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HUMAN HEALTH | ENVIRONMENTAL HEALTH

Using Flow Switching Devices to Improve GC-MS Productivity in Environmental Analyses

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Content

- A novel reversed Deans' heartcutting system for application switching
  - The need
  - The design approach
  - Application to environmental analyses

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The Need for Application Switching

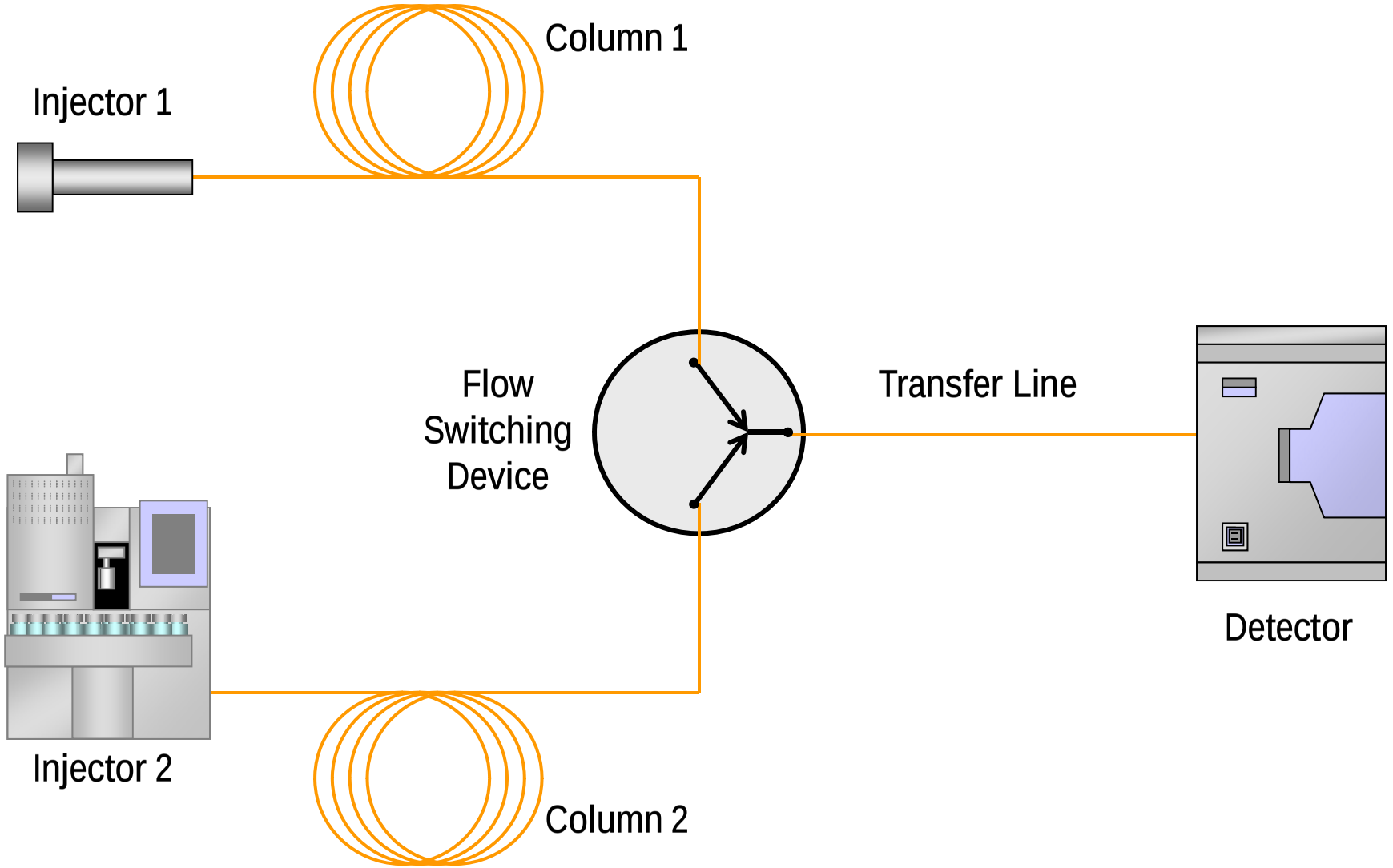
The Need

- Many labs wish to run more than one application on an expensive and sensitive analytical system such as a GC-MS.

- To do this, they must either:
  - Purchase more than one system
    - Expensive
    - Requires more bench-space
    - May be under-used
  - Swap columns and conditions
    - Time consuming
    - Prone to mistakes
    - Stresses system
    - Affects sample throughput

- Application switching offers convenience and ease of use without the significant added cost or increased bench-space requirements of multiple systems.

The Application Switching Concept



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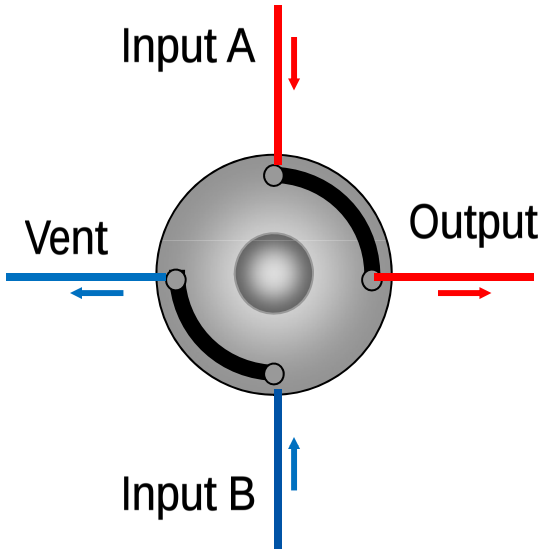
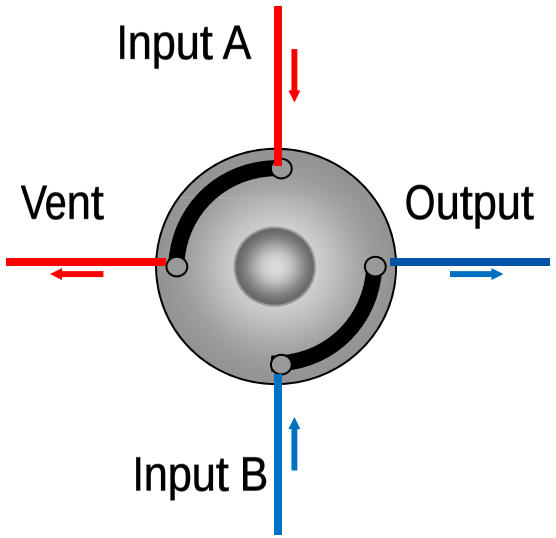
The Design Approach to Application Switching

Mechanical Switching Valve

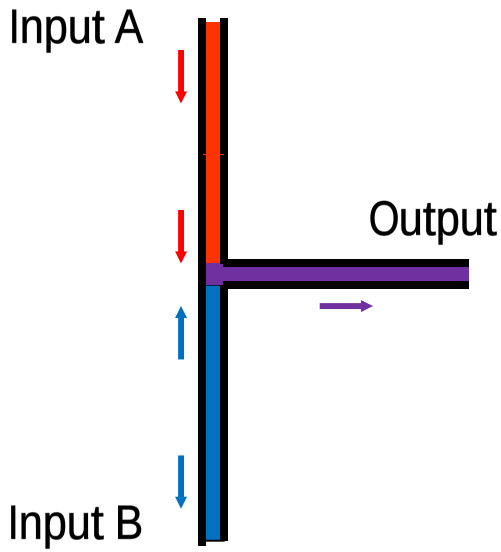
- High thermal mass - causing thermal lag
- Limited temperature limit – restricts application
- Moving parts – may wear and leak
- Metal surfaces – risk of activity
- Large internal volumes – risk of dispersion
- Causes flow disruption – baseline artifacts





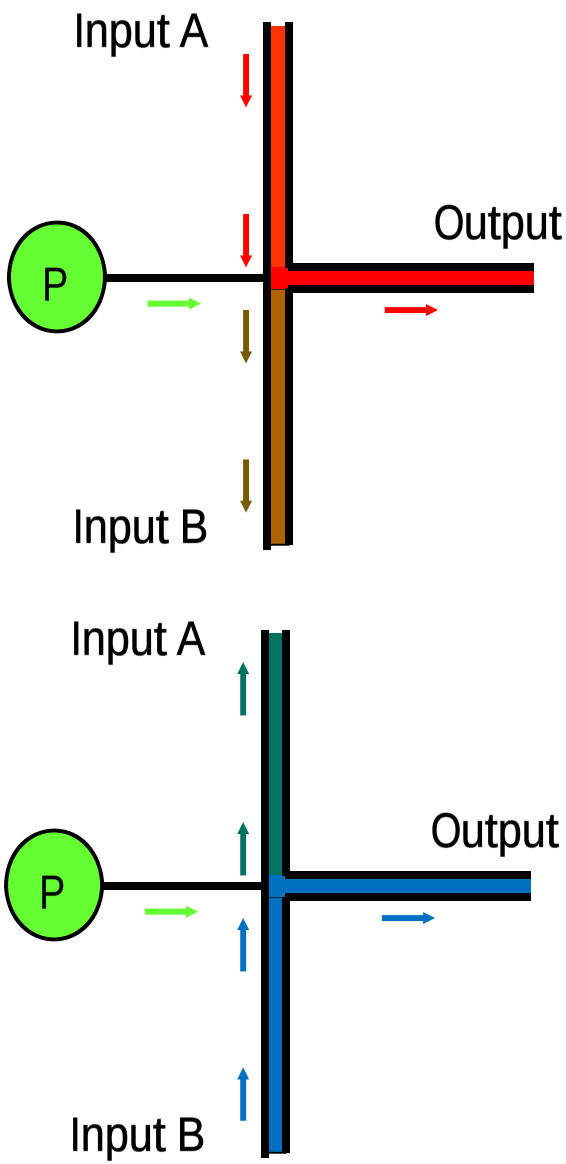
T-Piece

- Simple – doesn't need many parts
- Low thermal mass – no thermal lag
- Low dead volumes – gives good peak shape
- Easy to deactivate – high inertness
- Both columns always active – possible increase in column bleed and other contamination
- Output flow rate increased- may give problems with the MS



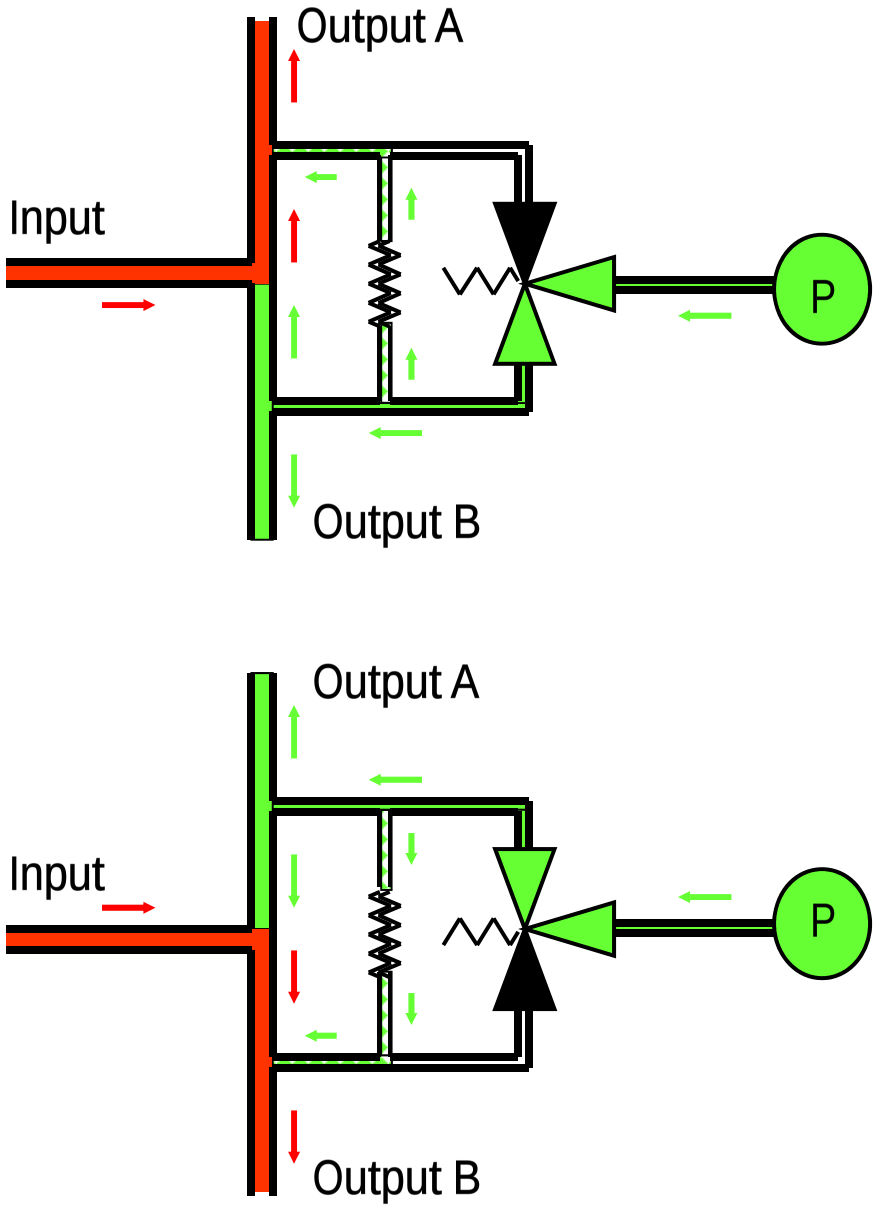
Pressure Controlled T-Piece

- Column is deactivated by dropping its inlet pressure
- Simple – doesn't need many parts
- Low thermal mass – no thermal lag
- Low dead volumes – gives good peak shape
- Easy to deactivate – high inertness
- Only one column is active – other is backflushed. No increase in bleed etc.
- Flow rate into detector only increased slightly – good for MS
- Sample from active column is split into inactive column – loss in effective sensitivity and cross-contamination issues



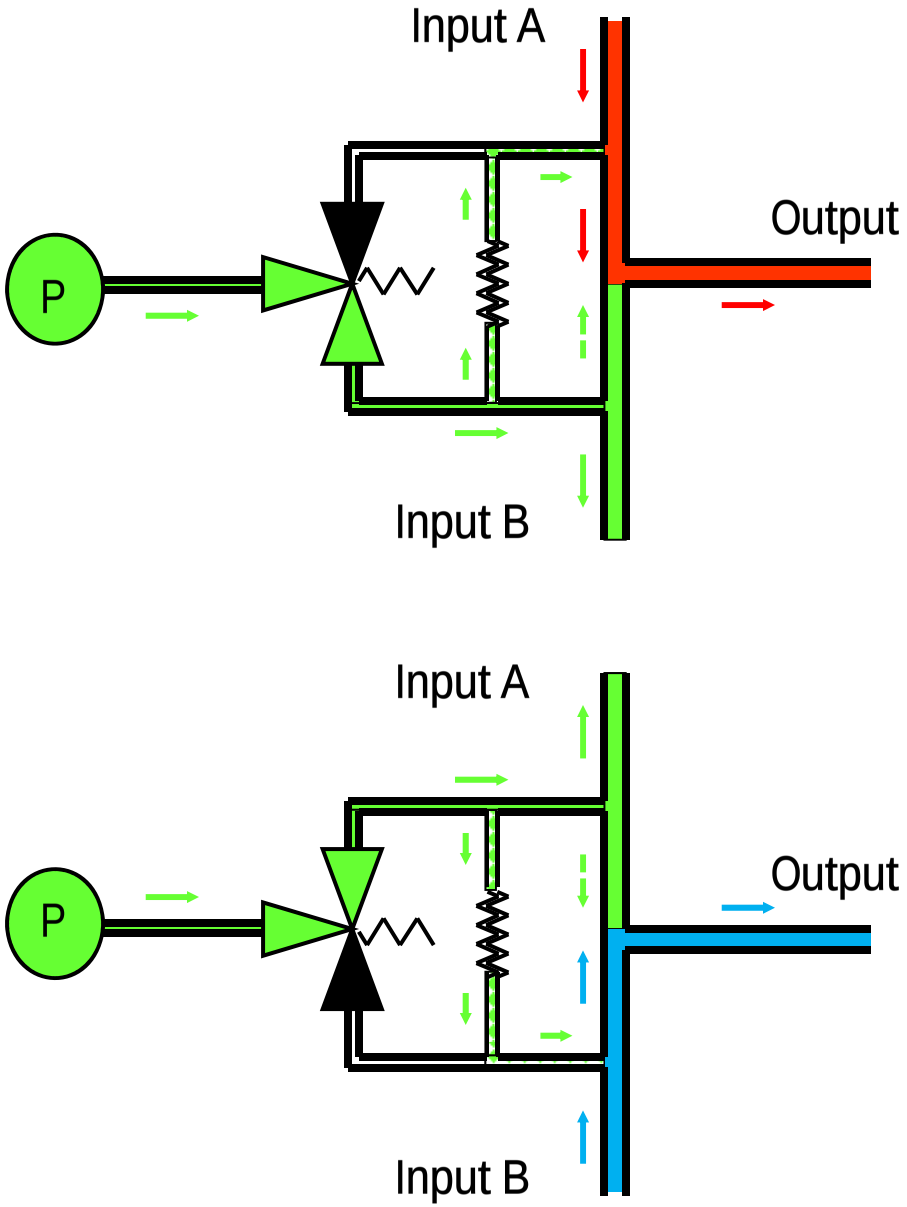
The Traditional Deans' Switch

- The simple switching of a solenoid valve directs the input flow between two outputs
- Low dead volumes
- Low thermal mass
- No moving parts
- Fast response
- Inert
- No disruption to flows



The Reversed Deans' Switch

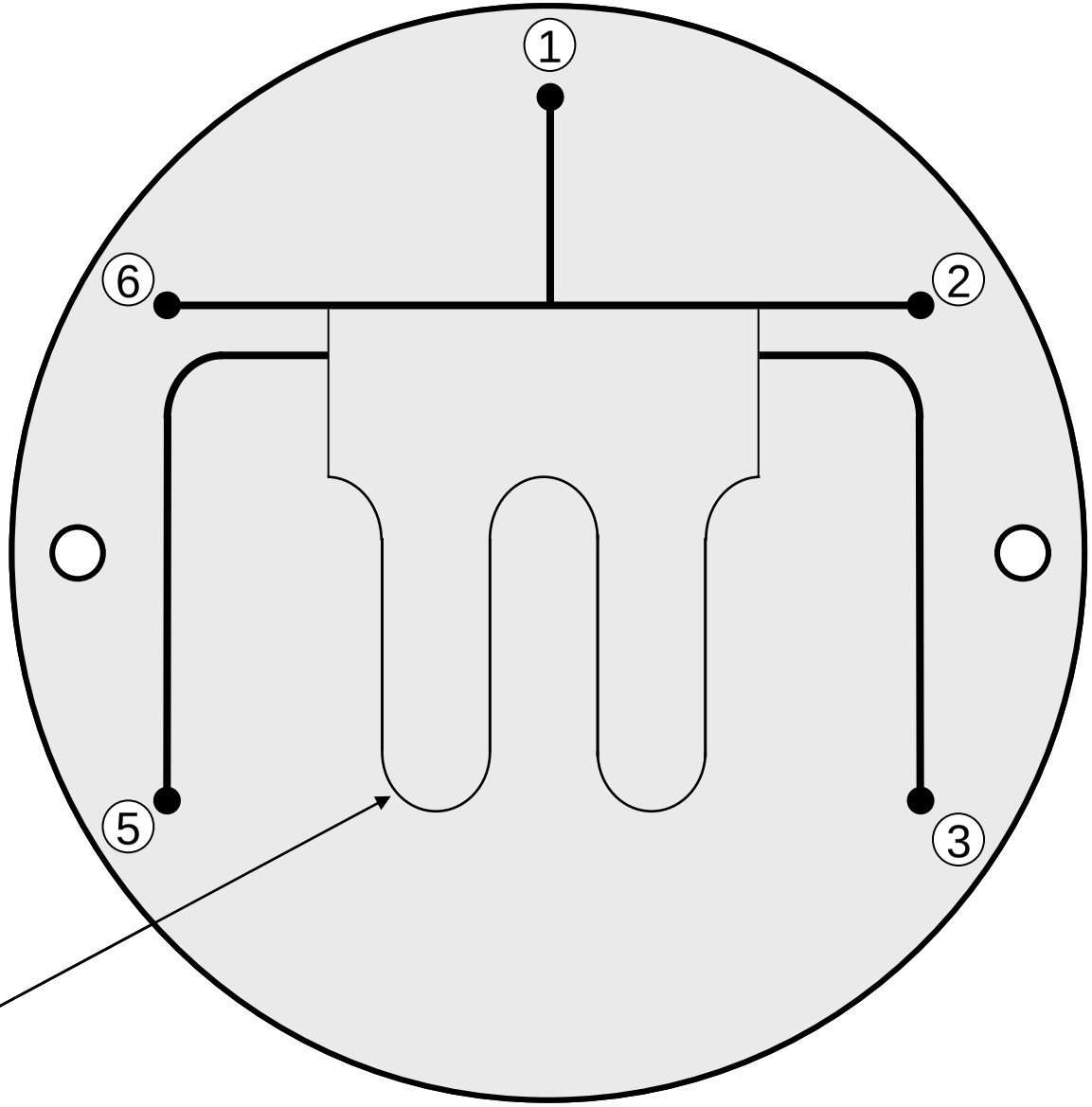
- The simple switching of a solenoid valve directs the output flow between two inputs
- An input is made inactive by dropping its pressure slightly (e.g. by lowering the pressure at the column injector)
- No disruption to detector flow
- Pressure balancing is not so important as long as the Golden Rule is observed: *the flow from the active input must be less than the output flow*.



The Swafer Technology

Ports 1,2 and 6 are configured according to application

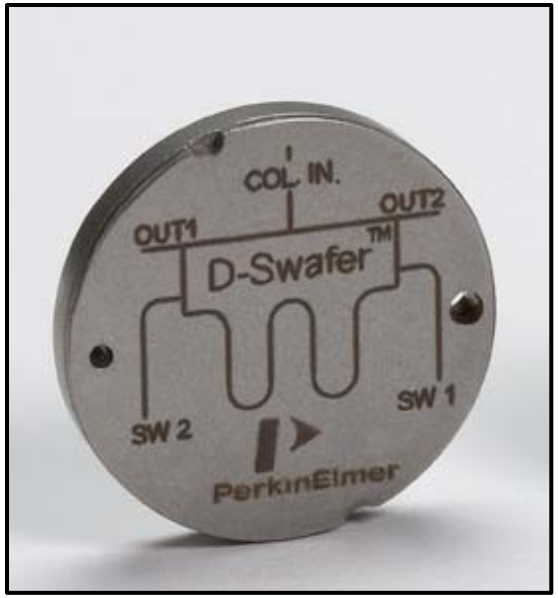
Ports 3 and 5 are inputs for switching gas



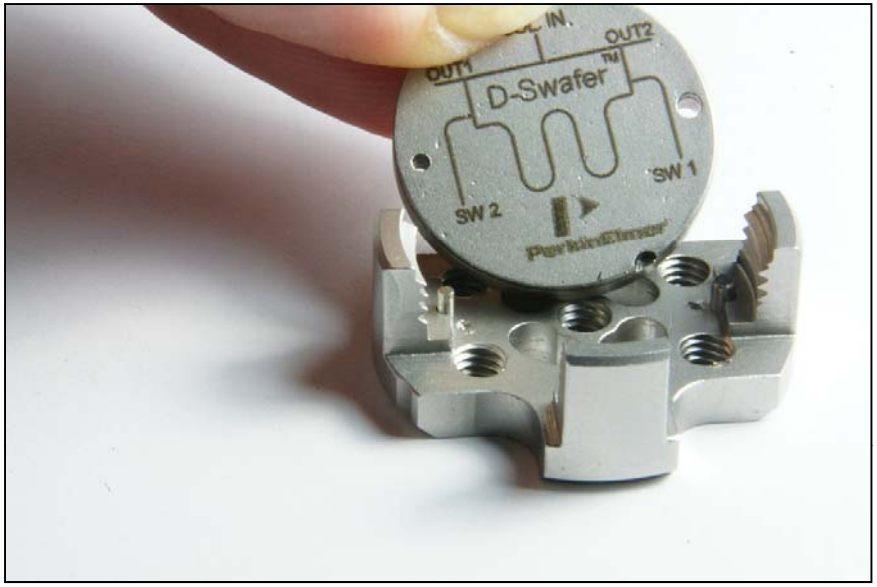
Integrated by-pass restrictor

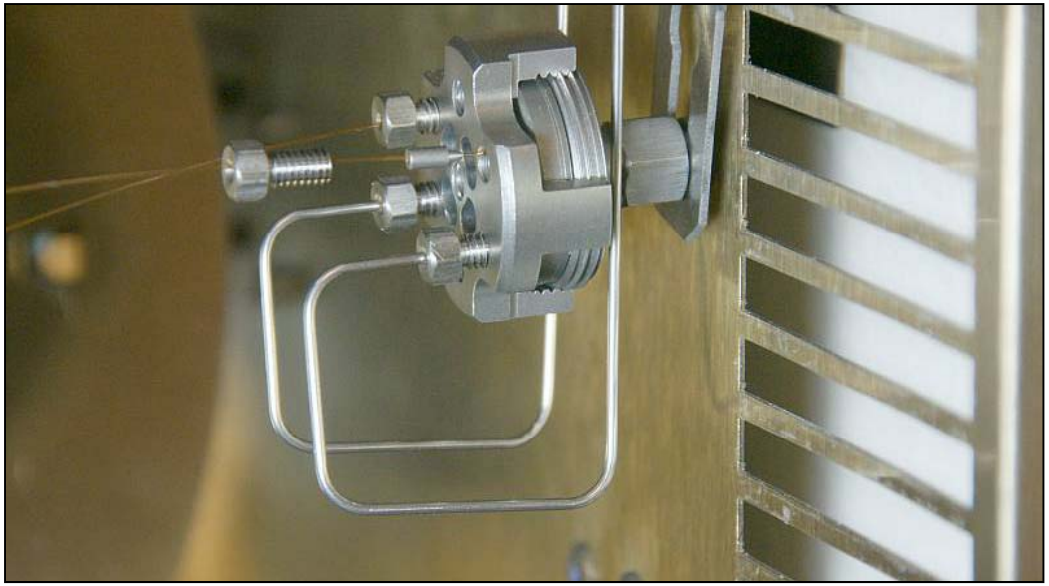
Swafer Technology

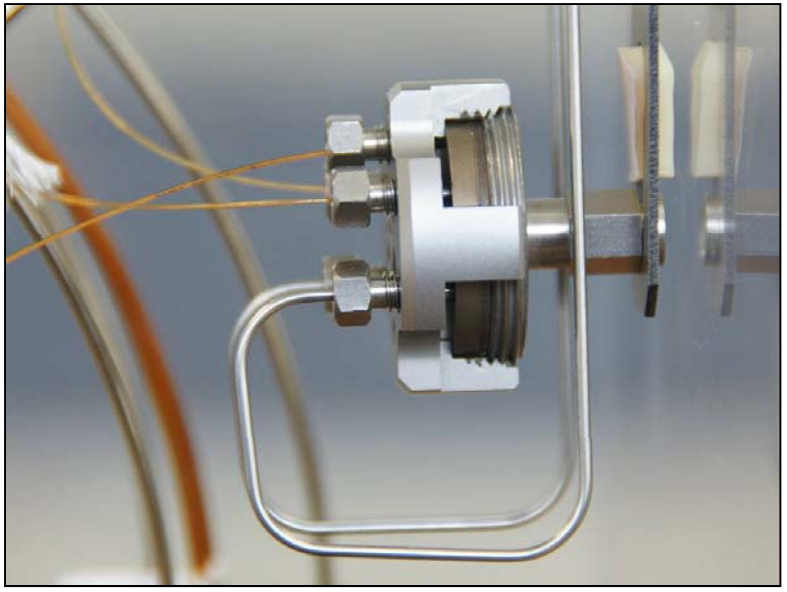
- About the same size as a US 5-cent coin
- Layers are 80µm thick
- Channels are laser-fabricated to different widths down to 50µm
- Channels are chemically deactivated
- Easy to remove and replace
- Low effective thermal mass holder



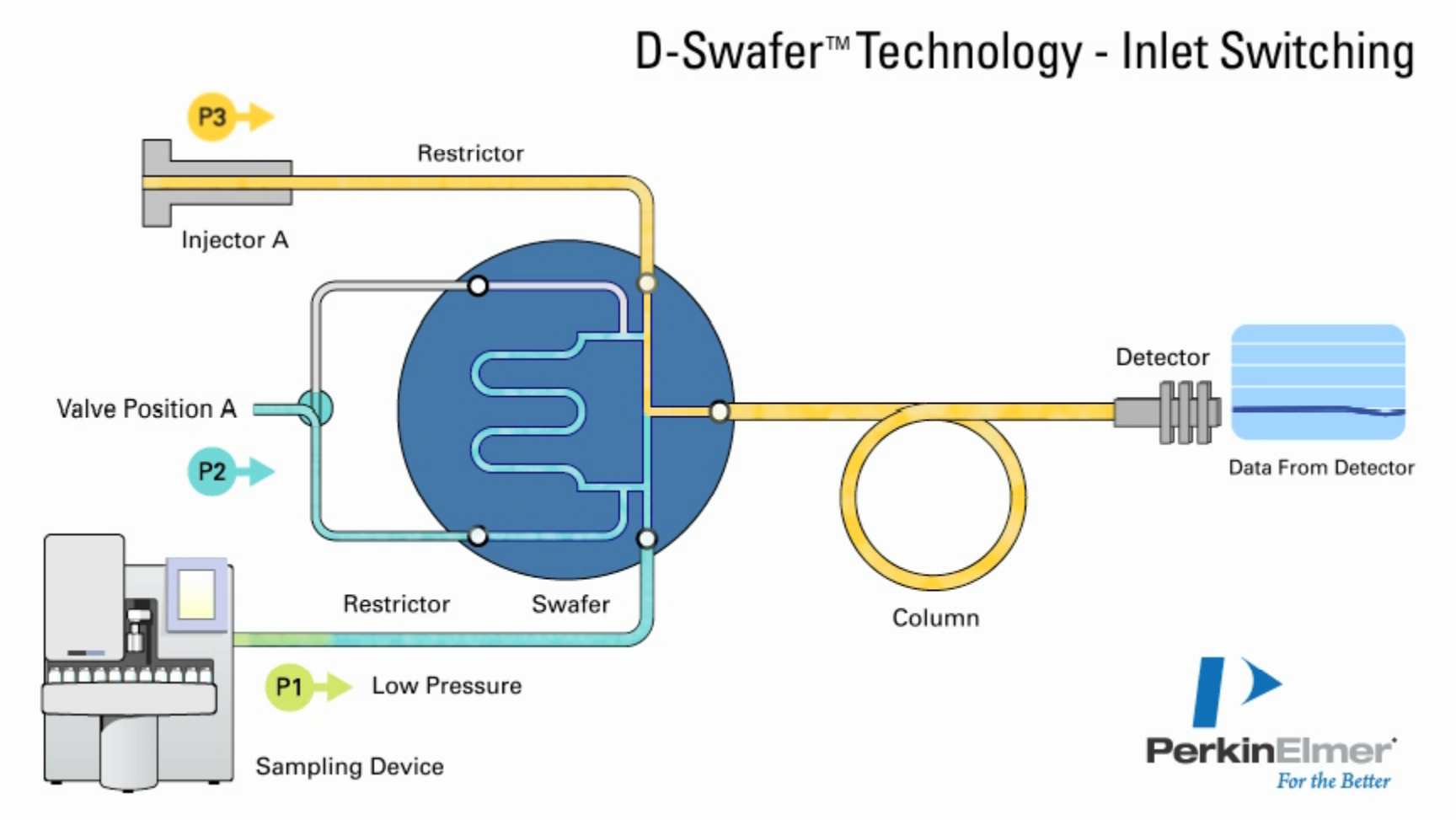
Photos of the Technology







Demo Animation



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Example A
Simple HS/GC/MS Methods

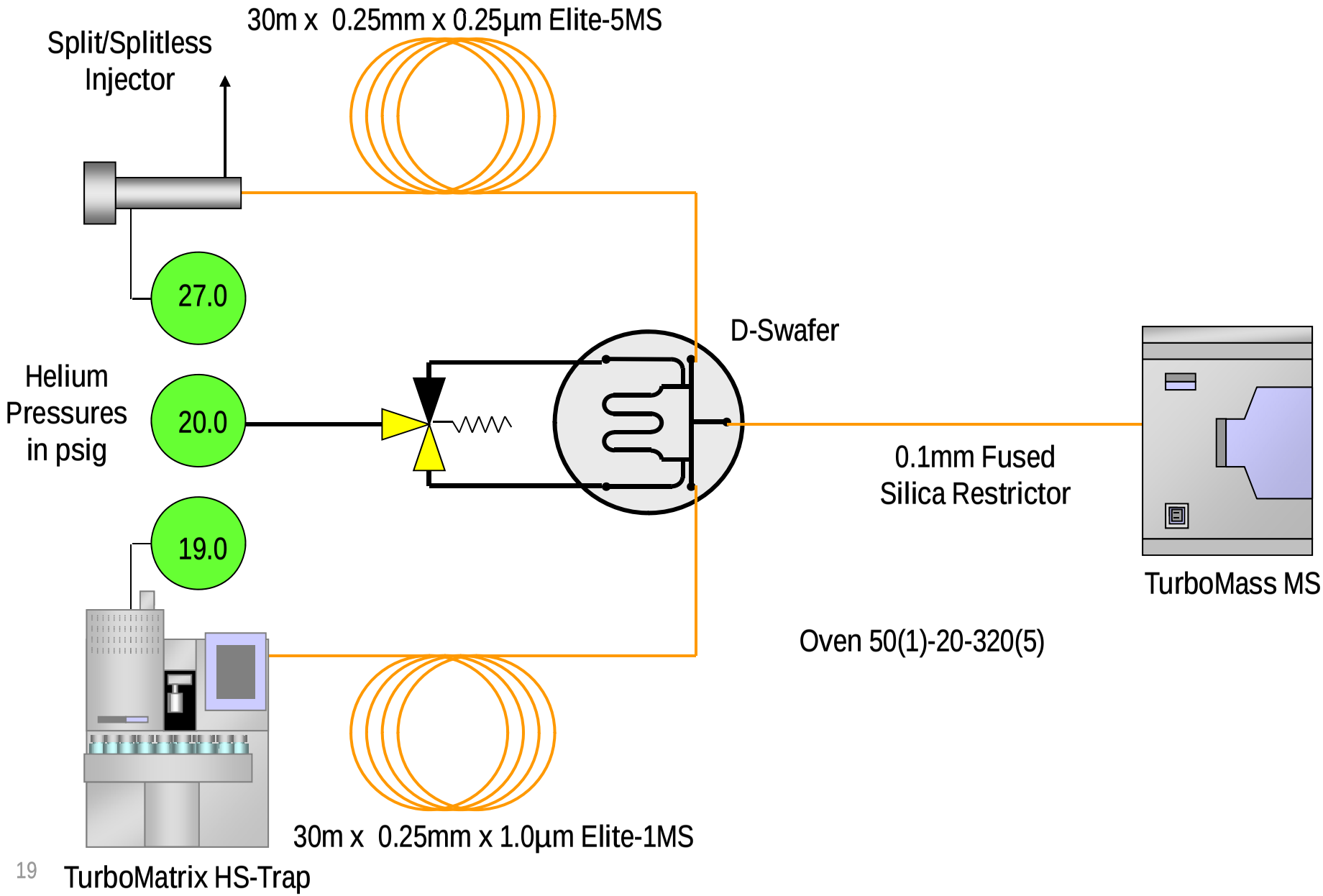
Example A - 3 Simple Applications on 1 GC/MS

- PAH Analysis
  - Liquid extraction of liquid or solid matrix
  - Liquid autosampler introduction of extract
  - Thin Film Column
- Nonylphenol Analysis
  - Liquid extraction
  - Liquid autosampler introductions of extract
  - Thin film column
- Trihalomethane Analysis
  - HS Analysis of Water
  - Thick film column

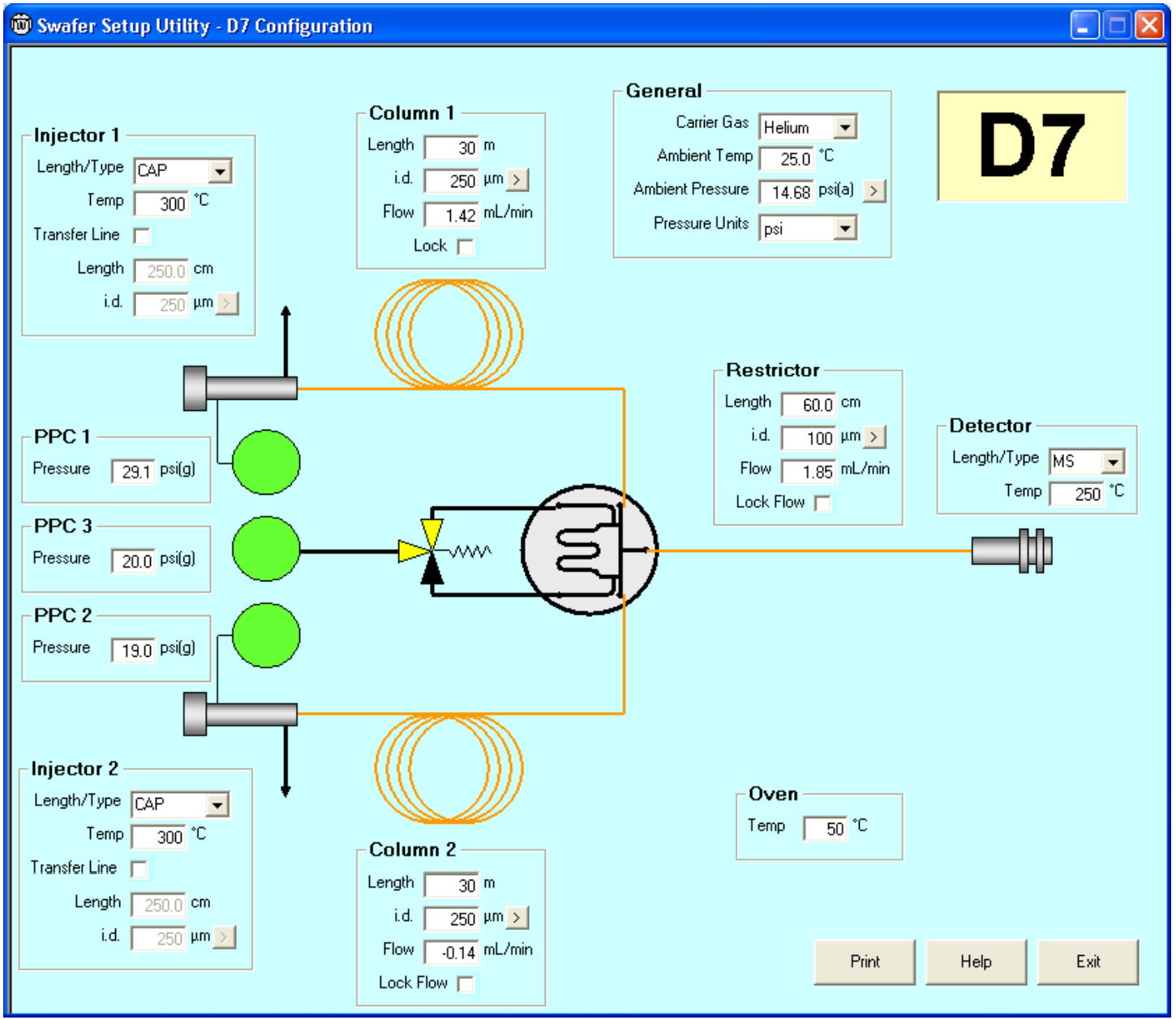
Requirements

- PAH to be calibrated between 0.2 - 10 ug/mL
- Nonylphenol to be calibrated between 5 – 50 ug/mL
- Trihalomethanes should have a quantification limit of 1.0 pg/mL
- It should be possible to switch between HS Trap analysis and liquid injection in a sequence automatically – unattended.

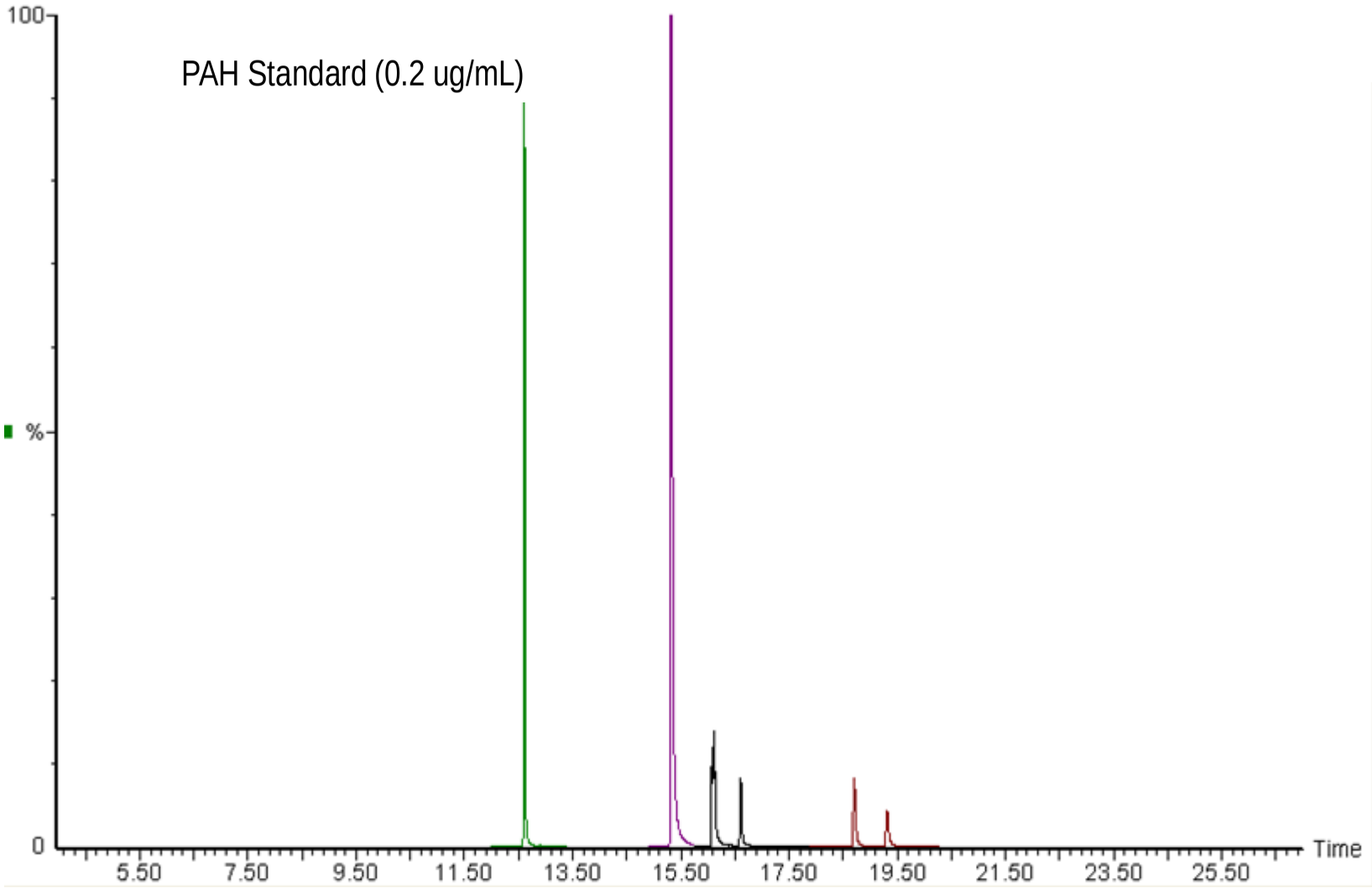
Conditions for PAH Analysis



Software Tool to Aid Method Development

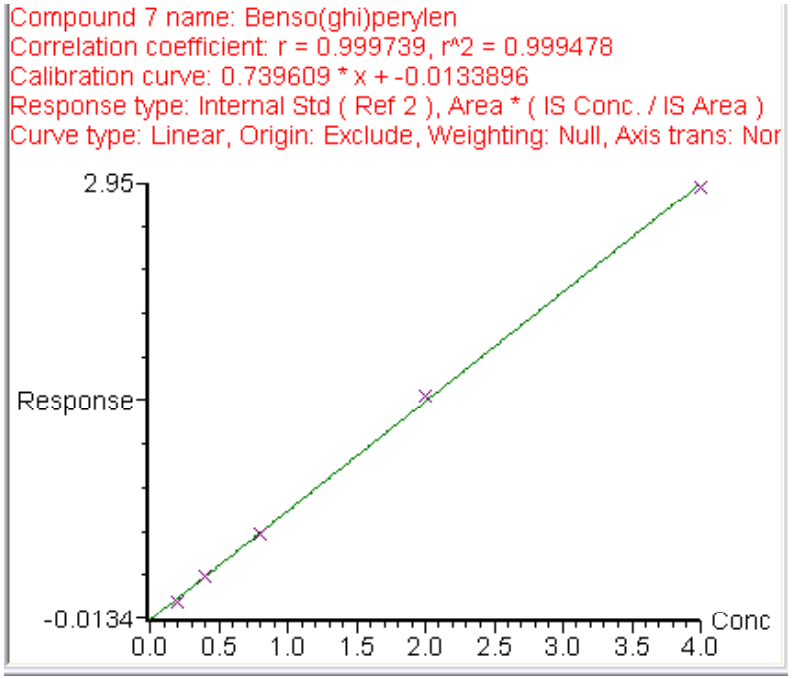


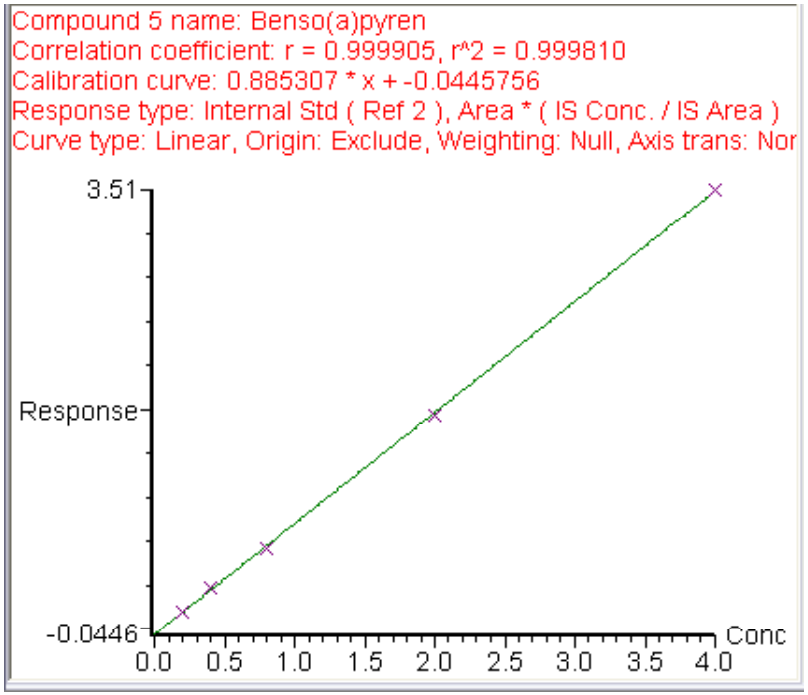
PAH Analysis



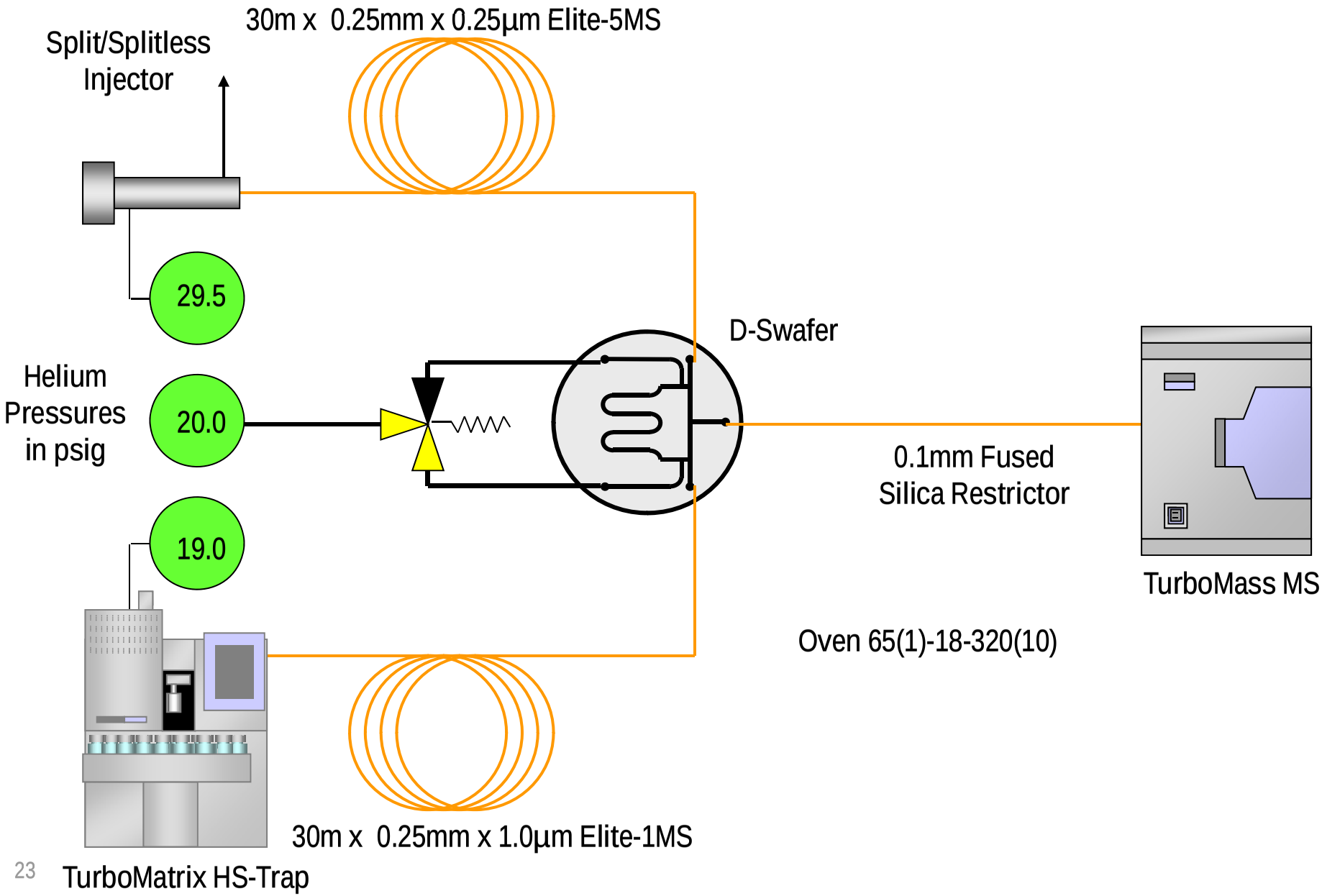
PAH Quantitative Performance

PAH	1	2	3	4	5	% RSD (n=5)	r^2
benzo(b)fluoranthene	0.2	0.4	0.8	2	4	1.9	0.9935
benzo(k)fluoranthene	0.2	0.4	0.8	2	4	2.2	0.9986
benzo(a)pyrene	0.2	0.4	0.8	2	4	3.2	0.9998
indeno(1,2,3-cd)pyrene	0.4	0.8	1.6	4	8	7.3	0.9985
benzo(ghi)perylene	0.2	0.4	0.8	2	4	5.3	0.9995

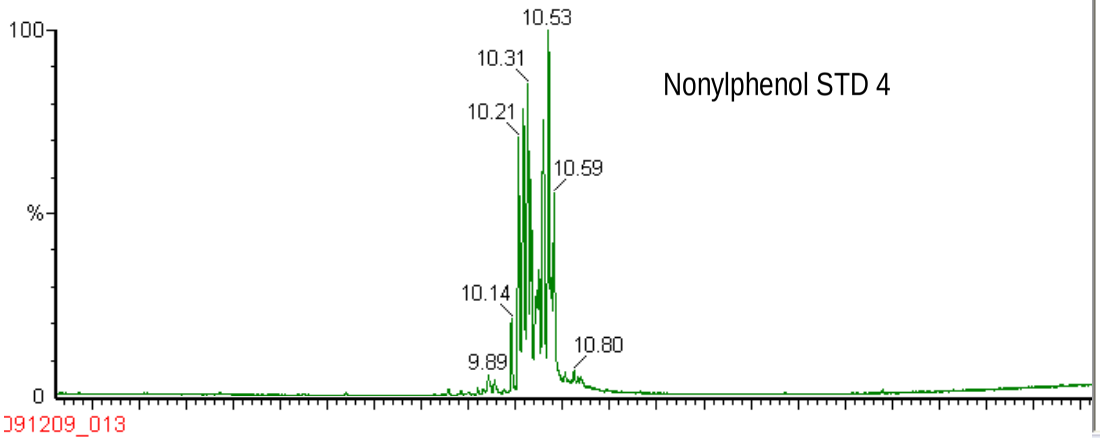




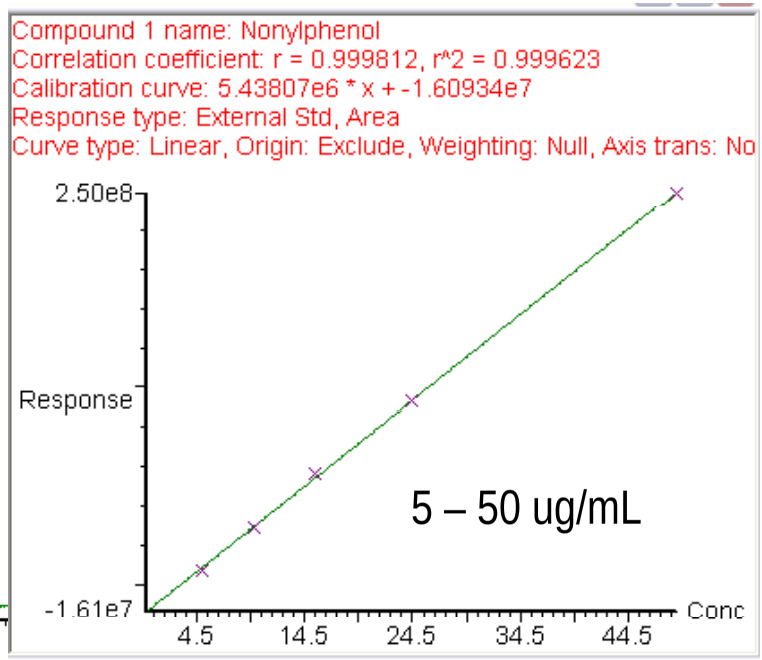
Conditions for Nonylphenol Analysis

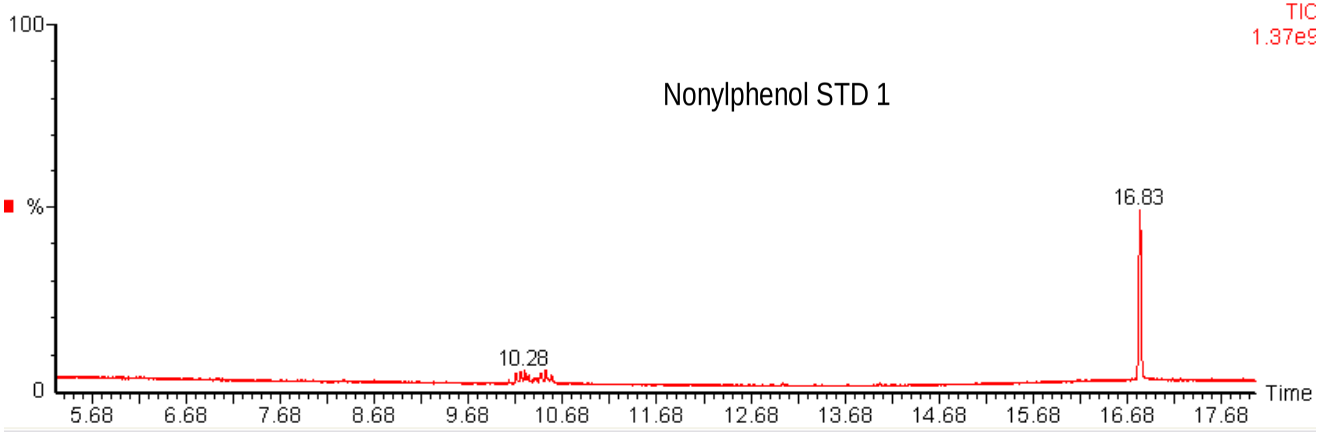


Nonylphenol Performance



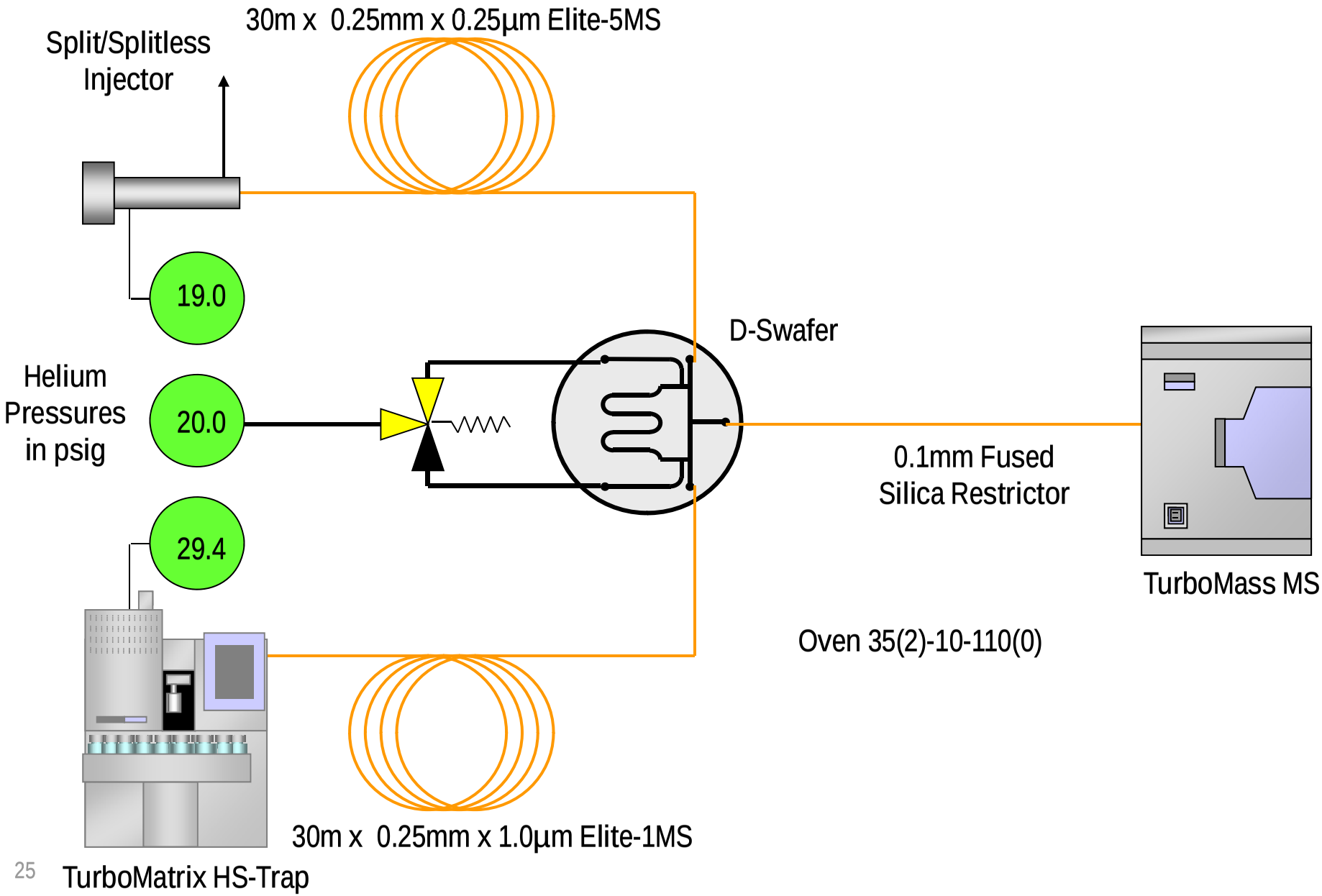
Nonylphenol STD 4





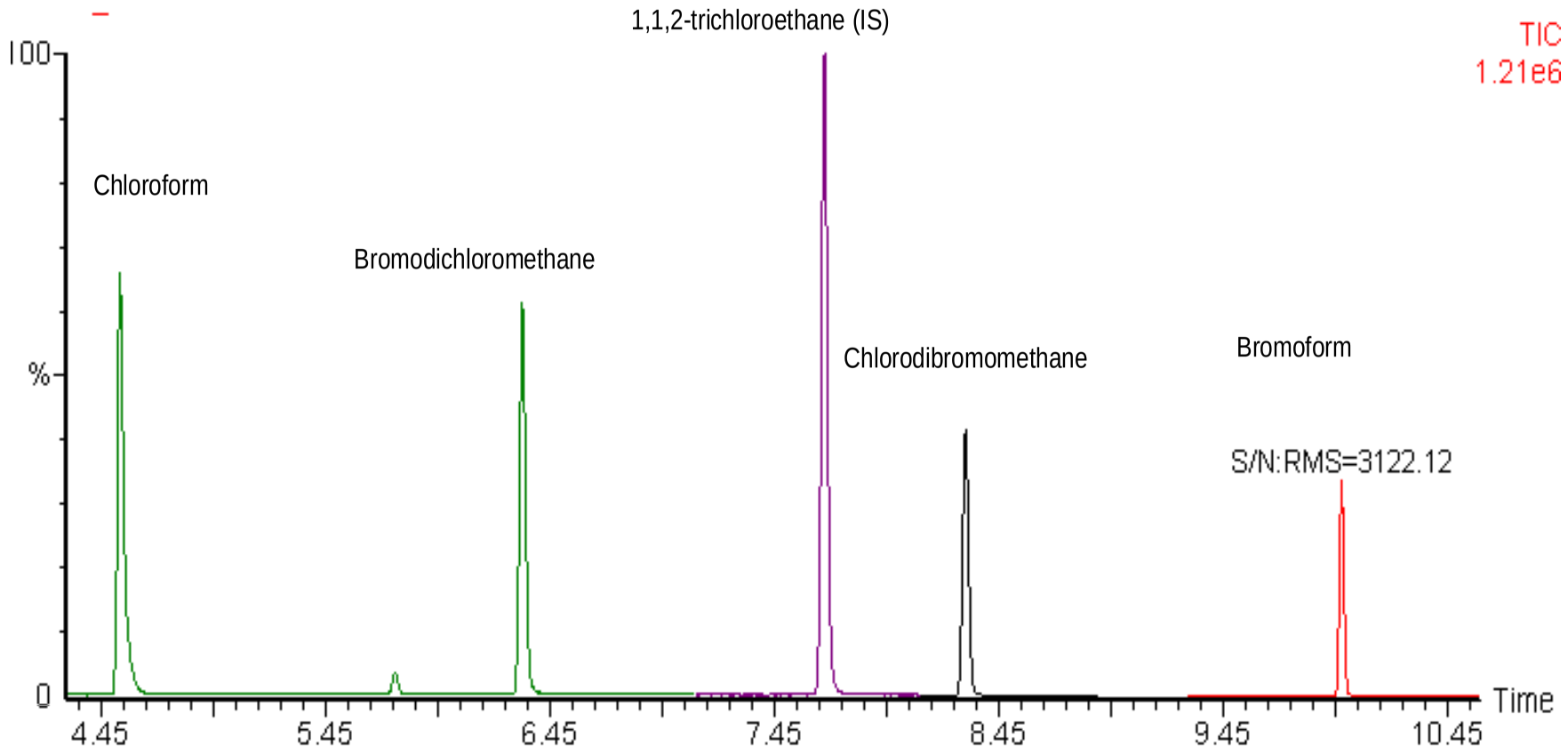
Nonylphenol STD 1

Conditions for Trihalomethane Analysis

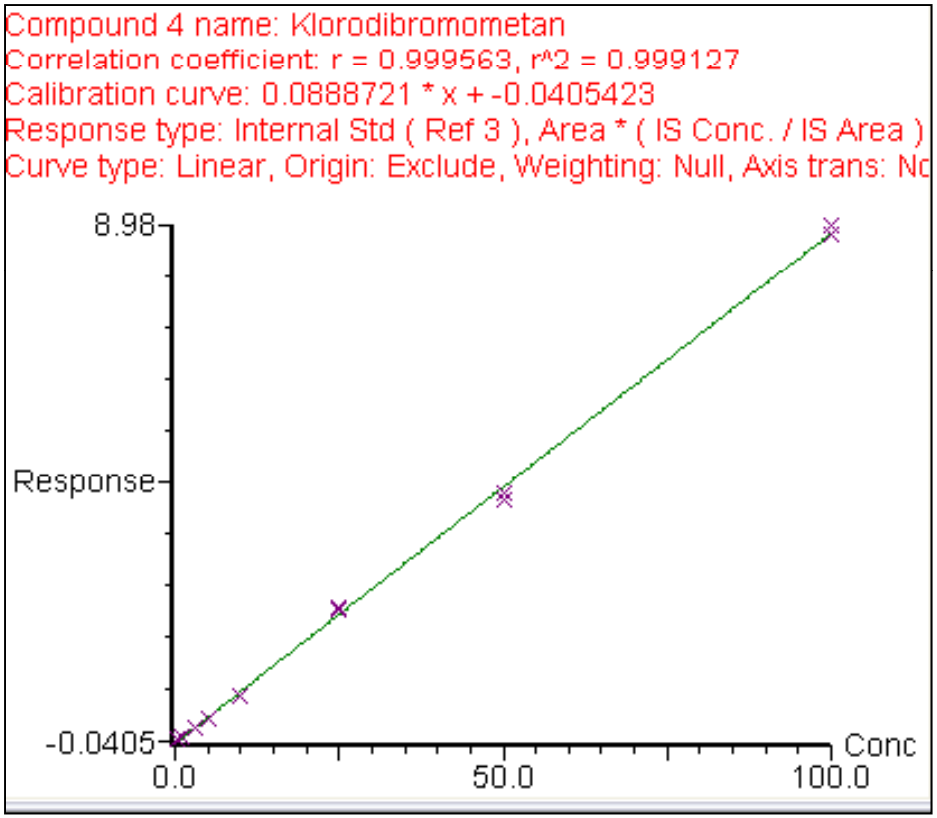


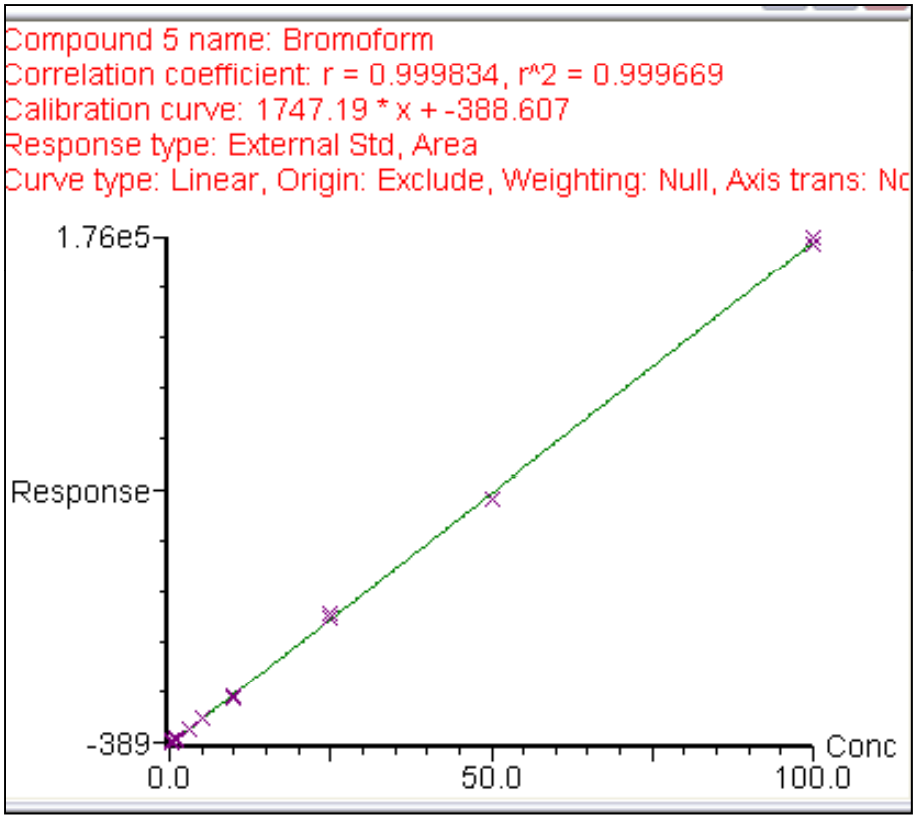
Trihalomethane Analysis

SIR, 10 mL (H2O), 0.5 ng/mL trihalomethane standard

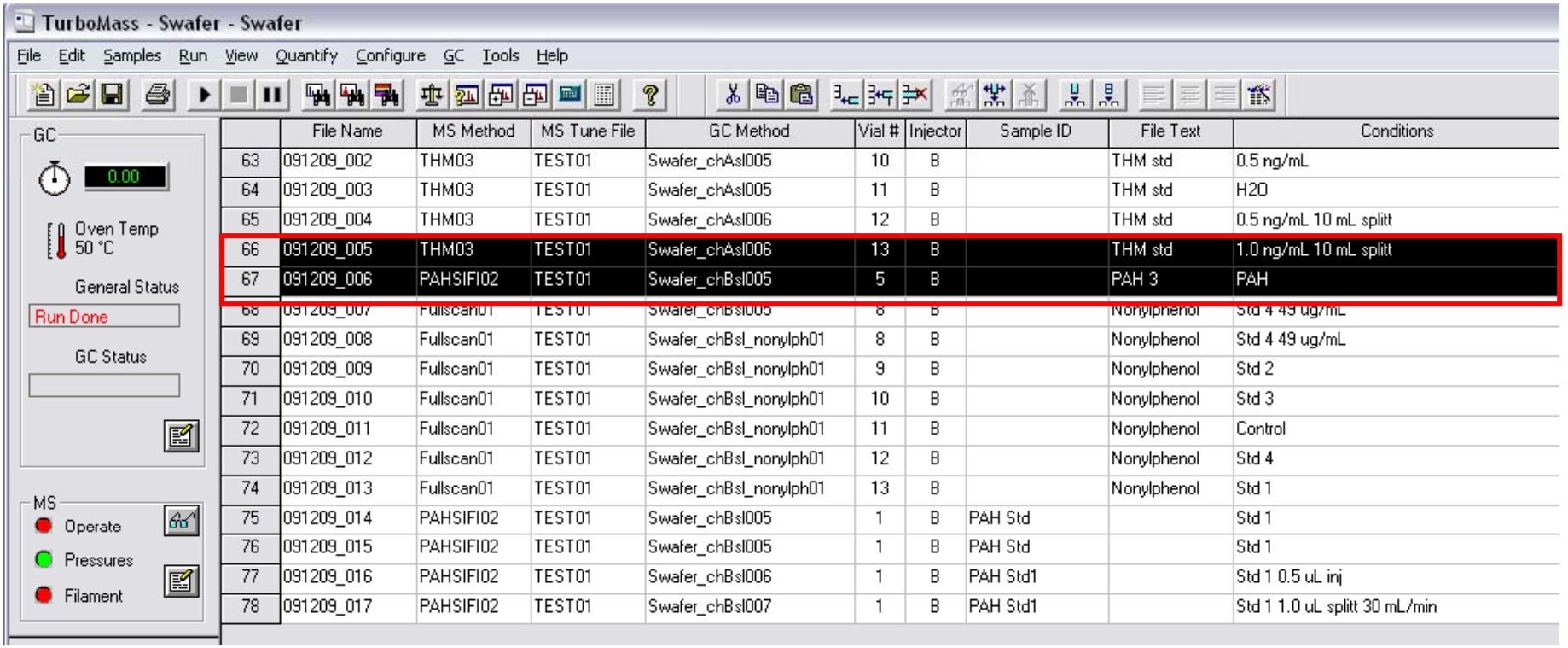


Trihalomethane Analytical Results



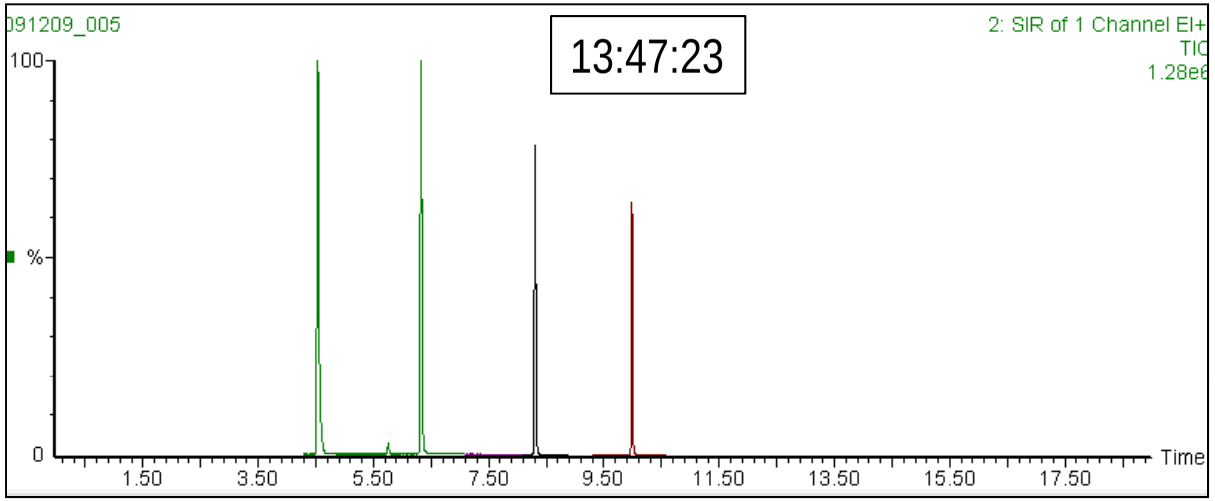


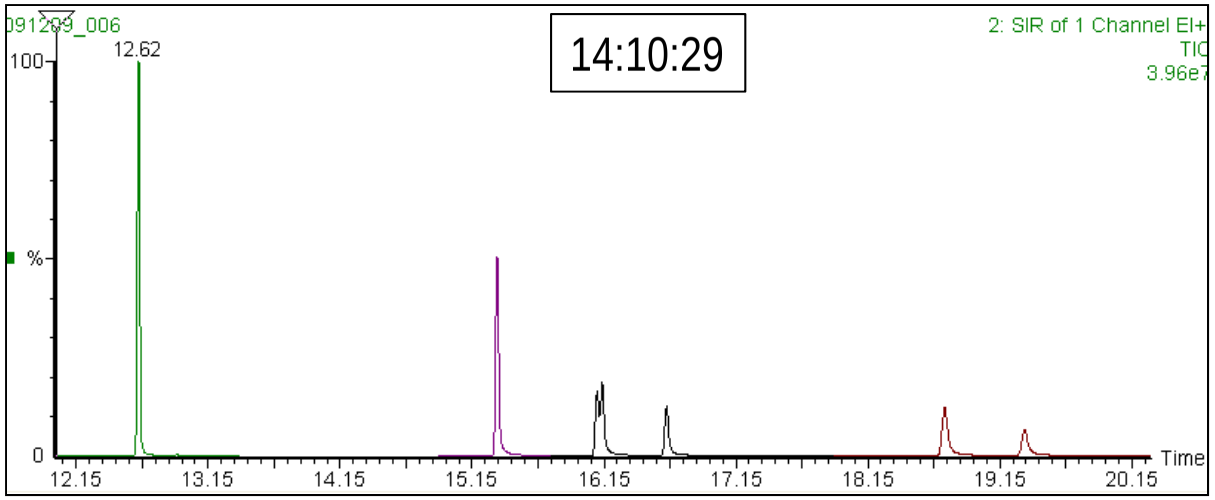
Switching between Liquid and HS Injection



	File Name	MS Method	MS Tune File	GC Method	Vial #	Injector	Sample ID	File Text	Conditions
63	091209_002	THM03	TEST01	Swafer_chAsl005	10	B		THM std	0.5 ng/mL
64	091209_003	THM03	TEST01	Swafer_chAsl005	11	B		THM std	H2O
65	091209_004	THM03	TEST01	Swafer_chAsl006	12	B		THM std	0.5 ng/mL 10 mL splitt
66	091209_005	THM03	TEST01	Swafer_chAsl006	13	B		THM std	1.0 ng/mL 10 mL splitt
67	091209_006	PAHSIFI02	TEST01	Swafer_chBsl005	5	B		PAH 3	PAH
68	091209_007	Fullscan01	TEST01	Swafer_chBsl005	8	B		Nonylphenol	Std 4 49 ug/mL
69	091209_008	Fullscan01	TEST01	Swafer_chBsl_nonylph01	8	B		Nonylphenol	Std 4 49 ug/mL
70	091209_009	Fullscan01	TEST01	Swafer_chBsl_nonylph01	9	B		Nonylphenol	Std 2
71	091209_010	Fullscan01	TEST01	Swafer_chBsl_nonylph01	10	B		Nonylphenol	Std 3
72	091209_011	Fullscan01	TEST01	Swafer_chBsl_nonylph01	11	B		Nonylphenol	Control
73	091209_012	Fullscan01	TEST01	Swafer_chBsl_nonylph01	12	B		Nonylphenol	Std 4
74	091209_013	Fullscan01	TEST01	Swafer_chBsl_nonylph01	13	B		Nonylphenol	Std 1
75	091209_014	PAHSIFI02	TEST01	Swafer_chBsl005	1	B	PAH Std		Std 1
76	091209_015	PAHSIFI02	TEST01	Swafer_chBsl005	1	B	PAH Std		Std 1
77	091209_016	PAHSIFI02	TEST01	Swafer_chBsl006	1	B	PAH Std1		Std 1 0.5 uL inj
78	091209_017	PAHSIFI02	TEST01	Swafer_chBsl007	1	B	PAH Std1		Std 1 1.0 uL splitt 30 mL/min

Switching between HS and Liquid





- HS Trap run of trihalomethanes standard (1.0 ng/mL) followed by a liquid injection of PAH standard 3.
- HS Sample injected at 13:47
- Liquid sample injected at 14:10
- Time difference 23 minutes.
- Headspace Trap method run time 18.8 min.
- GC oven cool down and liquid autosampler wash cycles = 4.2 minutes

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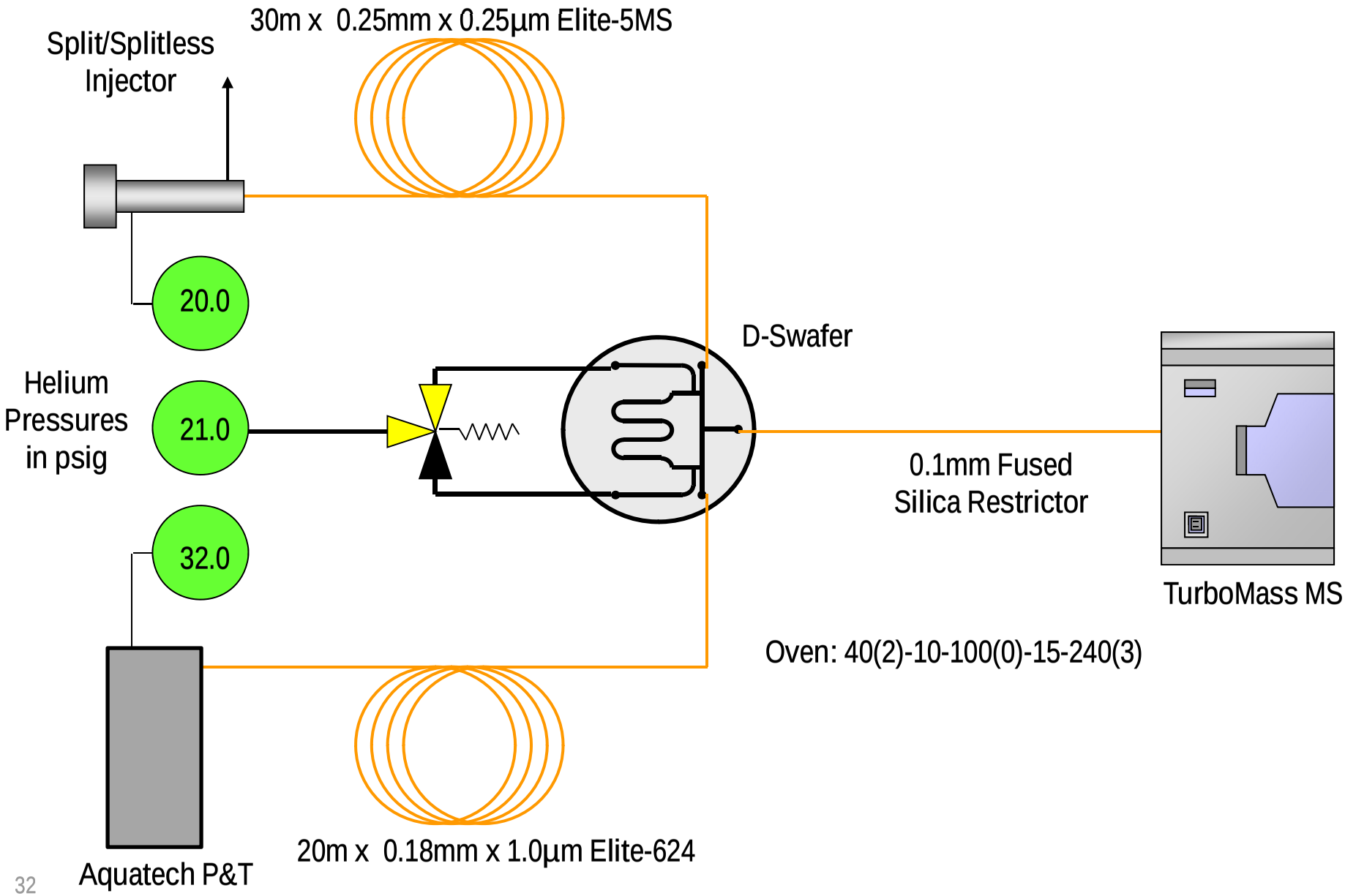
Example B
US-EPA Methods 8260 & 8270

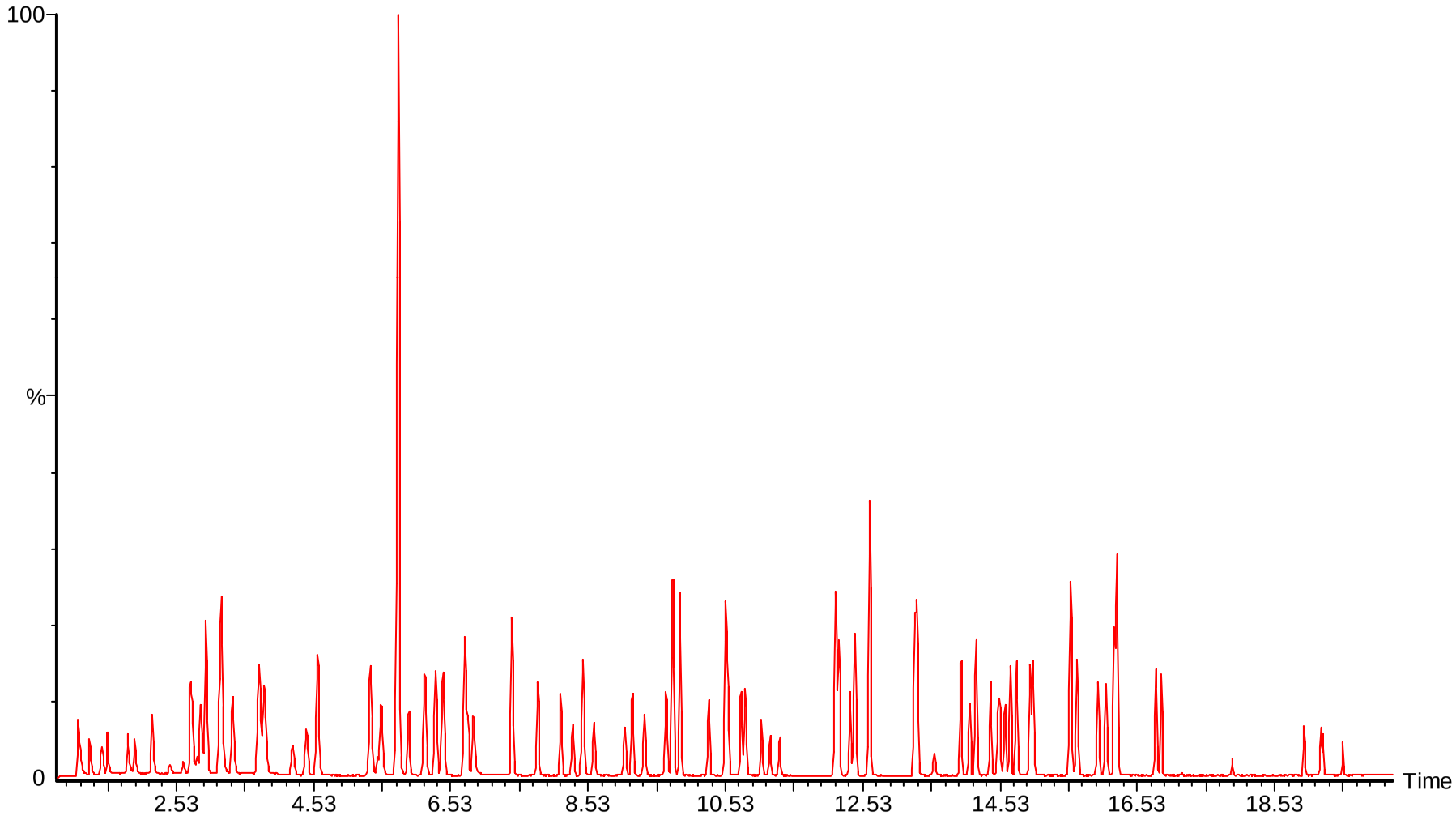
System for Switching Between Methods 8260 and 8270

- 8260: Determination of VOCs by Purge & Trap and GC/MS
- 8270: Determination of semi-volatiles by liquid injection and GC/MS

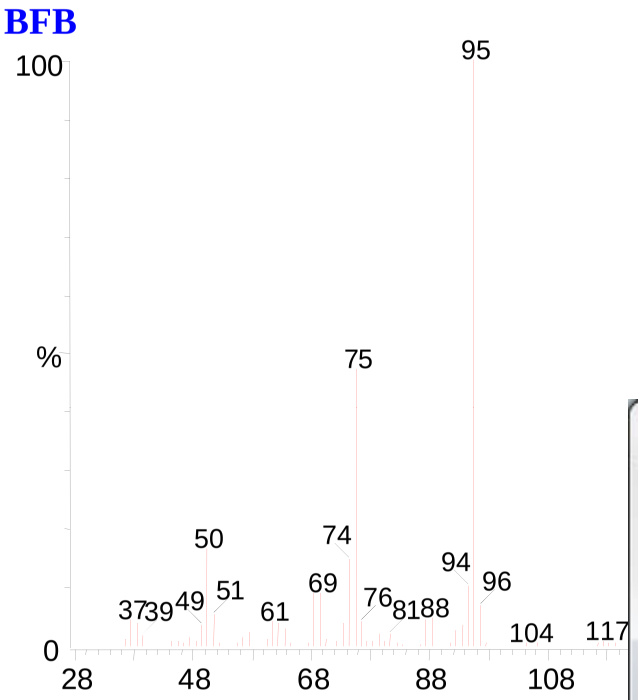
Conditions for 8260 Analysis



Volatiles 8260 Chromatogram



4-Bromofluorobenzene (BFB) Tune Evaluation



EPA Report

Test: BFB 624/8260 TEST Result: Test Passed

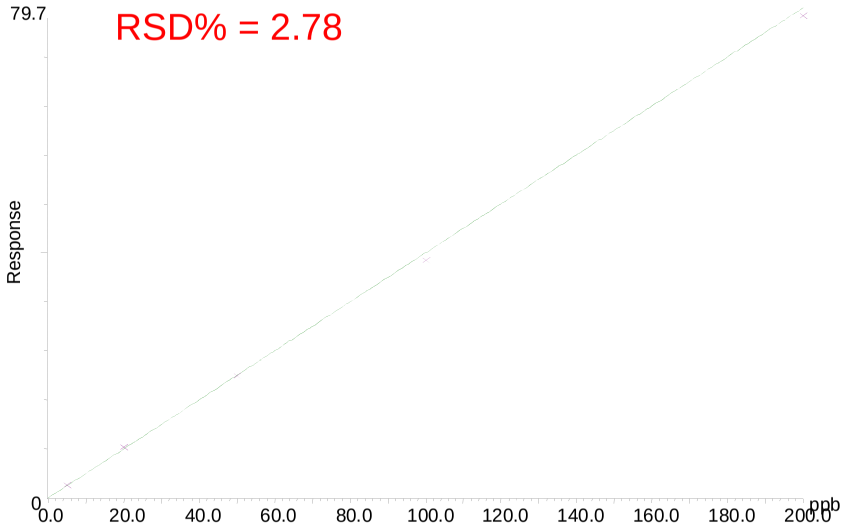
Save | Print Options | Print | Exit

Mass	Reference Mass	Relative Abundance	Criterion	Pass/Fail
			COMBINE(304:307)-(296)	Pass
50	95	16.6%	>= 15% and <= 40%	Pass
75	95	47.2%	>= 30% and <= 60%	Pass
95	BPI	100%	= 100%	Pass
96	95	6.6%	>= 5% and <= 9%	Pass
173	174	0.6%	< 2%	Pass
174	95	78.6%	> 50% and < 100%	Pass
175	174	6.4%	>= 5% and <= 9%	Pass
176	174	97.9%	> 95% and < 101%	Pass
177	176	6.2%	>= 5% and <= 9%	Pass

Example 8260 Calibration Data (5 – 200 ug/mL)

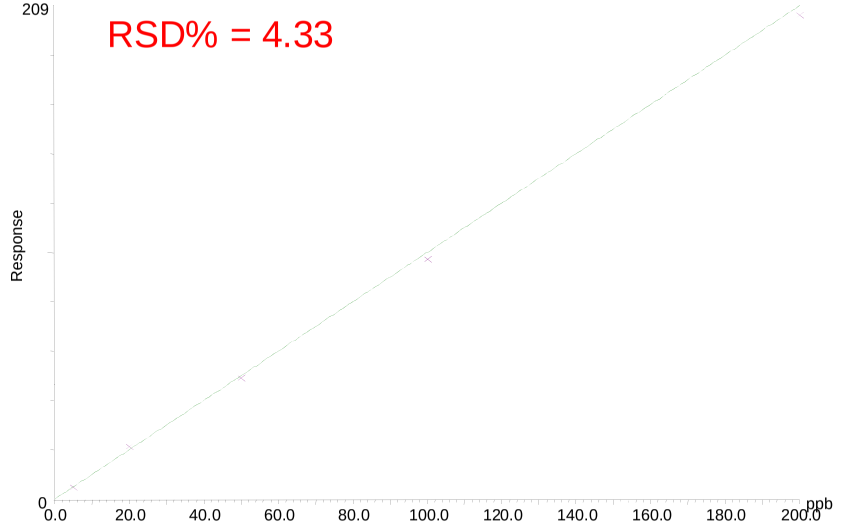
Compound 11 name: 1,1-Dichloroethene

RSD% = 2.78



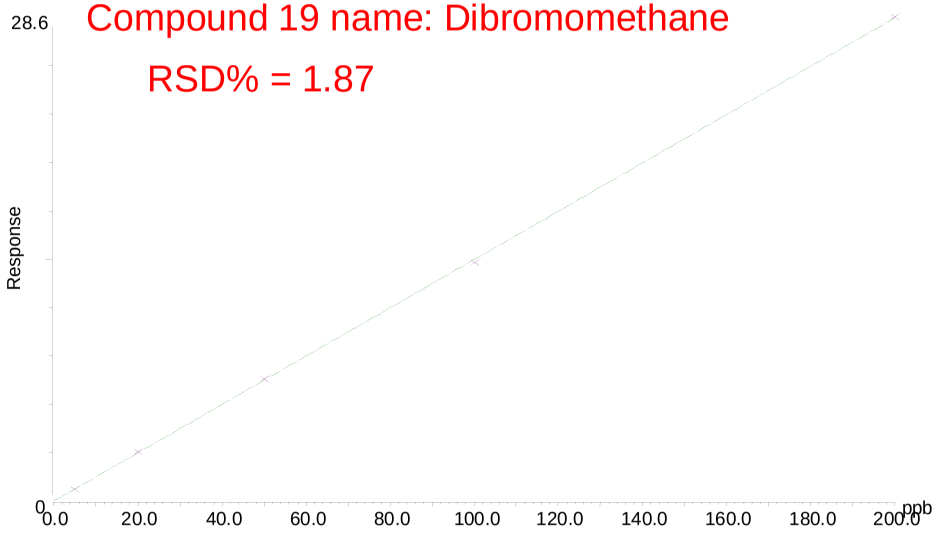
Compound 32 name: 1,3-Dichlorobenzene

RSD% = 4.33



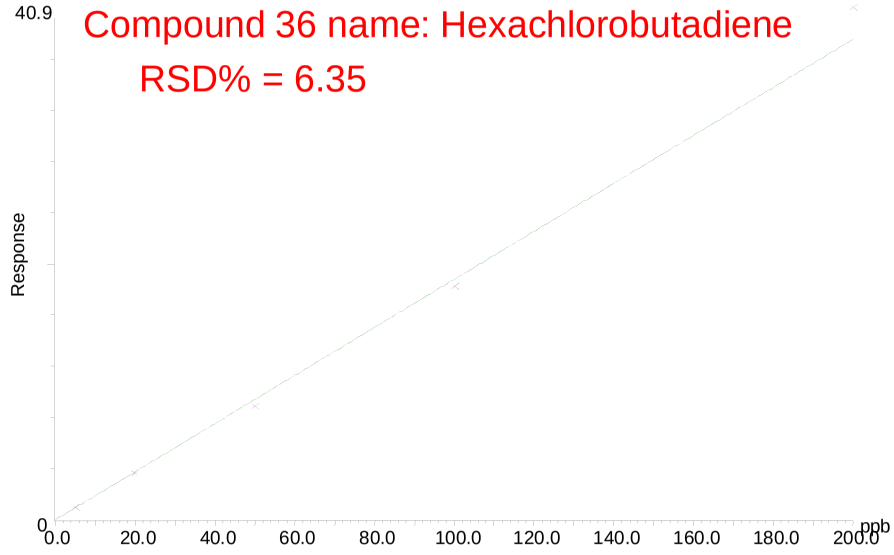
Compound 19 name: Dibromomethane

RSD% = 1.87

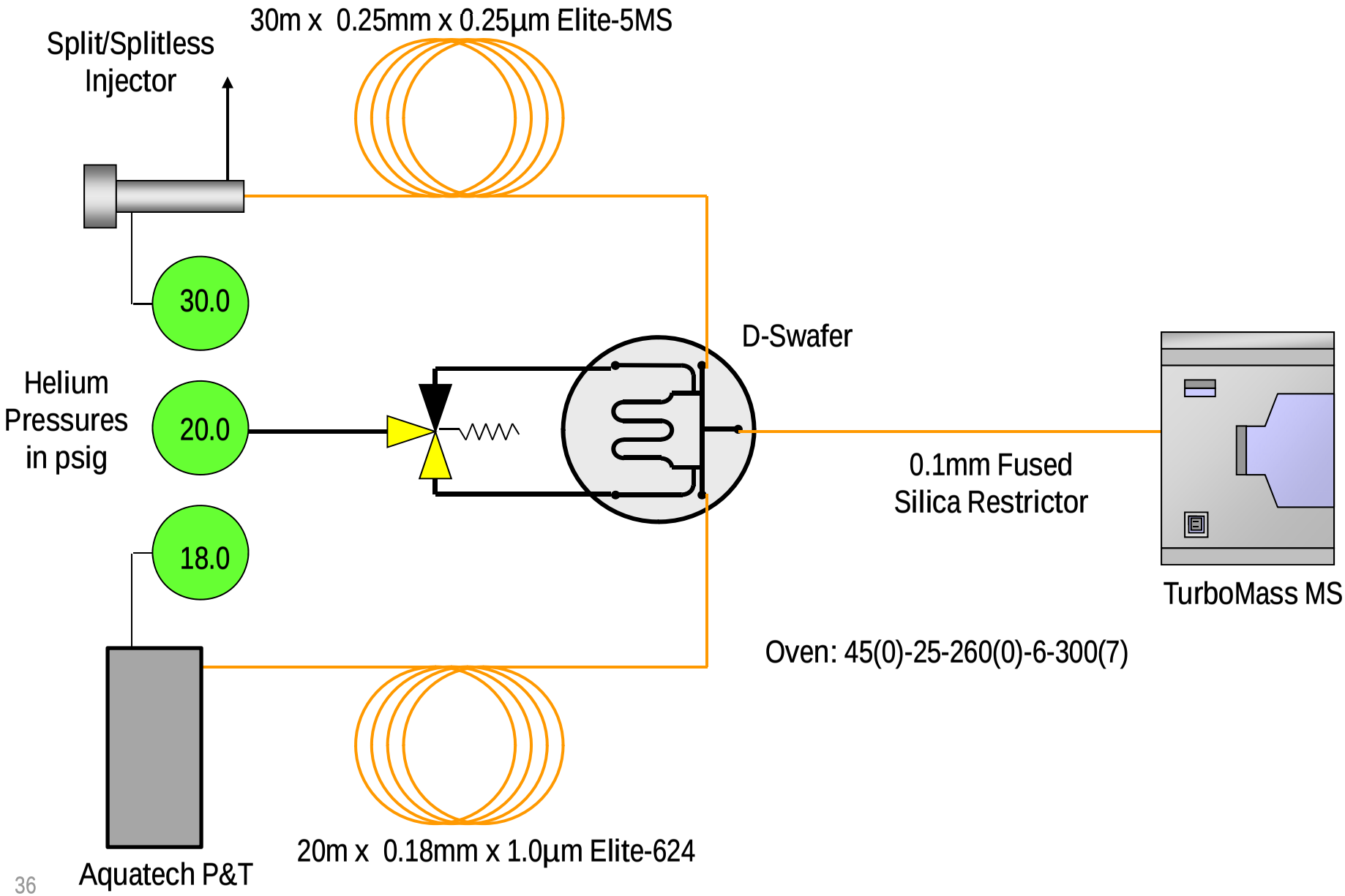


Compound 36 name: Hexachlorobutadiene

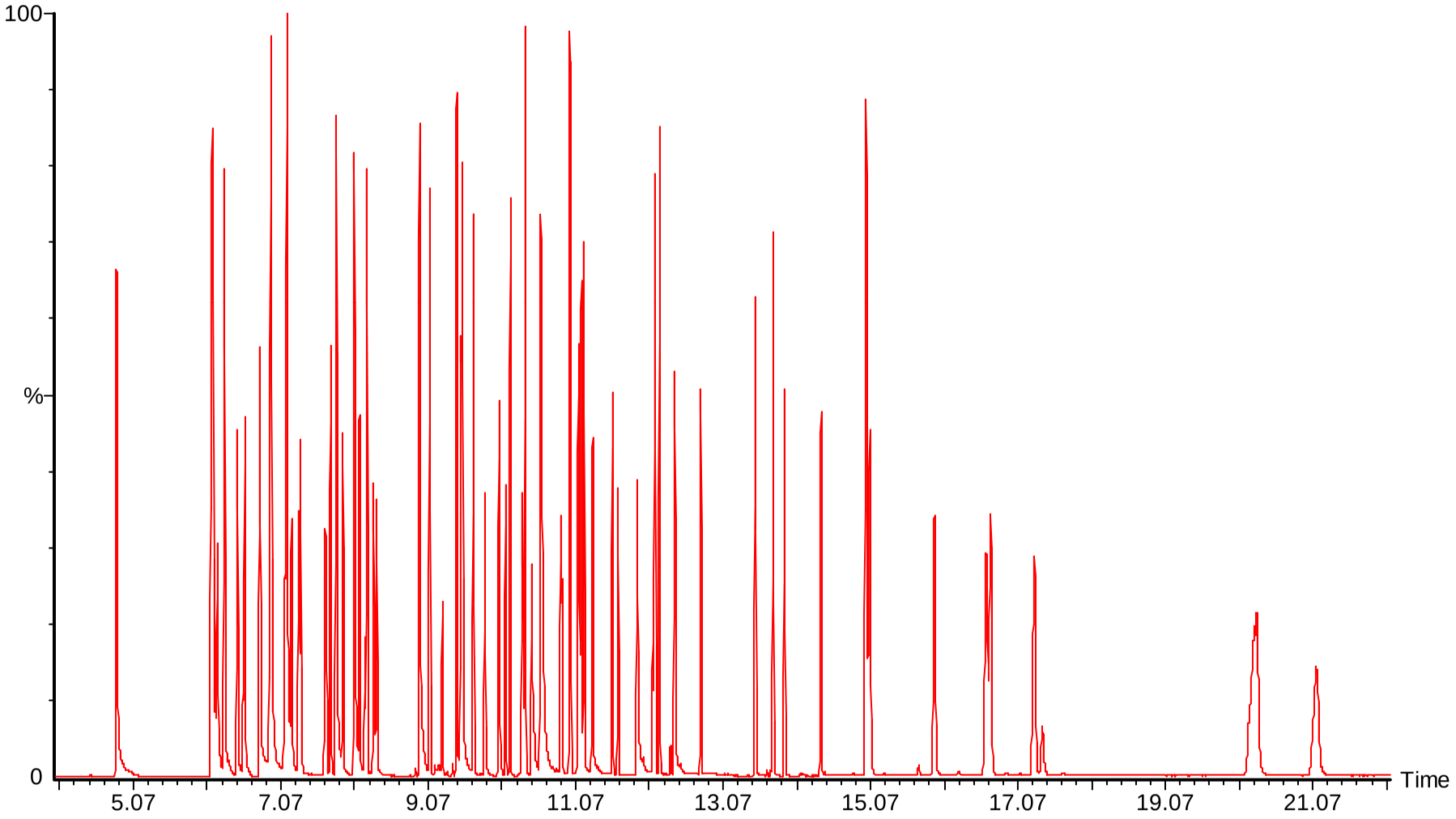
RSD% = 6.35



Conditions for 8270 Analysis

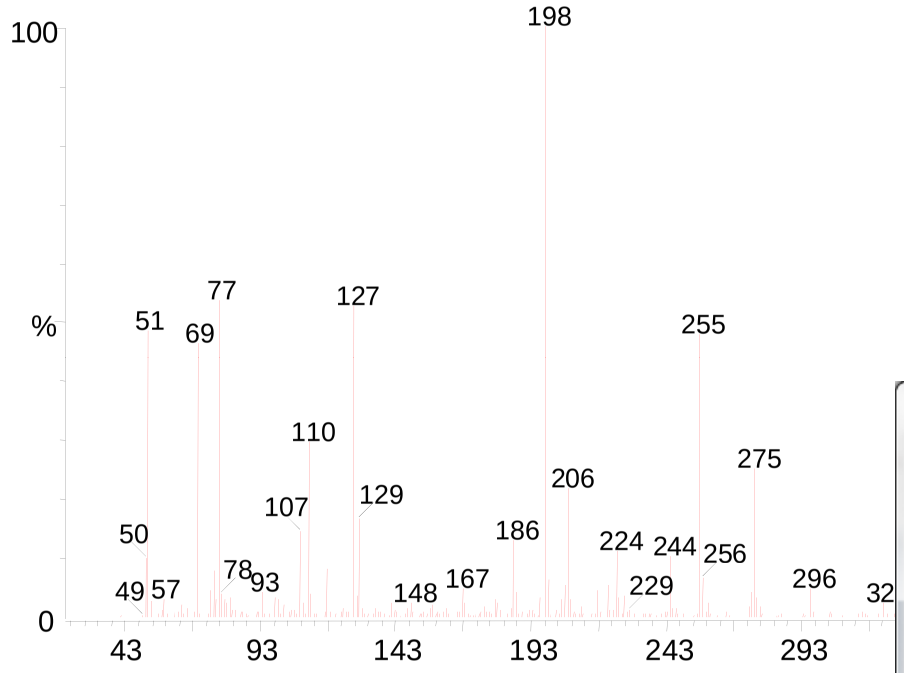


8270 Semivolatile Chromatogram



Decafluorotriphenylphosphine (DFTPP) Tune Evaluation

DFTPP)



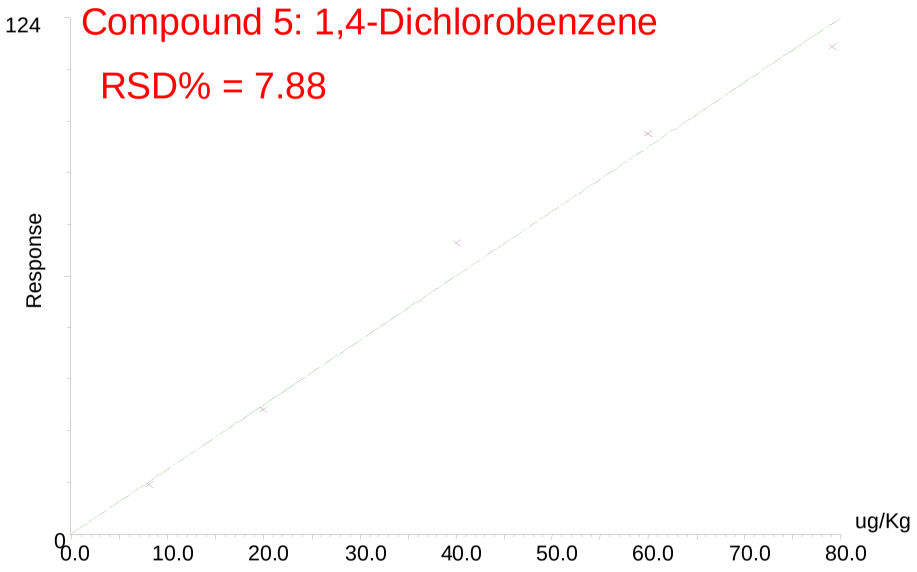
EPA Report

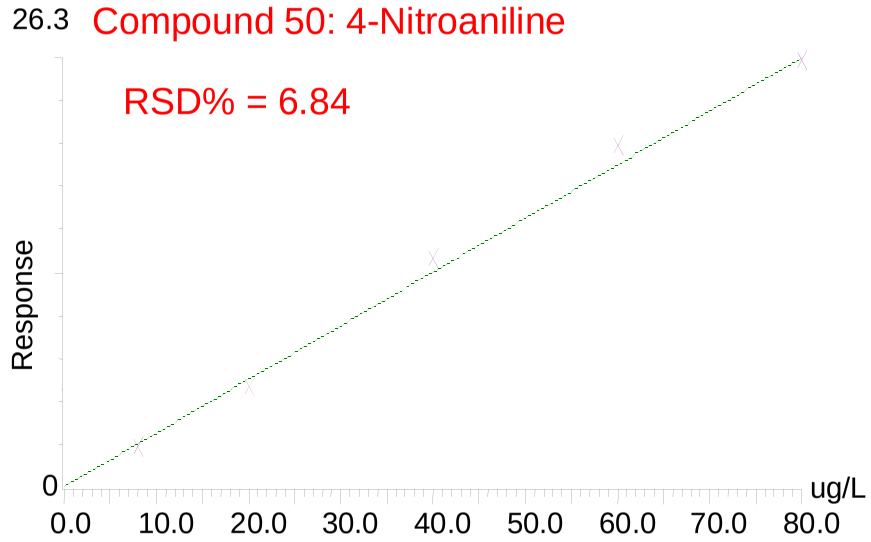
Test: DFTPP 625/8270 TEST Result: Test Passed Save

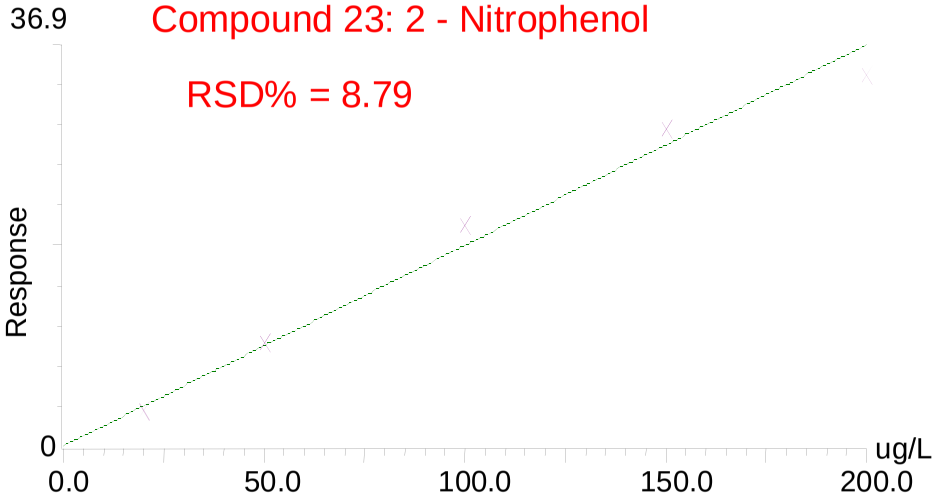
Mass	Reference Mass	Relative Abundance	Criterion	Pass/Fail
			COMBINE(2508:2513)-(2488)	Pass
51	198	46.3%	>= 30% and <= 60%	Pass
68	69	1.3%	< 2%	Pass
69	BPI	45.5%	NOT TESTED	Pass
70	69	0.5%	< 2%	Pass
127	198	52.1%	>= 40% and <= 60%	Pass
197	198	0%	< 1%	Pass
198	BPI	100%	= 100%	Pass
199	198	6.2%	>= 5% and <= 9%	Pass
275	198	24.1%	>= 10% and <= 30%	Pass
365	BPI	2.1%	> 1%	Pass
441	443	76.2%	< 100% and > 0	Pass
442	198	93.3%	< 100% and > 40%	Pass
443	442	19.4%	>= 17% and <= 23%	Pass

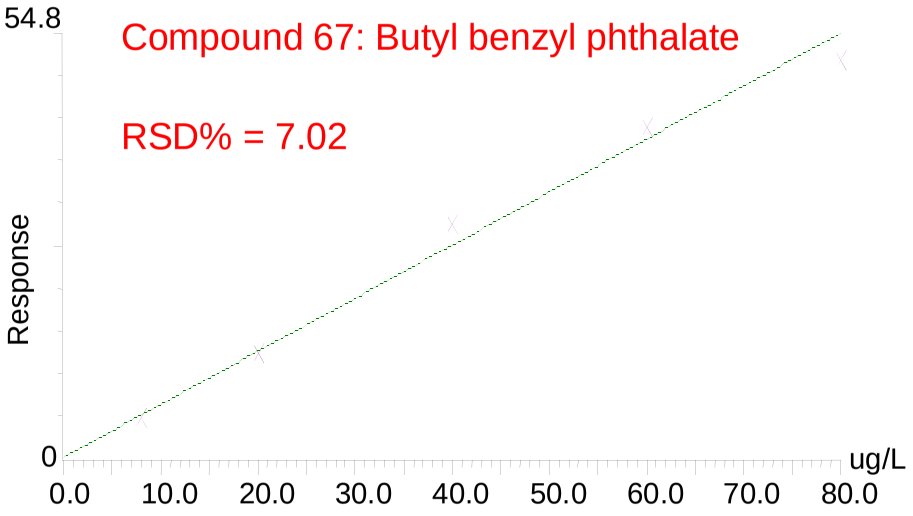
Print Options Print Exit

Semivolatile 8270 Calibration (10 – 80 ug/ml)









Conclusions

- The reversed heartcut switching technique based on this micro-channel wafer technology is a highly effective means of switching applications between a single MS as illustrated by these example environmental analyses.
- The technique reduces the need for multiple instruments to run multiple applications or saves hours of otherwise lost time in venting the detector, exchanging columns, etc.
- Analytical performance is not significantly affected.
- The system may also be used, without modification, to backflush late-eluting unwanted sample material from either or both channels to further save time and increase analytical throughput.