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Requirements of an Automated Sample Delivery System in Today's Realm of Ever Increasing Sensitivity Demands Utilizing the Atomx Concentrator/Multimatrix Autosampler.

Application Note

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Abstract

As today's instrumentation and experimental techniques improve on sensitivity and efficiency, we must also provide better ways to decrease carryover and ensure the instrumentation is running at its optimum capability. In this study, data was collected to evaluate carryover of target analytes in subsequent blanks following high concentration samples. The sample analysis was automated using the Atomx, Teledyne Tekmar's new robotic autosampler in combination with a Purge and Trap (P&T) concentrator. The Atomx was interfaced to a Gas Chromatograph/Mass Spectrometer (GC/MS) system for quantitative analysis. This investigation will consider the effectiveness of different rinse solutions, temperatures, volumes, and frequency.

Introduction

Teledyne Tekmar has consistently been at the cutting edge of P&T technology. In keeping with this tradition, Teledyne Tekmar has developed a new combination P&T concentrator(PTC)/Autosampler, the Atomx. The Atomx was developed to fully automate water, soil, and methanol extraction in accordance with the USEPA methods for volatile analyses. The Atomx offers our proprietary #9 U-shaped adsorbent trap, as seen with our Stratum PTC. The Autosampler utilizes an 80 position carousel drive platform for proven reliability. The Atomx offers a reduced sample pathway, Siltek™ coated stainless tubing, and PEEK® tubing to aid in the reduction of carryover. What makes the Atomx unique is the option to not only to rinse the entire sample pathway with hot water, but also the capability to rinse the sample pathway in methanol (patent pending).

In this study, data was collected to evaluate carryover of target analytes in water and soil matrices utilizing the Atomx. The effectiveness of the different rinse options available were evaluated for each matrix. The Atomx proved to be an excellent system in minimizing carryover of target analytes.

Experimental-Instrument Conditions

The Atomx, an Agilent 7890A GC and a 5975C inert XL MSD were used for this analysis. The Atomx was equipped with a #9 adsorbent trap. Tables 1 and 2 display the GC, MSD conditions while Tables 3 and 4 display the PTC/Autosampler conditions for water and soil matrices respectively.

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GC Parameters	
GC:	Agilent 7890A
Column:	J&W Scientific DB-VRX 30m x 0.250mm x1.4um
Oven Program:	35°C for 4 min; 16°C/min to 85°C for 0 min; 30°C /min to 210°C for 3 min, 14.29 min runtime
Inlet:	220°C
Column Flow	1.02mL/min
Gas:	Helium
Split:	80:1
Pressure:	20.14 psi
Inlet	Split/Splitless

MSD Parameters	
MSD:	5975C Inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Scan Range:	m/z 35-300
Scans:	5.19 scans/sec
Threshold:	400
MS Transfer Line Temp.	230°C

Tables 1 & 2: GC and MSD Parameters

Atomx Water Parameters			
Variable	**Value**	**Variable**	**Value**
Valve Oven Temp	140°C	Dry Purge Flow	100mL/ min
Transfer Line Temp	140°C	Dry Purge Temp	20°C
Sample Mount Temp	90°C	Methanol Needle Rinse	Varied
Water Heater Temp	90°C	Methanol Needle Rinse Volume	3.0mL
Sample Vial Temp	20°C	Water Needle Rinse Volume	7.0mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.50 min
Soil Valve Temp	125°C	Desorb Preheat Temp	245°C
Standby Flow	10mL/ min	GC Start Signal	Start of Desorb
Purge Ready Temp	40°C	Desorb Time	2.00 min
Condensate Trap Standby	45°C	Drain Flow	300mL/min
Presweep Time	0.50 min	Desorb Temp	250°C
Prime Sample Fill Volume	3.0mL	Methanol Glass Rinse	Varied
Sample Volume	5.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	Varied
Sweep Sample Flow	100mL/min	Number Of Bake Rinses	Varied
Sparge Vessel Heater	Off	Water Bake Rinse Volume	7.0mL
Sparge Vessel Temp	20°C	Bake Rinse Sweep Time	0.25 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100mL/min
Prepurge Flow	0mL/min	Bake Rinse Drain Time	0.40 min
Purge Time	11.00 min	Bake Time	4.00 min
Purge Flow	40mL/min	Bake Flow	200mL/min
Purge Temp	20°C	Bake Temp	280°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	1.00 min		

Table 3: Atomx Water Parameters (Parameters highlighted in green were not used.)

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Atomx Soil Parameters			
Variable	**Value**	**Variable**	**Value**
Valve Oven Temp	140°C	Purge Time	11.0 min
Transfer Line Temp	140°C	Purge Flow	40mL/min
Sample Mount Temp	90°C	Purge Temp	20°C
Water Heater Temp	90°C	Condensate Purge Temp	20°C
Sample Vial Temp	40°C	Dry Purge Time	1.00 min
Prepurge Time	0.00 min	Dry Purge Flow	100mL/ min
Prepurge Flow	0mL/min	Dry Purge Temp	20°C
Preheat Mix Speed	Off	Methanol Needle Rinse	Varied
Sample Preheat Time	2.00 min	Methanol Needle Rinse Volume	3.0mL
Soil Valve Temp	125°C	Water Needle Rinse Volume	7.0mL
Standby Flow	10mL/min	Sweep Needle Time	0.50 min
Purge Ready Temp	40°C	Desorb Preheat Temp	245°C
Condensate Temp Standby	45°C	GC Start Signal	Start of Desorb
Presweep Time	0.50 min	Desorb Time	2.00 min
Water Volume	10mL	Drain Flow	300mL/min
Sweep Water Time	0.25 min	Desorb Temp	250°C
Sweep Water Flow	100mL/min	Bake Time	4.00 min
Sparge Vessel Heater	Off	Bake Flow	200mL/min
Sparge Vessel Temp	20°C	Bake Temp	280°C
Purge Mix Speed	Medium	Condensate Bake Temp	200°C

Table 4: Atomx Soil Parameters (Parameters highlighted in green were not used.)

Calibration

A 50ppm working calibration standard was prepared in methanol. Calibration standards were prepared in 50 milliliter (mL) volumetric flasks filled to volume with de-ionized water. The water calibration range was 0.5-200ppb while the soil range was 1.0-200ppb. The water standards were transferred to headspace free 40mL vials for analysis while the soil standards were transferred to 40mL vials in 5mL aliquots. A 25ppm Internal Standard (IS) was prepared in methanol and transferred to the standard vessel on the Atomx. The Atomx, using the standards addition feature, then transferred the IS in 5µl aliquots to the samples in order to hold the IS concentration at a constant 25ppb.

The calibration data was processed using Agilent Chemstation software. The relative response factors of all of the analytes were evaluated for %RSD or for linearity. All of the analytes in the water and soil calibration curves met the USEPA Method 8260 performance criteria.

Carryover Evaluation

Carryover was evaluated by running three blanks after a 200ppb calibration standard for both the soil and the water matrices. The carryover for the soil and water matrix was evaluated utilizing the first blank after the 200ppb calibration standard for that matrix. Different rinse options were then evaluated to assess its effectiveness in limiting carryover for that respective matrix. The water rinse options evaluated were:

●90°C water bake rinse (1, 2, or 3 rinses)

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●one 90°C water bake rinse and one 3mL or one 5mL methanol rinse for the sparge vessel

●one 90°C water bake rinse, one 3mL methanol needle rinse and one 3mL methanol sparge vessel rinse

The soil rinse options evaluated were:

●one 90°C water needle rinse

●one 90°C water needle rinse and one 3mL methanol needle rinse

For this study, data pertaining to four of the higher molecular weight compounds and their respective matrices are summarized in Figures 1 and 2. These four compounds are typically the last compounds to elute from the GC and generally represent the worst case scenario for carryover. The chromatogram in Figure 3 illustrates the effectiveness of the methanol rinse of the glassware in limiting the carryover of the last four compounds. Overall, the Atomx displayed a remarkable capability of limiting carryover by utilizing Teledyne Tekmar's methanol rinse (patent pending) for the sparge vessel. In some instances, the methanol rinse resulted in a 50% decrease in carryover. It should be noted that one methanol rinse at 3mLs received the same benefit as the 5mL rinse, thus reducing overall methanol usage.

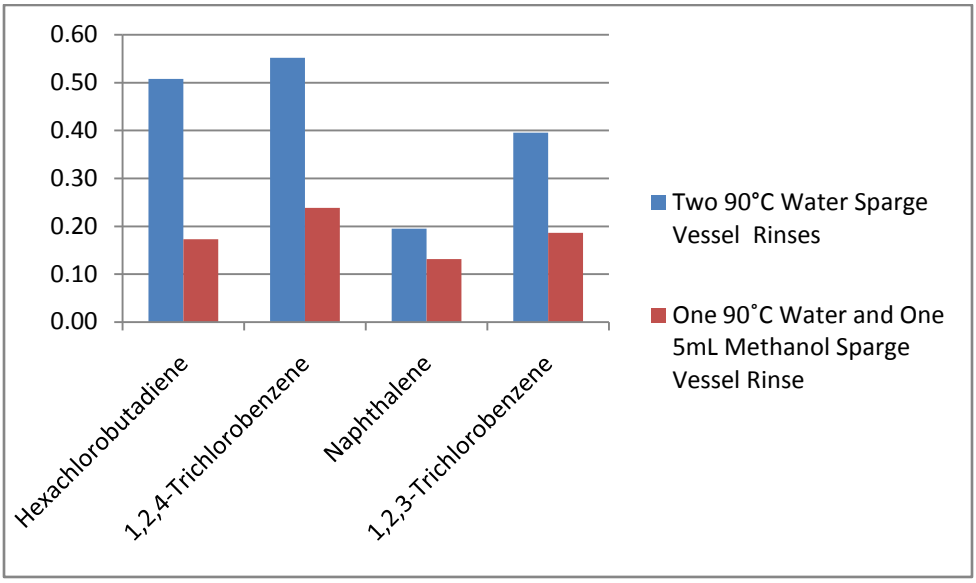

Figure 1: Percent Carryover Results of Last Four Compounds for Water Analysis

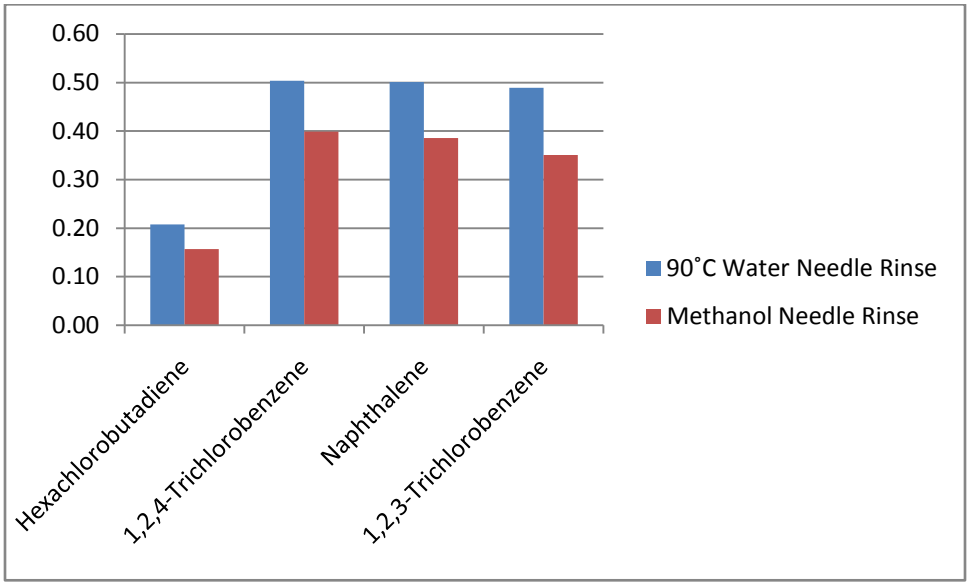

Figure 2: Percent Carryover of Last Four Compounds for Soil Analysis

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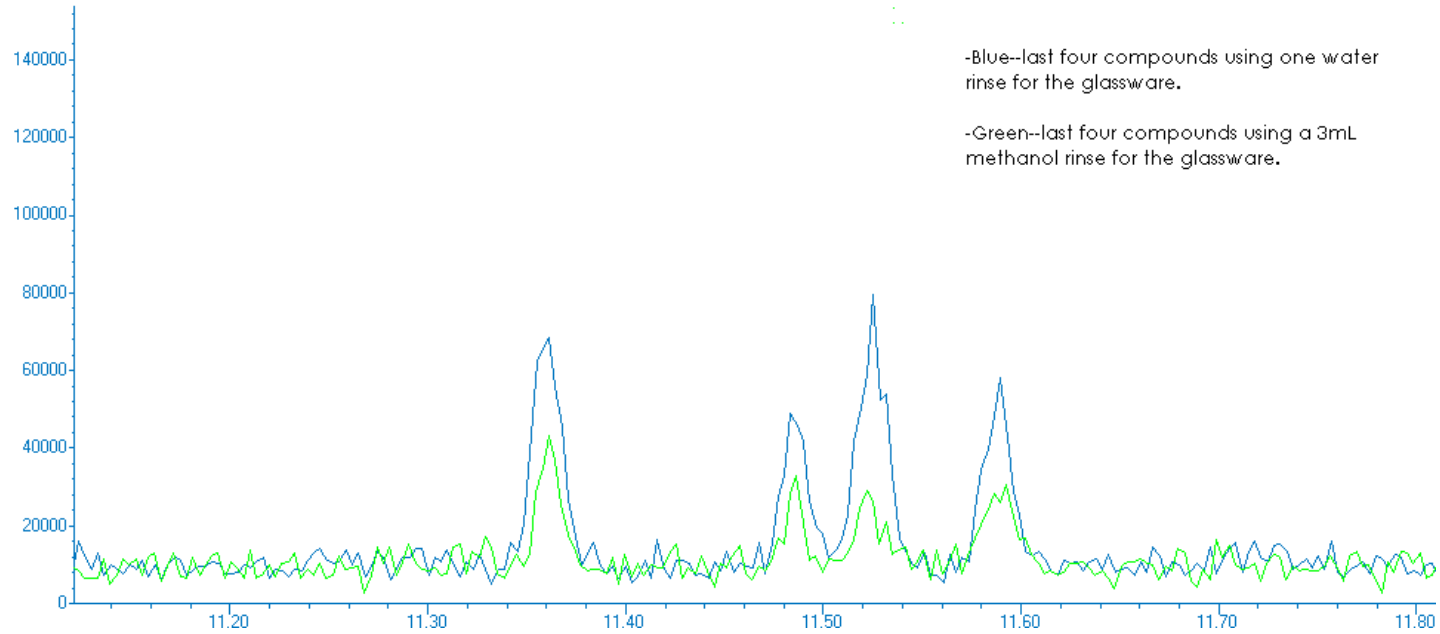

Figure 3: Chromatogram of the Last Four Compounds with Different Water Rinse Options.

Conclusions

The new Atomx offers the best of both worlds, combining the autosampler and the PTC into one system resulting in a shorter sample pathway and improved temperature uniformity. The system uses Siltek™ coated stainless steel and PEEK® tubing to aid in the reduction of carryover. Finally, the system offers several rinse options for carryover reduction. Rinsing options include the ability to rinse the multi-stage needle and/or the sparge vessel resulting in a thorough rinsing of the entire liquid sample path. The system is capable of rinsing the sample path with 90°C water, methanol, or both up to three times using varying volumes of either methanol or hot water. The methanol rinse (patent pending) featured with the Atomx system reduces carryover to less than 0.25% after a 200ppb water sample, this reduction in carryover is comparable to a standalone concentrator. The new methanol rinse option and its effectiveness in reducing carryover will increase productivity in any lab as it will aid in system clean up after high level samples.

References

1. USEPA Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December 1996.

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