 4.98%

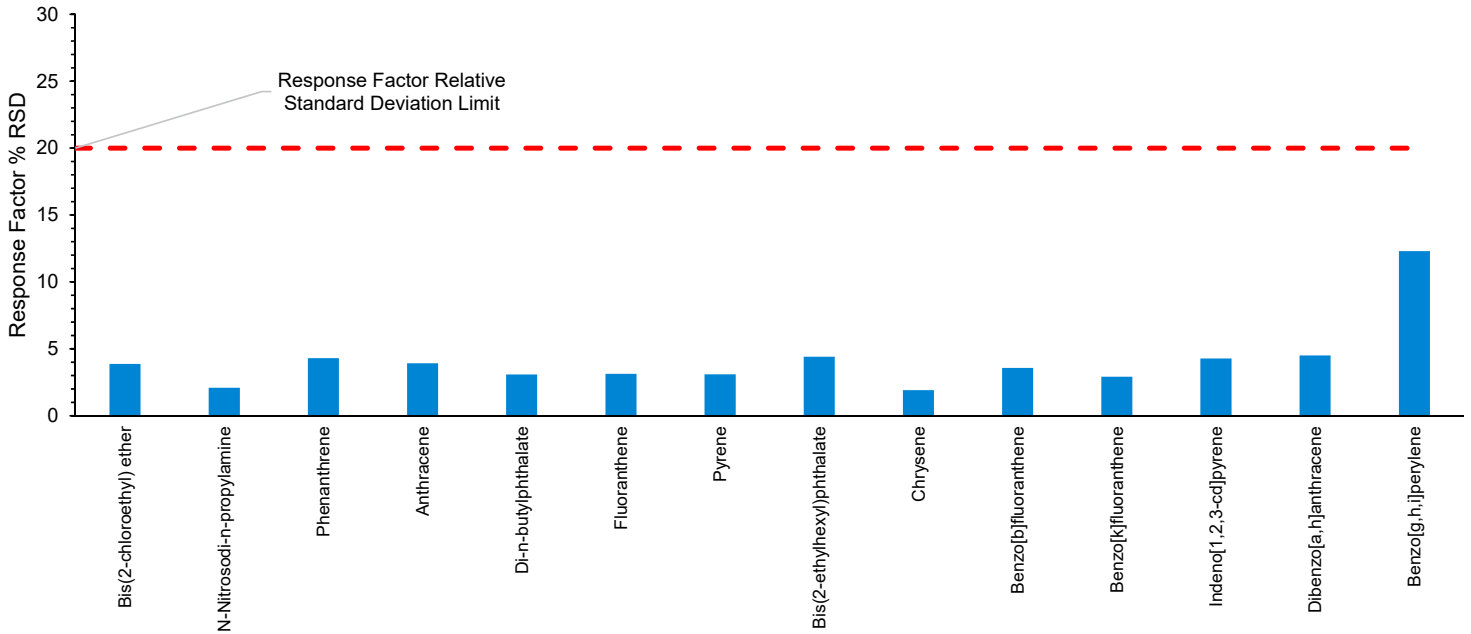


Calibration - Group II

Calibration: Average response factor

Range: 0.1 to 50 µg/mL for 14 of the 77 compounds

Average RSD: 4.10%



Calibration - Group III

Calibration: Weighted least squares calibration

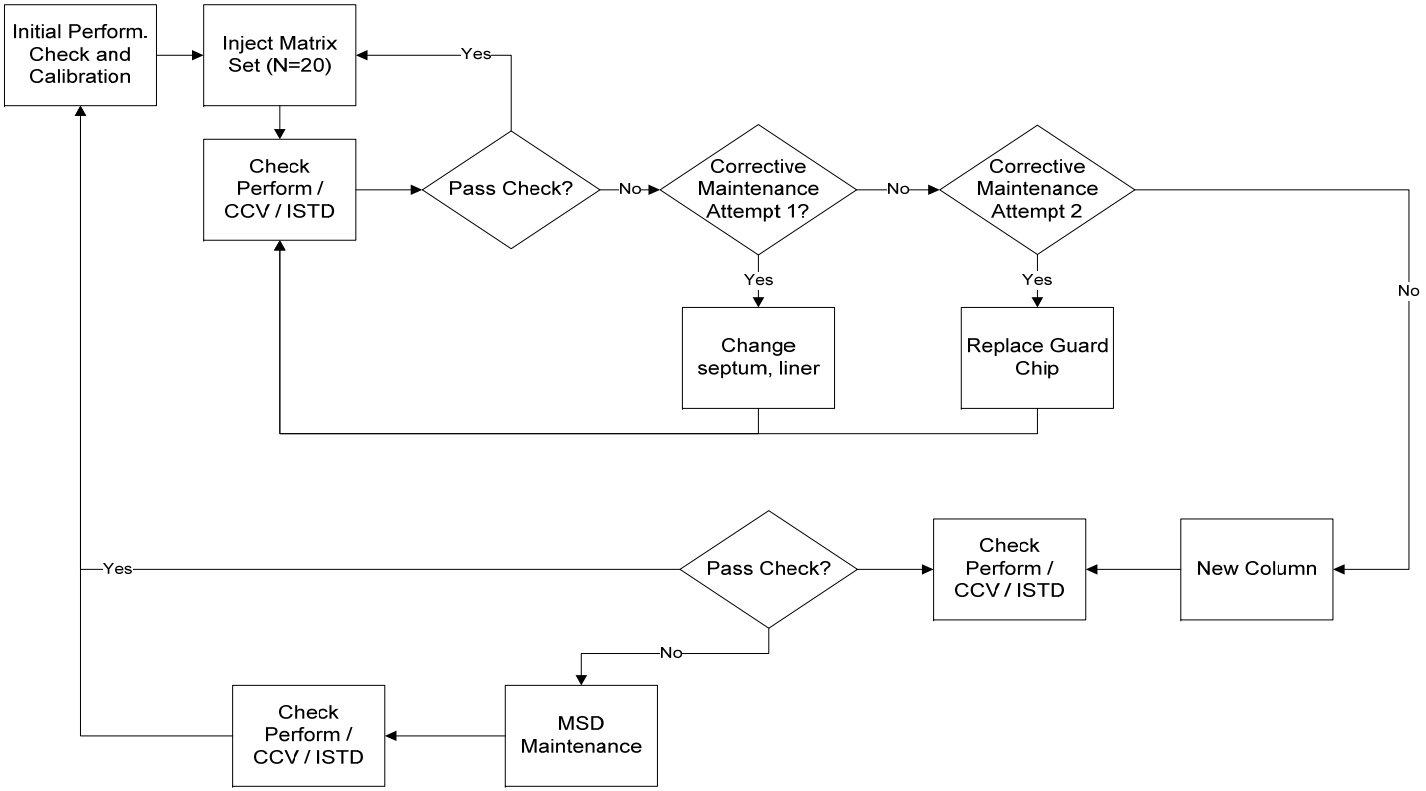
Range: Widest linear range while meeting minimum specification

Compound	R	Calibration Range (µg/mL)	Percent Difference of Lowest Level Standard (± 30% Required)
2,4-Dinitrophenol	0.9984	1.6 – 100	23.9
4-Nitrophenol	0.9994	0.8 - 100	22.9
4,6-Dinitro-2-methylphenol	0.9991	0.8 - 100	-1.3
2,4,6-Tribromophenol	0.9997	0.8 - 100	12.4
Pentachlorophenol	0.9992	0.8 - 100	23.4
Benzidine	0.9966	4 - 100	16.5

Matrix Study

Matrix Study

Decision Flow Diagram

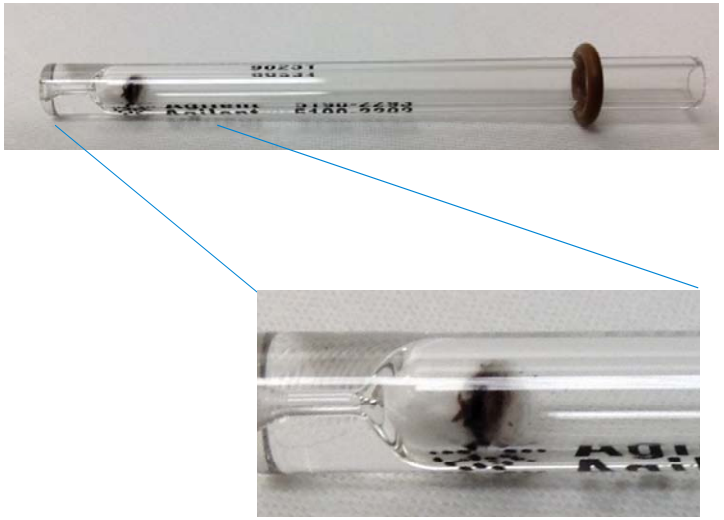


Matrix Extracts

Composite soil extracts in methylene chloride from Enviro Contract Lab in USA

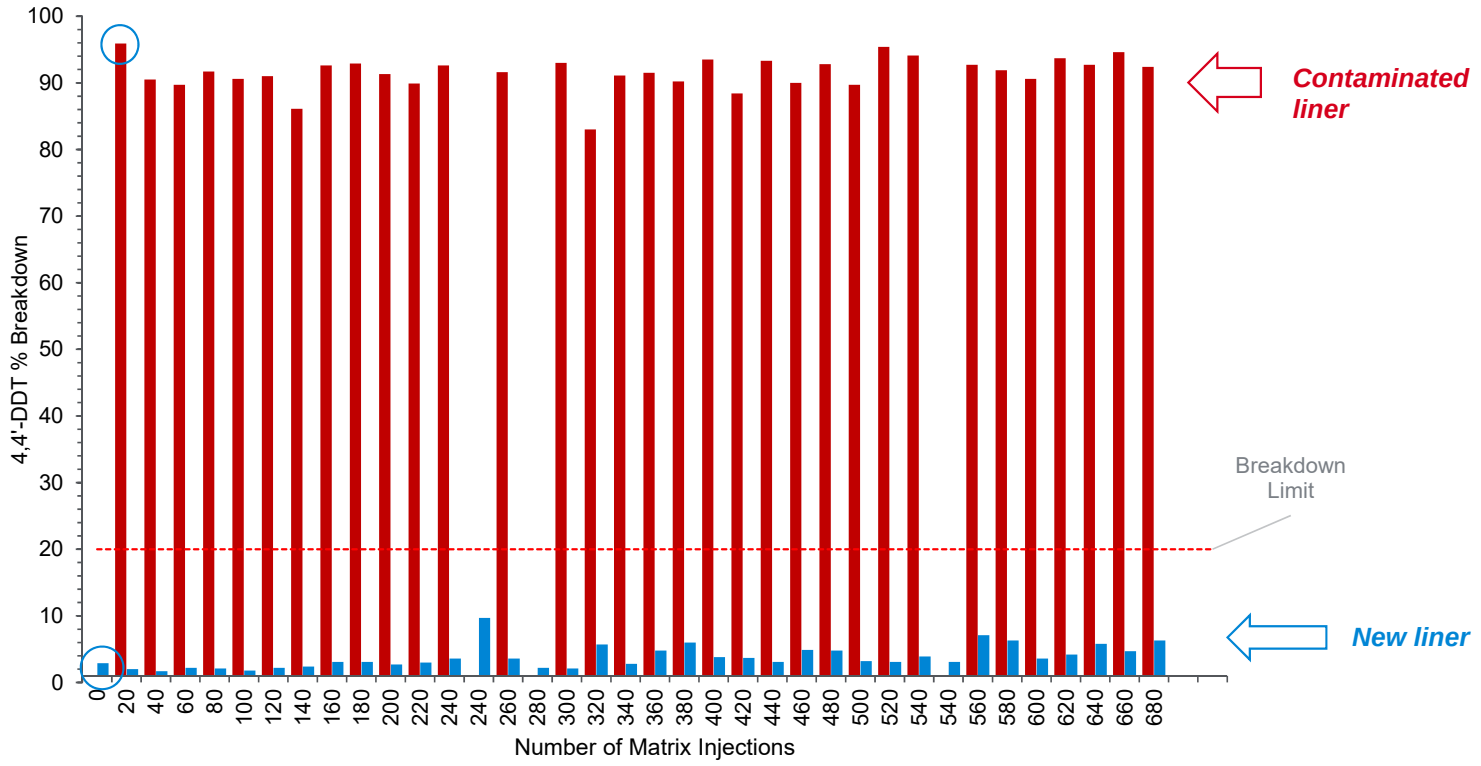
Typical of their "Worst Matrix"

20 sequential injections on a wool packed liner



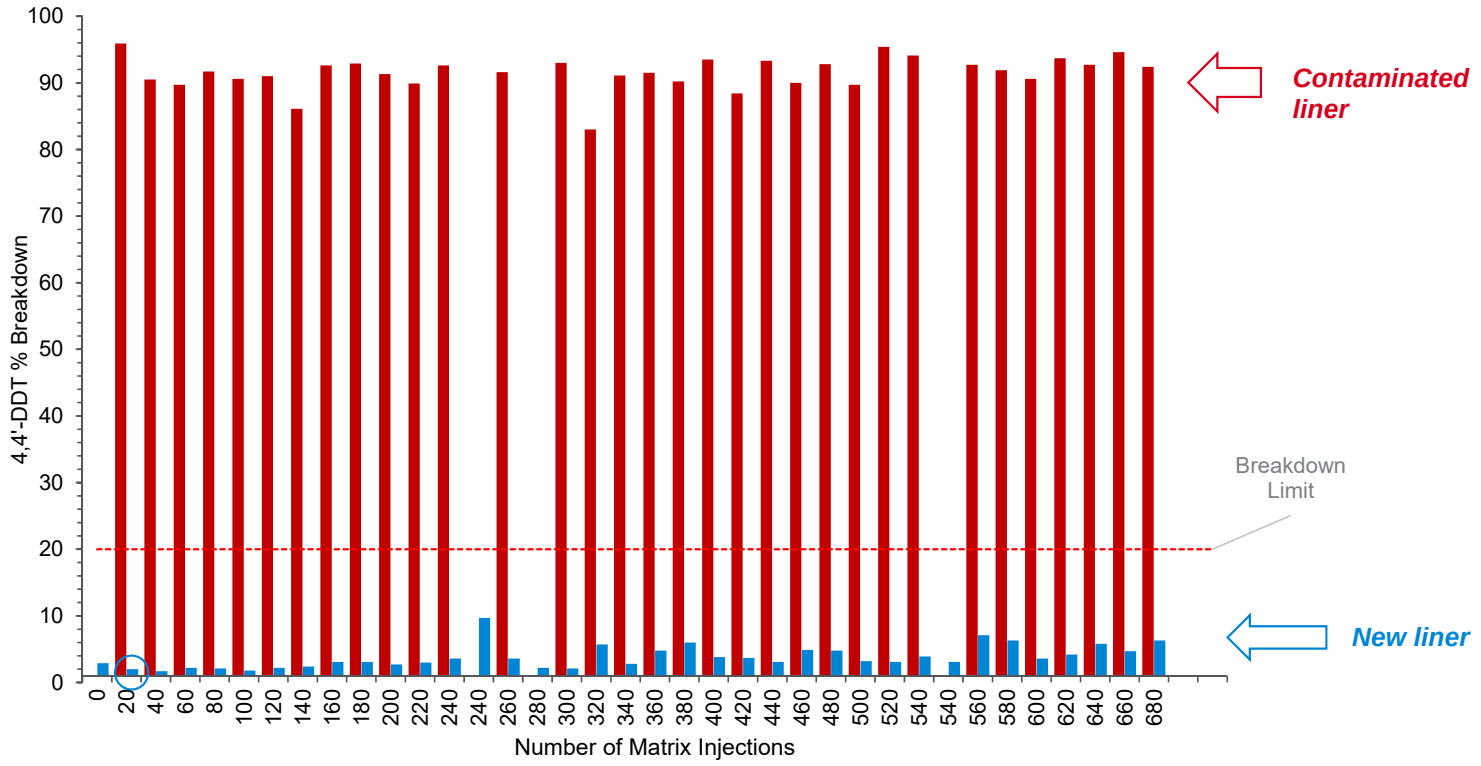
Matrix Study - Performance Check

DDT Breakdown



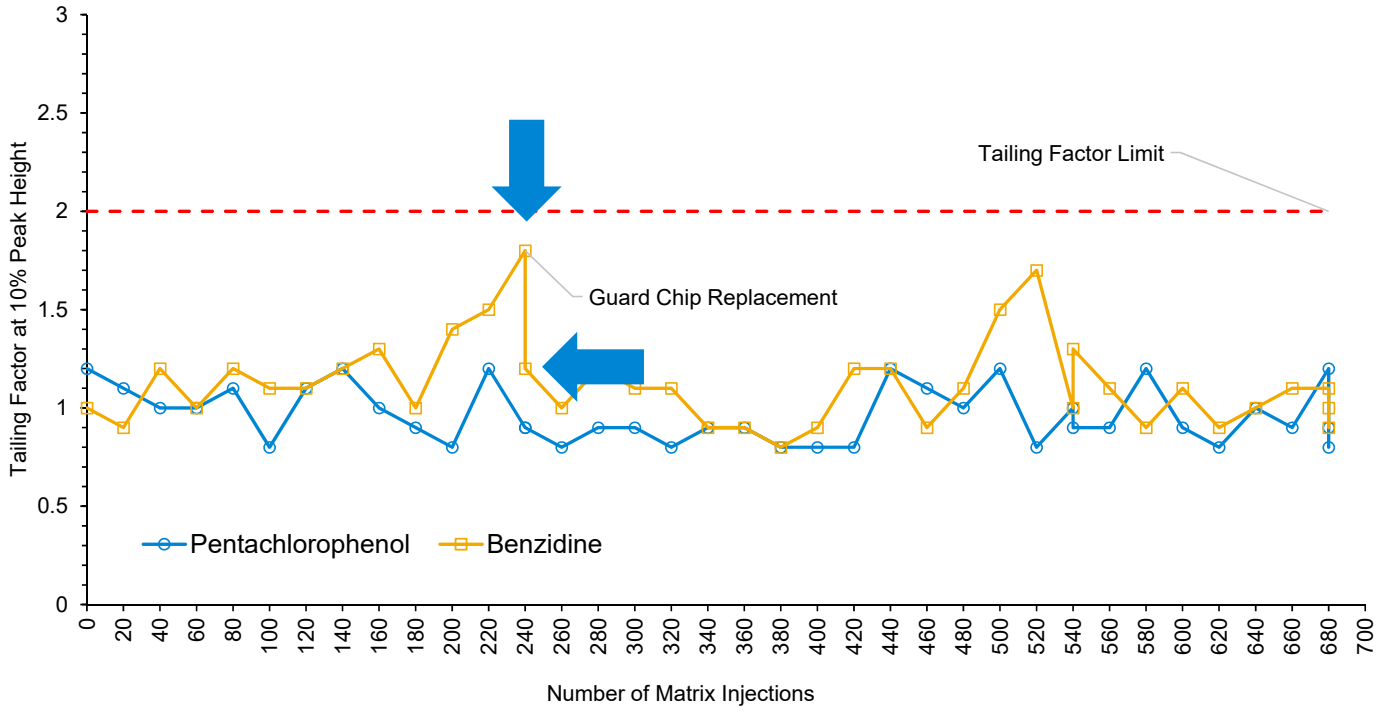
Matrix Study - Performance Check

DDT Breakdown



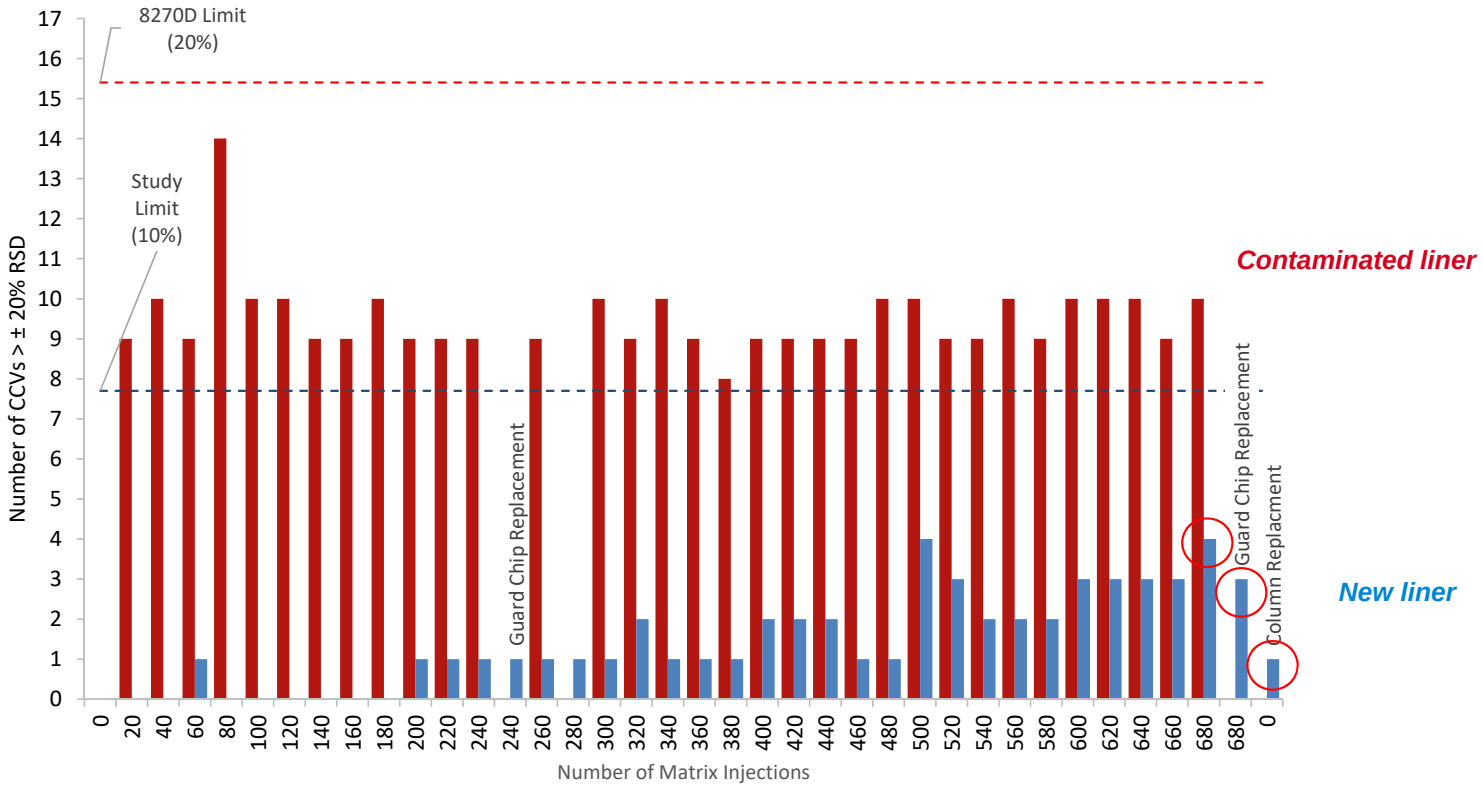
Matrix Study - Performance Check

Pentachlorophenol and Benzidine Tailing Factor – After Liner Change



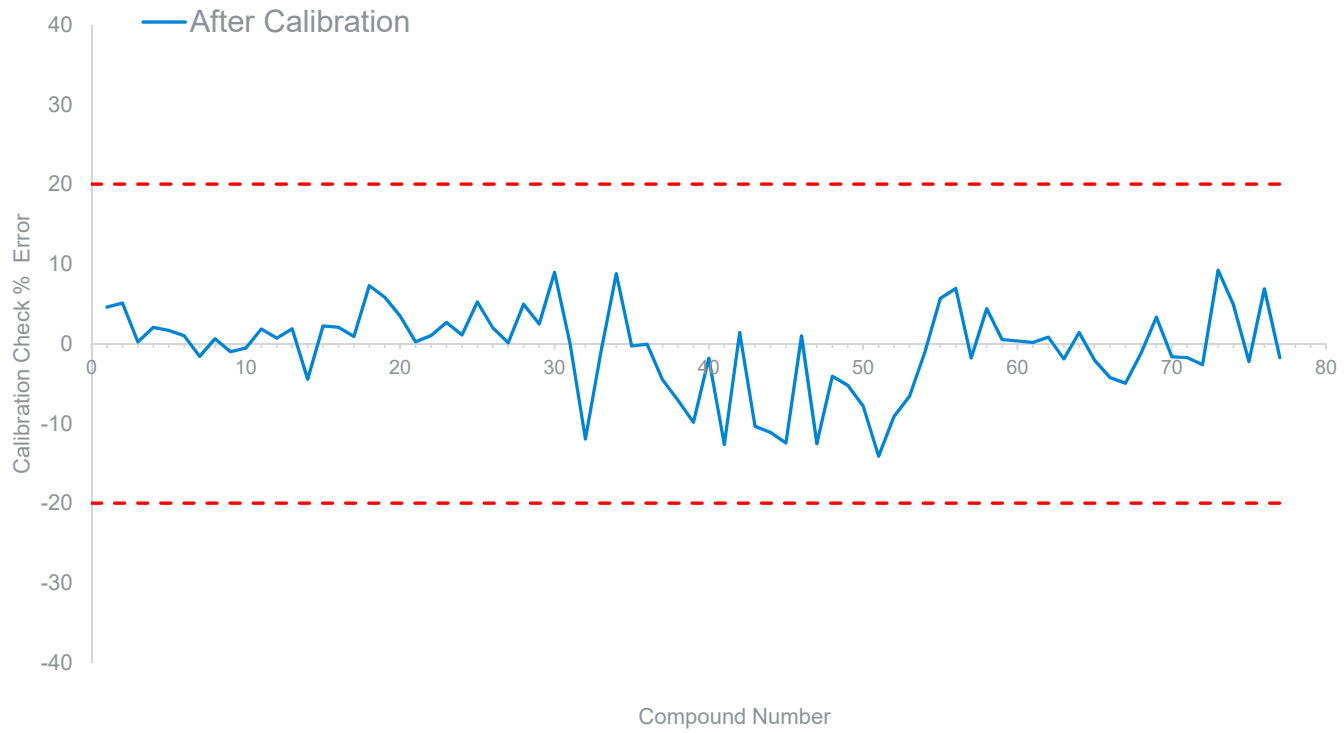
Matrix Study - Calibration

Continuing Calibration Verification



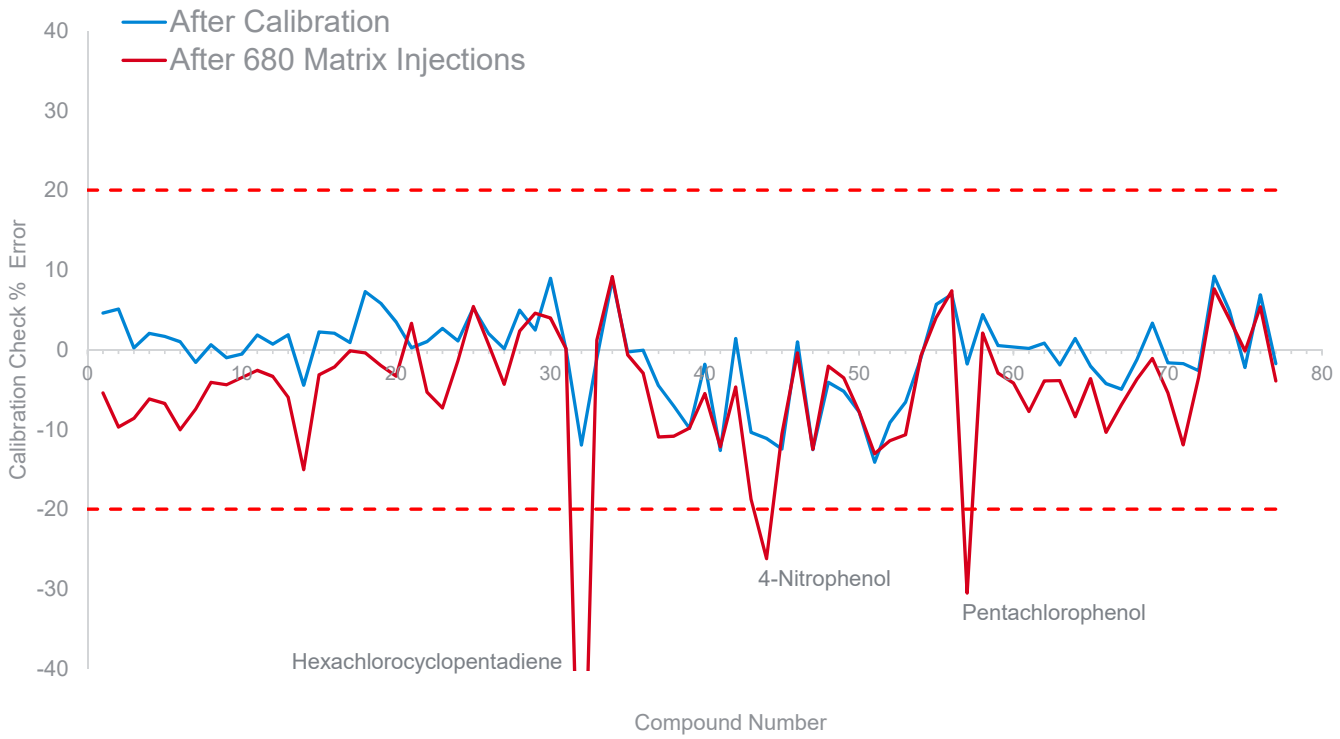
Matrix Study - Calibration

Continuing Calibration Verification



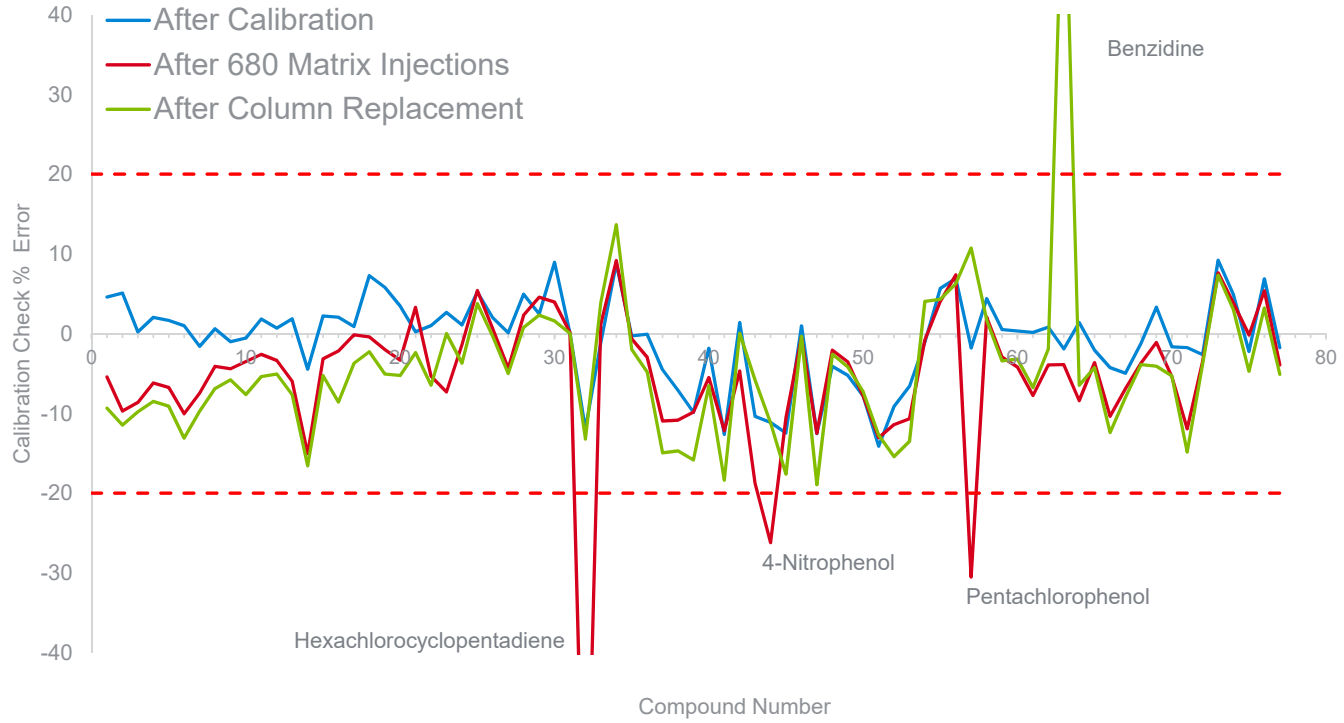
Matrix Study - Calibration

Continuing Calibration Verification



Matrix Study - Calibration

Continuing Calibration Verification



Comparison to 7890 – Matrix Study

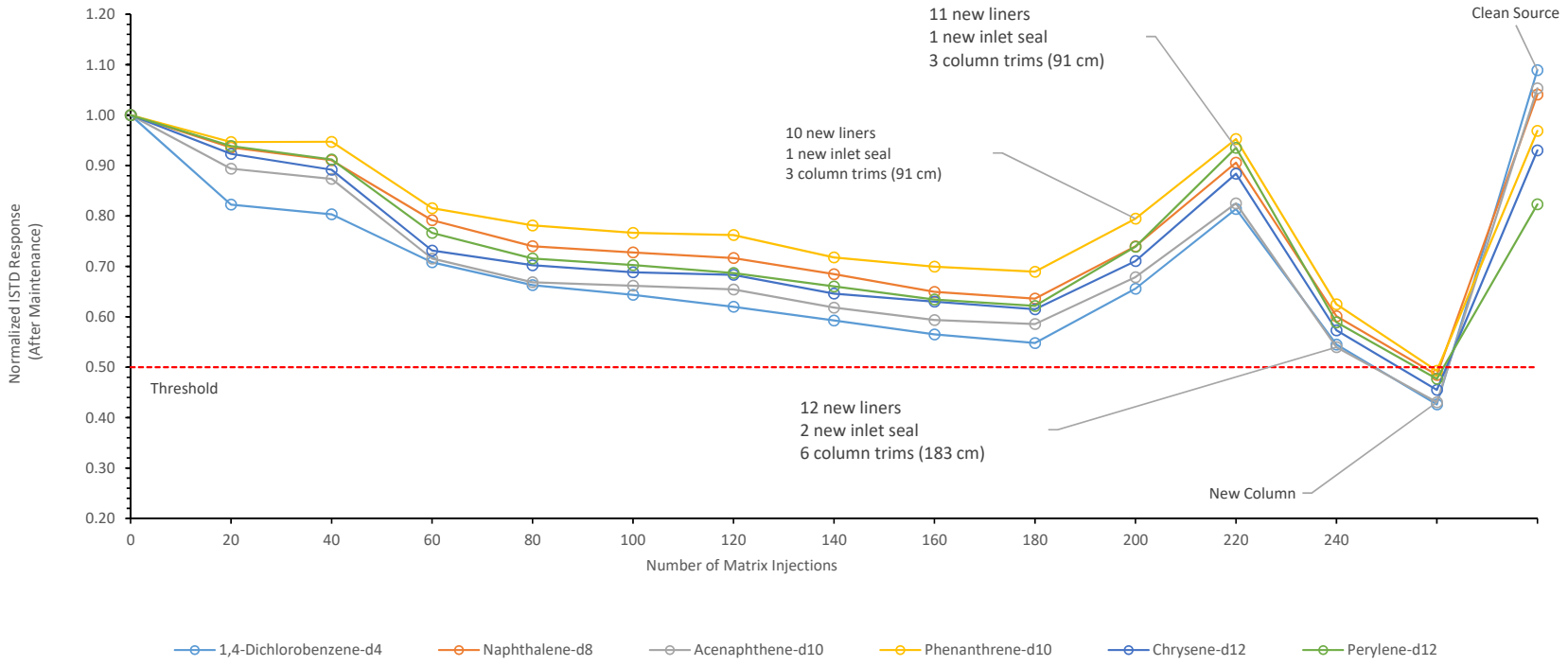
Parallel studies carried out on 7890 using three separate columns

Column Number	Number of Liners and Septa Replaced	Number of Inlet Seals Replaced	Number of Column Trims (30.5 cm each)	Total Number of Matrix Injections
1	12	2	6	240
2	4	2	5	80
3	6	3	4	120

Mechanisms of failure:

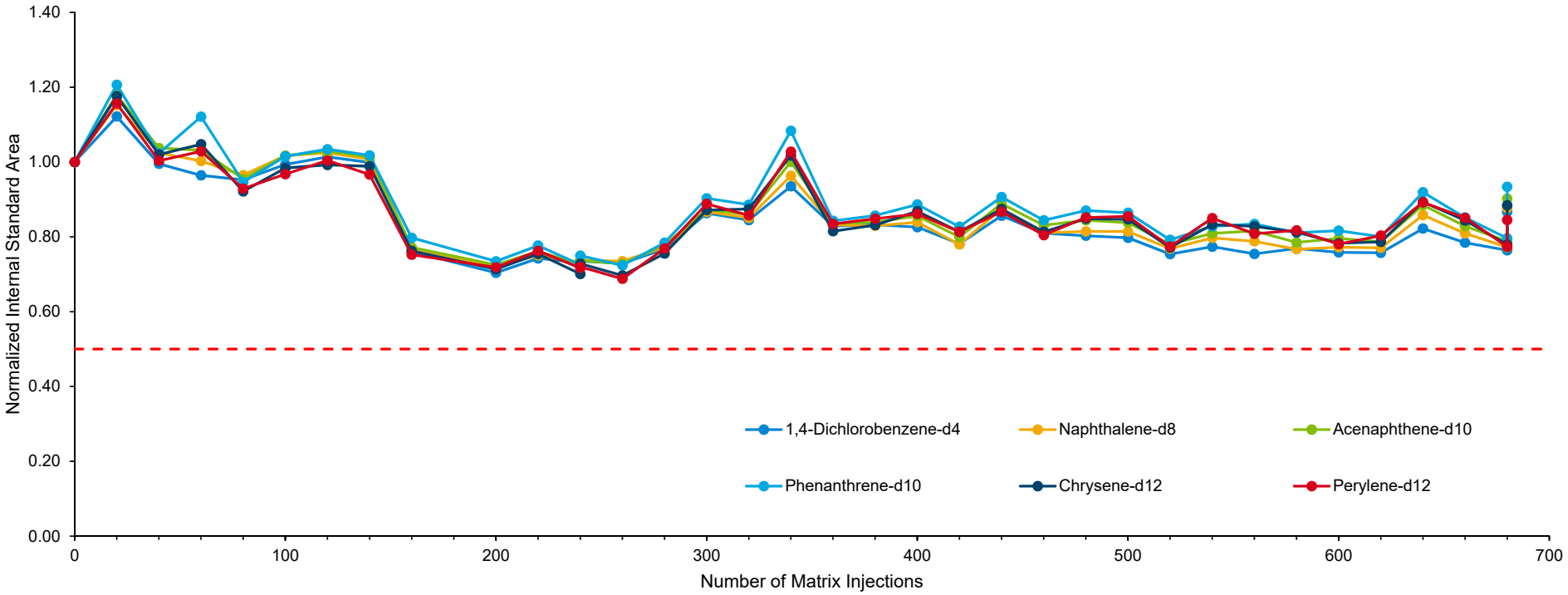
- Column 1 and 3
  - ISTD response below 50% and greater than 10% CCV failure
  - ISTD response recovered with source cleaning
- Column 2 – greater than 10% CCV failure
  - CCV failure recovered after column replacement

Internal Standard Check – 7890 Column 1 Study



Internal Standard Check – Intuvo Study

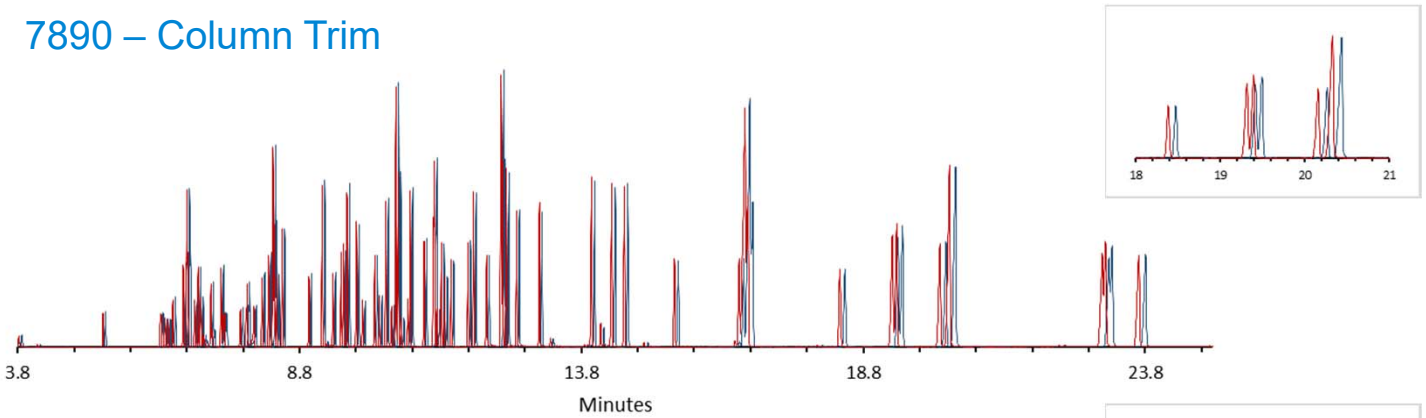
Internal Standard Response



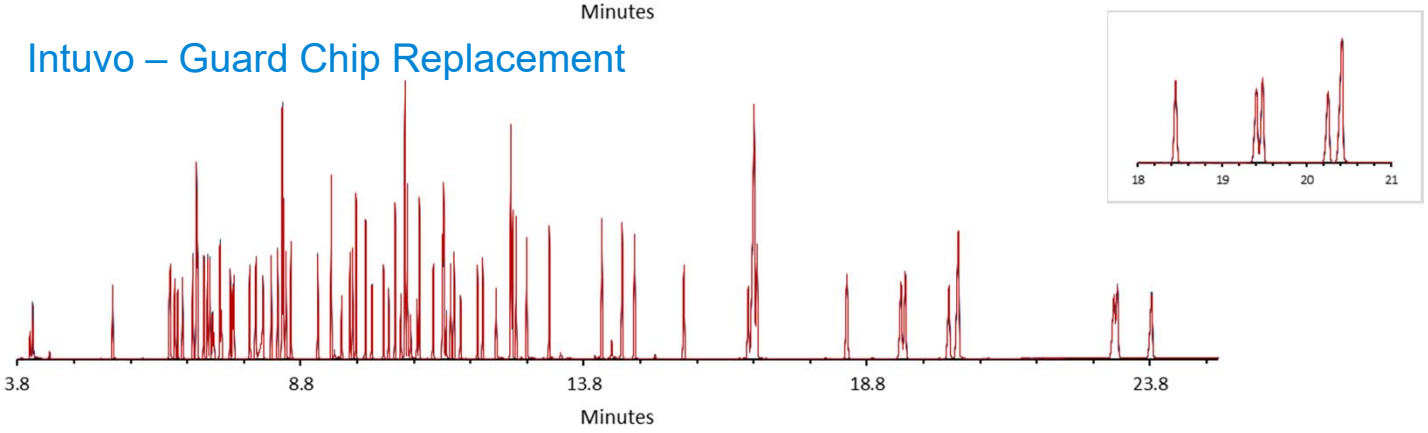
Comparison to 7890 – Retention Time Shift

Column trimming compared to guard chip replacement.

7890 – Column Trim



Intuvo – Guard Chip Replacement



Conclusions

SVOC retention times and responses on the Intuvo 9000 GC are equivalent to 7890 GC using equivalent methods.

Agilent 9000 easily achieves system suitability and calibration criteria as specified in USEPA 8270D.

Matrix study indicate substantial system robustness to soil extracts.

Guard chip replacement does not change retention time compared to column trimming on a 7890.

THANK YOU

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