Reducing Carryover in Environmental Water Samples

Application Note

Environmental

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Abstract

Carryover is a common problem resulting in sample reruns and reduced productivity in environmental labs. Many advances over the years have been developed to tackle this issue. These innovations vary from increasing bake times and bake flows to changing the flow path tubing to more inert materials. However, all these improvements do nothing to reduce the most common source of carryover, the sparge vessel. This application will evaluate a patented innovation for cleaning the sparge vessel during an analytical sequence.

Introduction:

In order to mitigate carryover, many environmental labs resort to running sample dilutions or adding blank samples after a highly contaminated sample. Both of these solutions are not ideal and can lead to a loss of analyte detection when diluting or a loss of profits when blank samples need to be run

Since, the primary source of carryover for water samples is the sparge vessel, purge and trap manufactures have incorporated a rinsing step in which the sparge vessel is "washed" with hot water in order to clean up the volatile compounds that may still reside in the sparge vessel. Expanding on the idea of limiting volatile compounds in the sparge vessel, EST Analytical developed a patented process of heating the sparge vessel during the bake step of the purge and trap process (Patent Number: US 8,075,842 B1) . This engineering innovation essentially performs two bakes; as a result both the trap and the sparge vessel are "cleaned" at the same time. No other concentrator is or can be engineered to do this.

Experimental:

The sampling system used for this study was the EST Analytical Evolution concentrator and the Centurion WS autosampler. The concentrator was affixed with a Vocarb 3000 trap and connected to an Agilent 7890A GC and 5975C inert XL MS. The GC was configured with a Restek Rxi-624 Sil MS 30m x 0.25mm x 1.4μm column. Refer to Table 1 for the sampling method parameters and Table 2 for GC/MS parameters.

Purge and Trap Concentrator	EST Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	150°C
Transfer Line Temp.	150°C
Trap Temp.	35°C
Moisture Reduction Trap (MoRT) Temp.	39°C
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	6psi
Desorb Time	0.5 min
Desorb Preheat Delay	15 sec
Desorb Temp.	260°C
Moisture Reduction Trap (MoRT) Bake Temp.	210°C
Bake Temp	270°C
Sparge Vessel Bake Temp.	120°C
Bake Time	8 min
Bake Flow	85mL/min
Purge and Trap Auto-Sampler	**EST Centurion WS**
Sample Type	Water
Water Volume	5ml
Internal Standard Vol.	5µl

Table 1: Purge and Trap Parameters

GC/MS	Agilent 7890A/5975C inert XL
Inlet	Split/Splitless
Inlet Temp.	220°C
Inlet Head Pressure	12.153 psi
Mode	Split
Split Ratio	40:1
Column	Rxi-624Sil MS 30m x 0.25mm I.D. 1.4µm film thickness
Oven Temp. Program	45°C hold for 1 min, ramp 15°C/min to 220°C, hold for 1.33 min, 14 min run time
Column Flow Rate	1mL/min
Gas	Helium
Total Flow	44mL/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 35-300
Scans	5.2 scans/sec
Solvent Delay	0.7 min

Table 2: GC/MS Experimental Parameters

A calibration curve was established with a linear range of 0.5 to 200ppb using USEPA Method 8260 standards from Restek. After the curve was determined, a series of ten 200ppb standards were run, each standard was followed by three blanks. The sparge vessel was "baked" out at 120°C after each sample in order to limit the carryover. Using the compound responses from the first blank following the 200ppb standard, percent carryover was calculated. The carryover data was then compared with published Atomx[1] data employing a hot water rinse to clean the sparge vessel.

Compound	Ave. 200ppb Area Count	Ave. Blank One Carryover Area Count	Ave. % Carryover
Benzene	5918681	2805	0.05
Toluene	3872665	1943	0.05
Ethylbenzene	8015734	4120	0.05
p&m-Xylene	12605336	6903	0.06
o-Xylene	6237140	3084	0.05
1,2,4-Trichlorobenzene	2370293	5891	0.25
Naphthalene	7009924	19673	0.28
Hexachlorobutadiene	883220	2415	0.27
1,2,3-Trichlorobenzene	2257852	6115	0.27

Table 3: Percent Carryover Results for Some of the Volatile Compounds

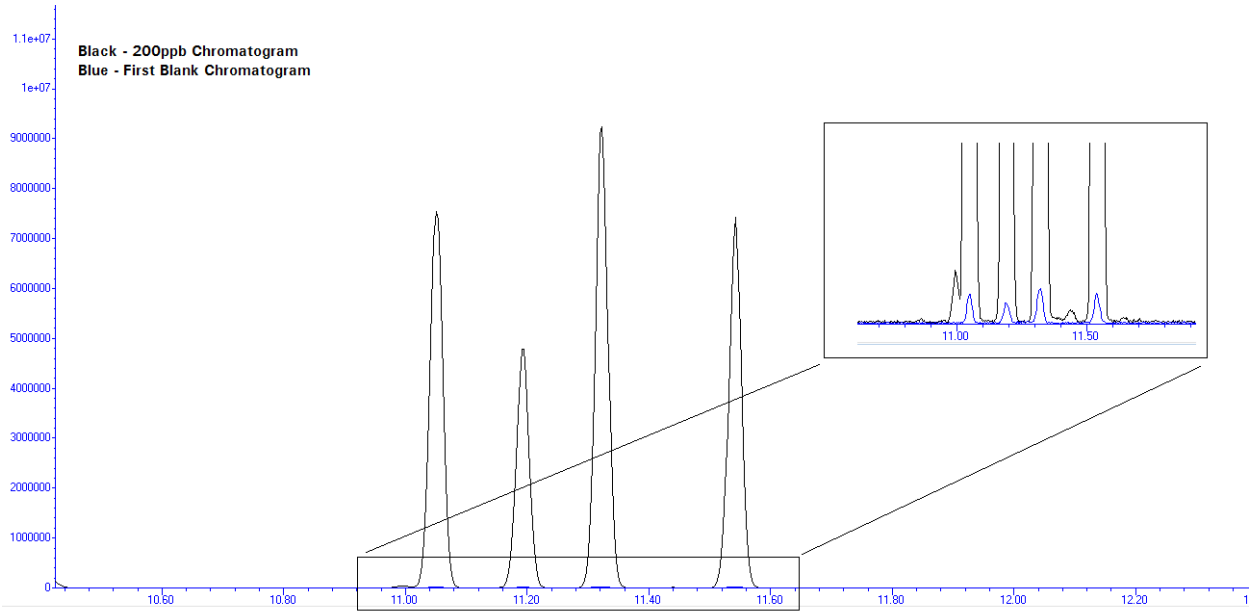

Figure 1: Carryover of the Late Eluting Volatile Analytes

Compound	Atomx % Carryover Hot Water Rinse Sparge Vessel[1]	Evolution % Carryover Hot Water Rinse with Sparge Vessel Bake
1,2,4-Trichlorobenzene	0.50	0.25
Naphthalene	0.45	0.28
Hexachlorobutadiene	0.31	0.27
1,2,3-Trichlorobenzene	0.40	0.27

Table 4: Carryover Comparison Table

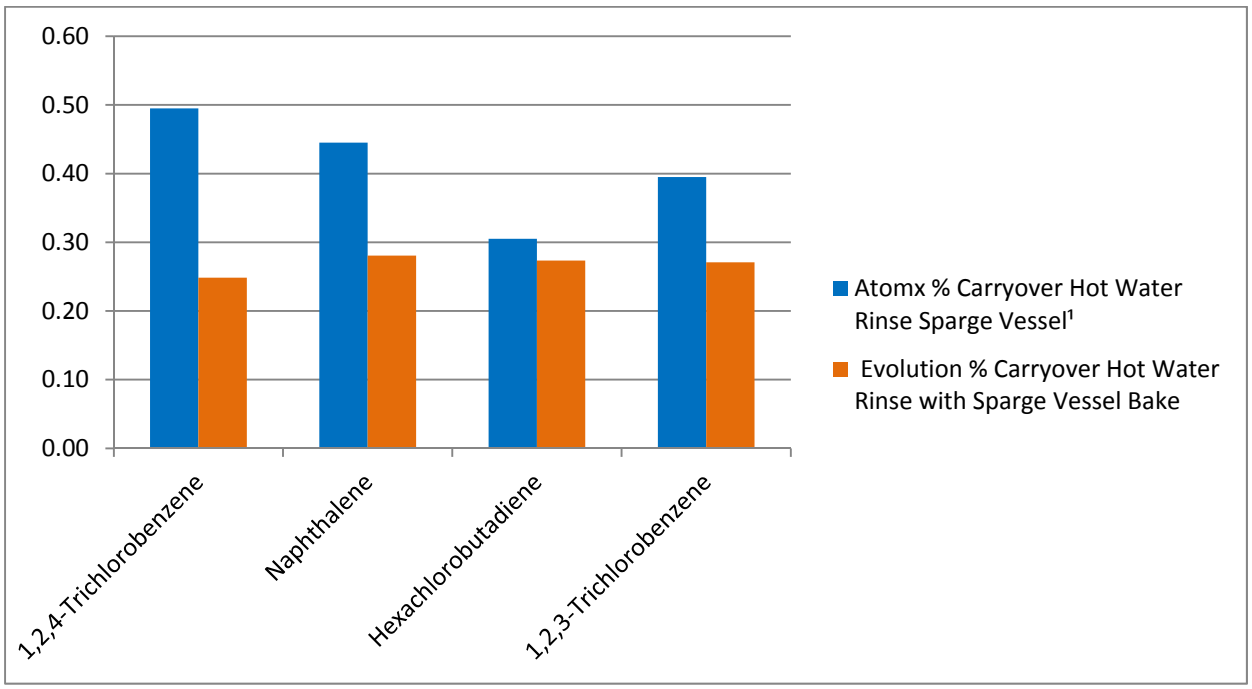

Figure 2: Carryover Comparison Graphic

Conclusions:

The Evolution purge and trap concentrator with its patented technique of heating the sparge vessel during the bake process proved to reduce carryover by almost half when comparing with hot water rinses alone. The advantages of reduced carryover are many. Primary among them is the ability to run more samples during the twelve hour tune window. This increase in productivity translates to more laboratory profits and better use of instrument time.

References:

1. *Analytical Trap Comparison for USEPA Method 8260C,* Teledyne Tekmar, February 2012.

For More Information

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