524.3 Purge Flow Study

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EST analytical

Optimize the analytical needs of businesses and industries around the world.

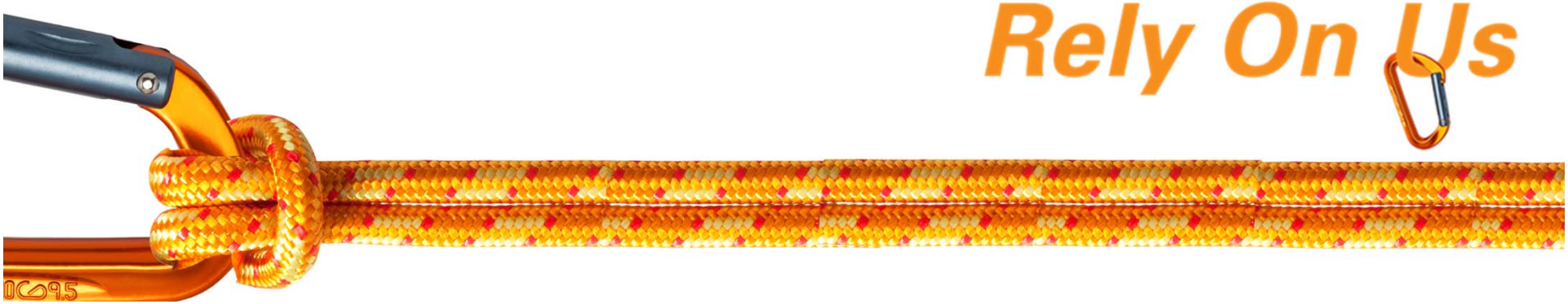
Overview

- Who is EST analytical?
- Abstract
- Experimental parameters examined
- Observations
- Conclusions
- Q&A

EST analytical

A Global Manufacturer of High Quality Sample Introduction Instruments

- Purge & Trap Experts
- Privately held
- Founded in 1990
- Environmental roots
- Full Support and Service



EST analytical

CENTURION
PURGE AND TRAP AUTOSAMPLER

The most reliable VOC autosampler on the market today

- IS 3% RSD
- No vial movement for water samples
- No lost vial, syringe or elevator errors
- Separate processing area for water and soil samples
- Rugged X, Y, Z engineering design

EPA Methods

502.1	524.2	601	624	5035	8010	8240
502.2	524.3	602		5030	8015	8260
	524.4	603			8020	
					8021	
					8030	

EVOLUTION
PURGE AND TRAP CONCENTRATOR

The most reliable VOC concentrator on the market today

- Superior moisture control (patented feature)
- Low carryover (patented feature)
- Easy maintenance & diagnostics
- Best in-class service & support
- 3 year warranty on electronic boards

EPA Methods

502.1	524.2	601	624	5035	8010	8240
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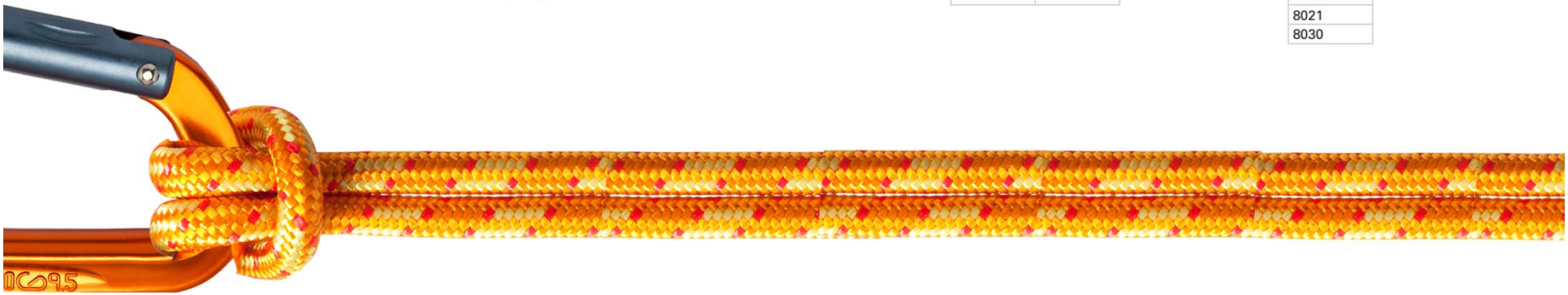


Abstract

During volatile analysis, samples are purged with an inert gas in order for the Volatile Organic Compounds (VOCs) to be swept out of the sample matrix and onto an analytical trap. For years, the established and recommended purge flow rate and time has been 40ml/min for 11 minutes.

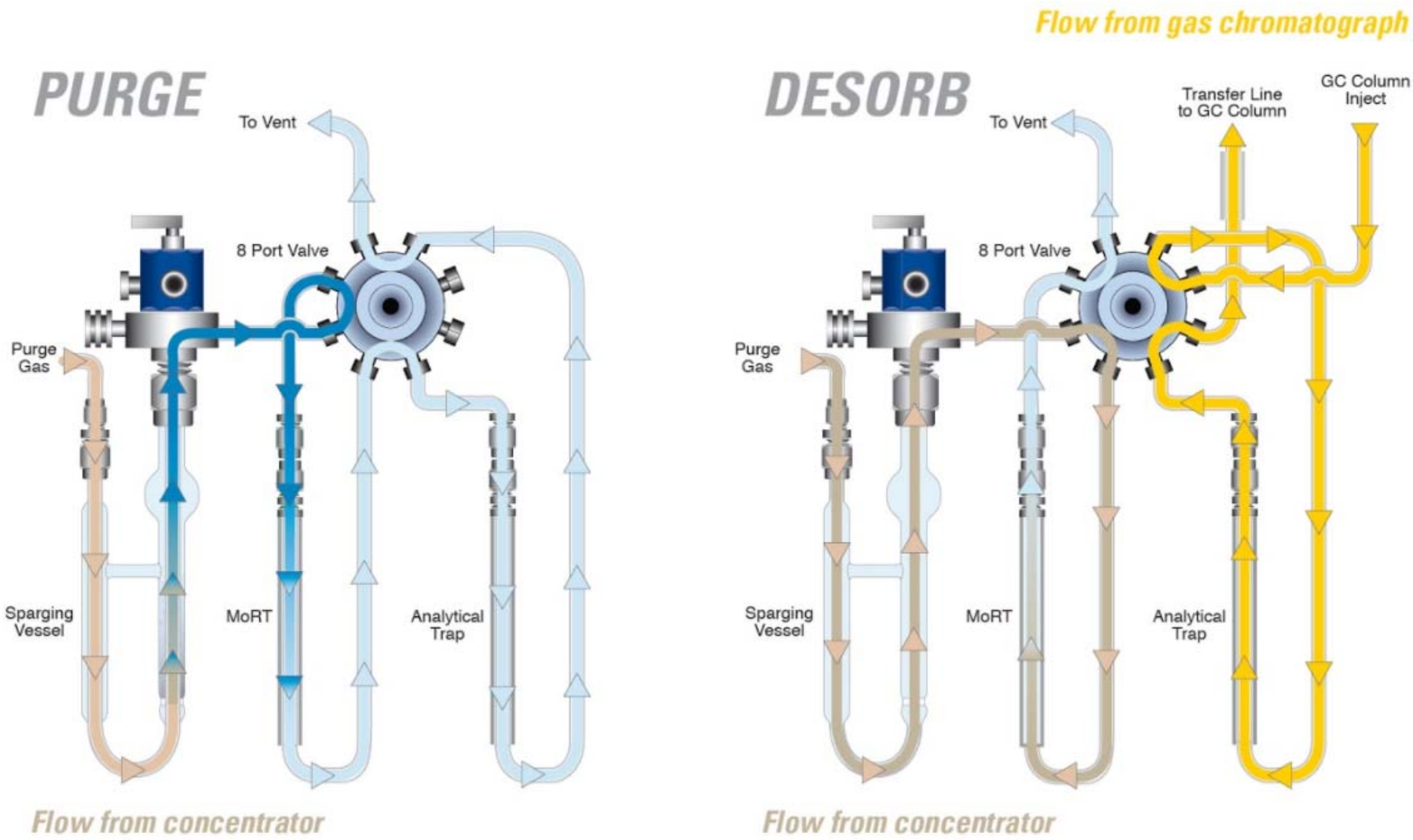
Why do the study?

1. EST analytical is a leader in Purge and Trap research and development.
2. EPA method 524.3 allows for different purge flows and times.
3. Evaluate the ability to shorten cycle times and run more samples.
4. Evaluate how purge times and flows can affect results.

Purge and Trap Steps

1. Standby
2. Purge
3. Dry purge
4. Desorb Preheat
5. Desorb
6. Trap Bake

This information is for scientific exchange only. EST analytical does not recommend or suggest methodology outside of published method parameters.


PURGE
To Vent
8 Port Valve
Purge Gas
Sparging Vessel
MoRT
Analytical Trap
Flow from concentrator
Flow from gas chromatograph
DESORB
To Vent
Transfer Line to GC Column
GC Column Inject
8 Port Valve
Purge Gas
Sparging Vessel
MoRT
Analytical Trap
Flow from concentrator

Purge Flow Rate

Defined as:

- The rate in mL/min that the purge gas flows through the sparge vessel during the purge cycle
- Current flow and time are 40mL/min for 11min

Purge Flow Study

- Current purge volume is 480mL, how does decreasing the purge volume affect the compound results?
- Current purge flow is 40mL/min, how does increasing the purge flow affect the compound results?

USEPA Method 524.3

Parameter	Recommended		Allowable	
	Min.	Max.	Min.	Max.
Purge Flow Rate	40mL/min	80mL/min	20mL/min	200mL/min
Purge Volume	360mL	520mL	240mL	680mL
Purge Volume + Dry Purge Volume	360mL	720mL	240mL	880mL

Purge Flow Study

Parameter	Experiment 1 (Baseline)	Experiment 2	Experiment 3	Experiment 4
Purge Flow Rate	40mL/min	65mL/min	100ml/min	65mL/min
Purge Time	11min	6.5min	4min	4min
Purge Volume	440mL	422.5mL	400mL	260mL
Purge Volume + Dry Purge Volume	480mL	462.5mL	440mL	300mL

GC/MS Parameters

GC/MS	GC/MS
Inlet	Split/Splitless
Inlet Temp.	220ºC
Inlet Head Pressure	12.153 psi
Mode	Split
Split Ratio	40:1
Column	30m x 0.25mm I.D. 1.4µm film thickness (624)
Oven Temp. Program	45ºC hold for 1 min, ramp 15ºC/min to 220ºC, hold for 1.33 min, 14 min run time
Column Flow Rate	1mL/min
Gas	Helium
Total Flow	44mL/min
Source Temp.	230ºC
Quad Temp.	150ºC
MS Transfer Line Temp.	180ºC
Scan Range	m/z 35-300
Scans	5.2 scans/sec
Solvent Delay	0.7 min

Evolution Purge and Trap Parameters

Purge and Trap Concentrator	EST Analytical Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	130ºC
Transfer Line Temp.	130ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp.	39ºC
Purge Time	Varied
Purge Flow	Varied
Dry Purge Temp.	ambient
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	5psi
Desorb Time	0.5 min
Desorb Preheat Delay	10 sec
Desorb Temp.	250ºC
Moisture Reduction Trap (MoRT) Bake Temp.	210ºC
Bake Temp	260ºC
Sparge Vessel Bake Temp.	110ºC
Bake Time	6 min
Bake Flow	85mL/min

Centurion Autosampler Parameters

Purge and Trap Auto-Sampler	EST Centurion WS
Sample Type	Water
Water Volume	5ml
Sample Prime Time	7 sec
Loop Equilibration Time	5 sec
Sample Transfer Time	10 sec.
Syringe Rinse	On/10 ml
Number of Syringe Rinses	2
Sample Loop Rinse	On/10 sec
Sample Sweep Time	5 sec
Number of Sparge Rinses	On/2
Rinse Volume	5 ml
Rinse Transfer Time	10 sec
Rinse Drain Time	15 sec
Water Heater Temp.	85 sec
Internal Standard Vol.	5 µl

Standard Preparation

524.3 Standard at 200ppm			
Amount	**Standard**	**Concentration**	**Final Vol.**
200ul	524.3 VOA Mega Mix	2000ug/ml	2ml
200ul	524.3 Gas Cal Mix	2000ug/ml	2ml
200ul	Iodomethane	2000ug/ml	2ml

Use 2ml volumetric flask and dilute standards to 2.0ml in purge and trap methanol

524.3 Standard at 20ppm

200µl of 200ppm standard diluted to 2.0ml in purge and trap methanol

524.3 Internal Standard/Surrogate Standard at 5ppm			
Amount	**Standard**	**Concentration**	**Final Vol.**
5µl	524.3 IS/SS Mix	2000 ug/ml	2.0ml

Use 2ml volumetric flask and dilute standards to 5.0ml in purge and trap methanol

Calibration Curve Preparation

524.3 Curve			
Concentration	**Standard**	**Standard Amount**	**Final Vol.**
0.5ppb	20ppm	2.5µl	100ml
1ppb	20ppm	5µl	100ml
2ppb	20ppm	10µl	100ml
5ppb	20ppm	25µl	100ml
10ppb	200ppm	5µl	100ml
20ppb	200ppm	10µl	100ml
40ppb	200ppm	20µl	100ml

Calibration Standards

Fill 40ml Vial with final standard leaving no headspace in the vial.

Preserved Water Preparation

Preservation of Water		
Preservative	**Mass (mg)**	**Final Volume (ml)**
Ascorbic Acid	25	40
Maleic Acid	200	40
Ascorbic Acid	625	1000
Maleic Acid	5000	1000

Experimental

Baselined the system:

- Prepared a calibration curve and ran it using 40mL/min for 11 minute purge parameters
- Ran method detection limit and precision and accuracy samples
- Compiled all of the data for comparison

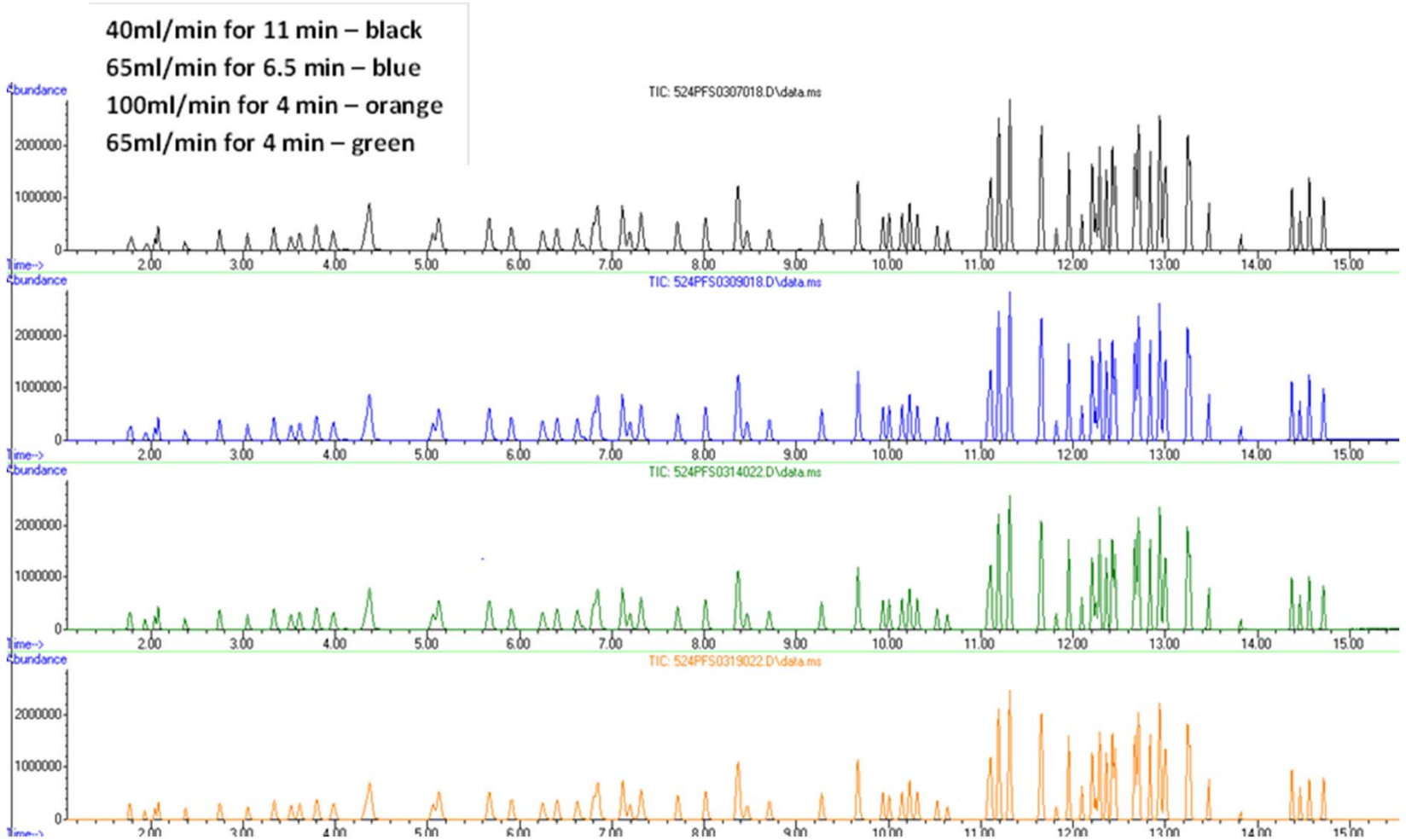
Experimental

- Prepared and ran calibration curves, MDLs and precision and accuracy samples for each of the purge flows and times outlined
- Compiled all of the data from each purge flow study

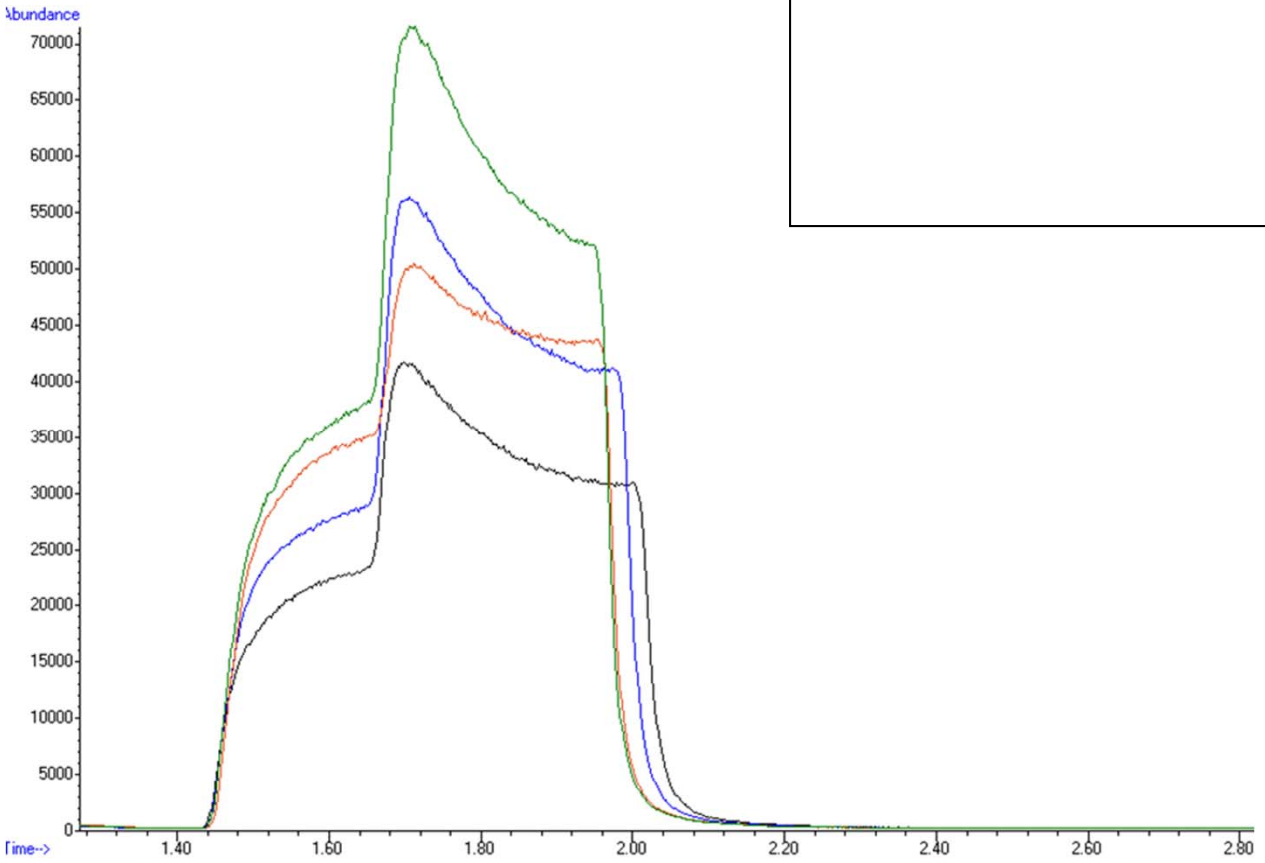
Results

Purge Flows and Times	Avg. Curve Quadratic Regression	Avg. Curve RF	Avg. Upper PIR (prediction interval of results) (0.5µg/L)	Avg. Lower PIR (prediction interval of results (0.5µg/L)	Avg. % Recovery (20µg/L)	Avg. Precision, %RSD (20µg/L)
Baseline	1.000	1.110	110.23	81.65	103.57	4.03
65ml/min for 6.5 min	0.999	1.220	116.53	83.31	95.11	4.66
100ml/min for 4 min	0.999	1.273	105.73	68.57	95.85	5.22
65ml/min for 4 min	0.999	1.001	112.79	76.89	105.27	2.82

20ppb Chromatograms



Water Abundance Overlay



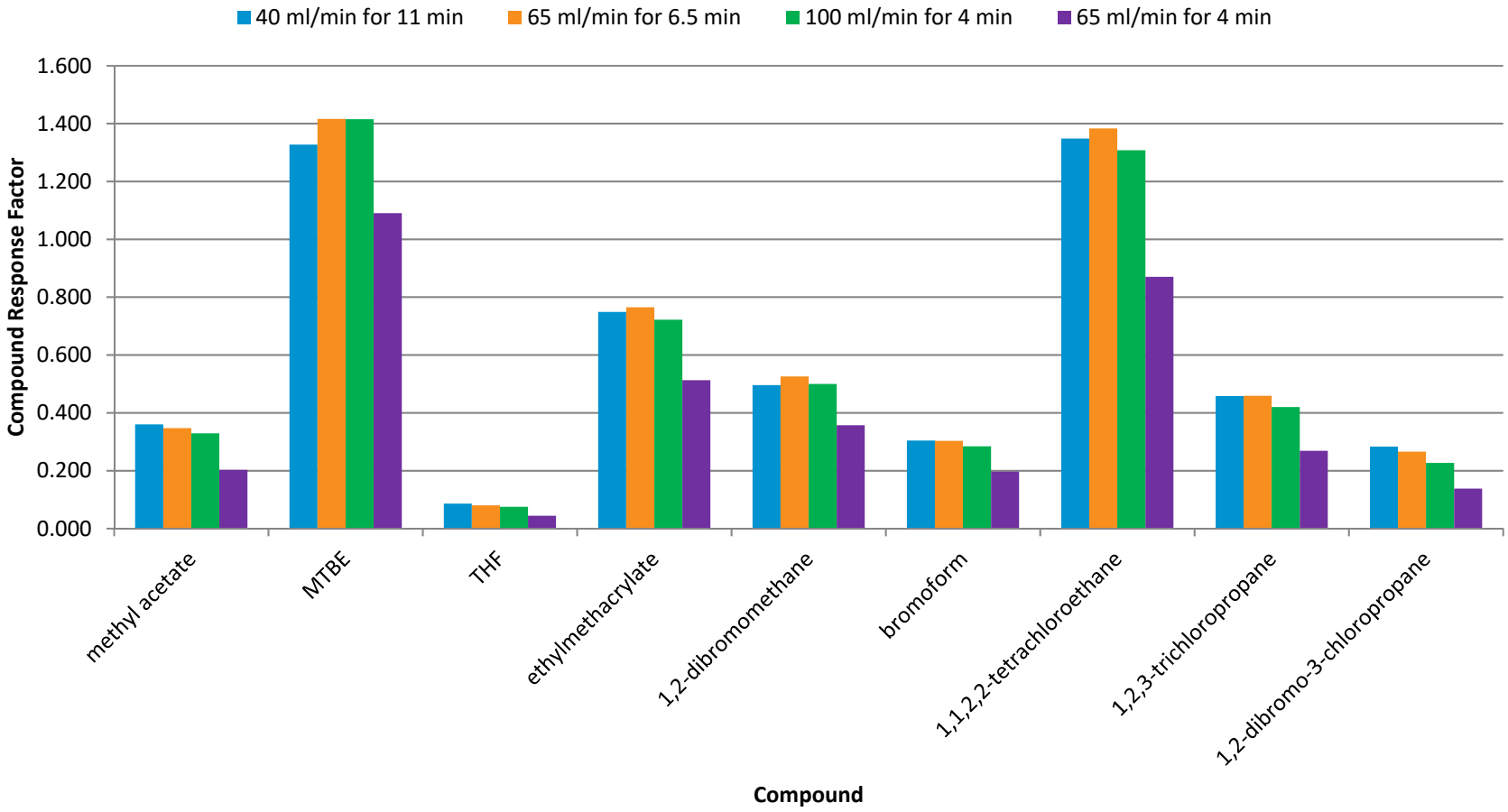
Outlier Prediction Interval Summary

Compound	40 ml/min for 11 min		65 ml/min for 6.5 min		100 ml/min for 4 min		65 ml/min for 4 min	
	Upper PIR	Lower PIR	Upper PIR	Lower PIR	Upper PIR	Lower PIR	Upper PIR	Lower PIR
bromomethane	96.36	63.07	103.14	54.57	111.26	75.60	94.79	41.21
methyl acetate	121.49	77.93	100.19	81.52	113.22	68.50	122.68	93.32
MTBE	120.98	66.45	130.63	112.23	109.33	70.10	103.59	87.26
THF	146.41	53.59	106.41	73.59	208.46	98.40	146.39	82.18
ethylmethacrylate	116.02	101.70	121.26	98.17	89.95	67.76	119.12	87.74
1,2-dibromomethane	100.41	78.45	114.05	85.95	101.47	66.53	133.85	95.29
bromoform	117.55	95.59	132.06	89.09	90.30	75.98	116.46	94.97
1,1,2,2-tetrachloroethane	121.77	103.37	122.39	85.04	99.97	64.60	136.82	108.90
1,2,3-trichloropropane	108.46	86.97	108.41	86.45	124.71	58.15	141.93	87.21
1,2-dibromo-3-chloropropa	129.11	88.60	123.33	84.10	114.15	24.14	131.75	47.68

Response Factor Outlier Summary

Compound	40ml/min for 11 min	65 ml/min for 6.5 min	100 ml/min for 4 min	65 ml/min for 4 min
methyl acetate	0.360	0.347	0.329	0.203
MTBE	1.327	1.416	1.415	1.090
THF	0.086	0.080	0.075	0.044
ethylmethacrylate	0.749	0.765	0.722	0.513
1,2-dibromomethane	0.496	0.526	0.500	0.357
bromoform	0.304	0.303	0.284	0.197
1,1,2,2-tetrachloroethane	1.348	1.383	1.308	0.870
1,2,3-trichloropropane	0.458	0.459	0.420	0.269
1,2-dibromo-3-chloropropane	0.283	0.266	0.227	0.138

Response Factor Results Bar Graph



Conclusions

- The effect of purge flow and purge volume can be significant
- The EPA recommended purge flow of 40ml/min for 11 minutes displayed the least amount of water contamination while the 100ml/min showed the most
- The 65ml/min flow had similar amounts of water even though the purge times/volumes were different

Conclusions

- The 65ml/min for 4 minutes produced and overall purge volume of 300ml when taking the dry purge into account. This lower purge volume affected the compound responses of polar compounds and the lower purge response compounds

Conclusions

- The optimum purge flow and time, when compared with the recommended 40ml/min for 11 minutes, were 65ml/min for 6.5 min
- The 65ml/min flow was slow enough not to cause water problems and the 6.5min purge time along with the dry purge provided a 462.5 ml purge volume which aided in compound response

Thank You

Questions

Information