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Evaluation of a New Analytical Trap for Gasoline Range Organics Analysis

Application Note

By Tyler Trent

Abstract

Purge and Trap (P&T) is a concentration technique used for the analysis of Volatile Organic Compounds (VOCs). The major component of any P&T system is the analytical trap. This trap is responsible for trapping the analytes purged from the sample and then releasing them upon heating for further analysis by the GC and GC/MS.

This study will evaluate a new analytical trap that is designed by Teledyne Tekmar. The packing material in this new trap improves the recovery and performance of Volatile Hydrocarbon Fractions found in Gasoline Range Organics (GRO) when prepared using Purge & Trap and Headspace sample concentration techniques used in tandem with GC.

Introduction

Purge and Trap (P&T) concentration is a typical technique used for the analysis of Volatile Organic Compounds (VOCs). The major component of any P&T system is the analytical trap. This trap is responsible for retaining the VOCs during purge and then releasing the analytes upon desorbing. The requirements for an analytical trap are as follows:

1. At low temperatures, it must retain the desired analytes while allowing oxygen and water to pass through unimpeded.
2. It must release the analytes quickly and efficiently upon heating
3. Must not contribute any volatiles of interest to the system.
4. Should have a reasonable price and lifetime.

The latest advancement in analytical traps by Teledyne Tekmar is the #11-VPH-Trap. This trap provides the features of Tekmar's #9 analytical trap while modifying the sorbent bed so a lesser amount of methanol is retained. The #11-VPH-Trap is ideal for running the Wisconsin Gasoline Range Organics (GRO) method **(e.g. methyl-tert-butyl-ether, toluene, benzene, ethylbenzene, xylenes, 1, 2, 4-trimethylbenzene, 1, 3, 5-trimethylbenzene and naphthalene)** and BTEX methods.

To evaluate the new #11-VPH-Trap, the analytical trap examined three Tekmar products the Atomx an automated VOC sample prep system (U-Shape), Velocity XPT Purge and Trap (straight trap) with an AQUATek 100 autosampler, and a HT3™ headspace analyzer (straight trap).

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For the Atomx, a calibration curve by summation, Replicate Lab Control Spikes (RLCS), Duplicate Lab Control Spikes (DLCS), Limit of Detection (LOD) and Limit of Quantitation (LOQ) were determined for water, high-level soil and low-level soil samples.[1] For the Velocity XPT a calibration by summation, RLCS, DLCS, LOQ and LOD were evaluated for water samples only.[1] The HT3 headspace analyzer generated individual calibration curves for all ten compounds listed above.

Atomx Automated VOC Sample Prep System

Instrument Parameters

The Atomx, equipped with a #11-VPH-Trap, and an Agilent 6890 GC/FID were utilized for this study. **Tables 1-4** show the GC/FID and P&T conditions for water, soil, and methanol extraction applications.

GC Parameters	
GC:	Agilent 6890
Detector	FID
Column	Restek RTX-VMS 20m x 0.18mmID x 1um
Oven Program:	40°C for 4 min; 16°C/min to 100°C for 0 min; 30°C /min to 200°C for 4 min, 15.083 min runtime
Inlet:	220°C
Column Flow	0.9mL/min
Gas:	Helium
Split:	80:1
Pressure:	21.542 psi
Inlet:	Split/Split less
FID	250°C, Hydrogen Flow 35.0mL/min, Air Flow 400.0mL/min, Constant Column and Makeup Flow 35mL/min

Table 1: GC/FID Parameters

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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	Off
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	0.50min
Soil Valve Temp	50°C	Desorb Preheat Time	245°C
Standby Flow	10mL/min	GC Start Signal	Start of Desorb
Purge Ready Temp	40°C	Desorb Time	2.00 min
Condensate Ready Temp	45°C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorb Temp	250°C
Prime Sample Fill Volume	3.0mL	Methanol Glass Rinse	Off
Sample Volume	5.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	3.0mL
Sweep Sample Flow	100mL/min	Number of Bake Rinses	1
Sparge Vessel Heater	On	Water Bake Rinse Volume	7.0mL
Sparge Vessel Temp	40°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	2.00 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	2.00 min		

Table 2: Atomx Water Parameters (Parameters highlighted in yellow were not used.)

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Atomx Soil Parameters			
Variable	**Value**	**Variable**	**Value**
Valve oven Temp	140°C	Purge Time	11.00 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	2.00 min
Prepurge Time	0.00 min	Dry Purge Flow	100mL/min
Prepurge Flow	0mL/min	Dry Purge Temp	20°C
Preheat Mix Speed	Slow	Methanol Needle Rinse	Off
Sample Preheat Time	0.00 min	Methanol Needle Rinse Volume	3.0mL
Soil Valve Temp	100°C	Water Needle Rinse Volume	7.0mL
Standby Flow	10mL/min	Sweep Needle Time	0.25 min
Purge Ready Temp	40°C	Desorbs Preheat Time	245°C
Condensate Ready Temp	45°C	GC Start Signal	Start of Desorbs
Presweep Time	0.25 min	Desorbs Time	2.00 min
Water Volume	10mL	Drain Flow	300mL/min
Sweep Water Time	0.25 min	Desorbs Temp	250°C
sweep Water Flow	100mL/min	Bake Time	2.00 min
Sparge Vessel Heater	Off	Bake Flow	400mL/min
Sparge Vessel Temp	20°C	Bake Temp	270°C
Purge Mix Speed	Fast	Condensate Bake Temp	200°C

Table 3: Atomx Soil Parameters (Parameters highlighted in yellow were not used.)

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Atomx Methanol Extraction 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	On
Soil Valve Temp	100˚C	Methanol Needle Rinse Volume	2.0mL
Standby Flow	10mL/min	Water Needle Rinse Volume	7.0mL
Purge Ready Temp	40˚C	Sweep Needle Time	0.25min
Condensate Ready Temp	45˚C	Desorb Preheat Temp	245˚C
Presweep Time	0.25min	GC Start Signal	Star of Desorb
Methanol Volume	7mL	Desorb Time	2.00min
Sparge Vessel Heater	On	Drain Flow	300mL/min
Sparge Vessel Temp	40˚C	Desorb Temp	250˚C
Prepurge Time	0.00min	Methanol Glass Rinse	On
Prepurge Flow	0mL/min	Number of Methanol Glass Rinses	1
Sample Mix Speed	Fast	Methanol Glass Rinse Volume	3.0mL
Sample Mix Time	4.00min	Number of Water Bake Rinses	1
Sample Settle Time	2.00min	Water Bake Rinse Volume	7.0mL
Sample Sweep Time	0.25min	Bake Rinse Sweep Time	0.25min
Sample Sweep Flow	100mL/min	Bake Rinse Sweep Flow	100mL/min
Purge Time	11.00min	Bake Rinse Drain Time	0.40min
Purge Flow	40mL/min	Bake Time	2.00min
Purge Temp	20˚C	Bake Flow	200mL/min
Condensate Purge Temp	20˚C	Bake Temp	280˚C
Dry Purge Time	2.00min	Condensate Bake Temp	200˚C

Table 4: Atomx Methanol Extraction Parameters

Calibration / Results

A 50ppm working calibration standard was prepared in methanol using a 1000ppm Wisconsin GRO/PRVO standard. Calibration standards were then serially diluted with de-ionized water over a range of 5-200ppb. A 25ppm surrogate standard of fluorobenzene was prepared in methanol and transferred to one of the three standard addition vessels on the Atomx. The Atomx delivered the surrogate in 5µL aliquots to the sample to give a final concentration of 25ppb.

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Using Agilent Chemstation software, the summation of the peak areas of each standard from methyl-tert-butyl-ether (MtBE) to naphthalene were used to generate the water and low-level soil calibration curves seen in **Figures 1-2**. The area of the surrogate fluorobenzene was subtracted from the total area to give the correct area requird for the Wisconsin GRO calibration.The calibrations passed method criteria with a correlation coefficient (r^2) of 0.9978 for water and r^2 of 0.9990 for low level soil. Using the #11-VPH-Trap it will allow for the resolution of MtBE down to 5ppb allowing for a calibration curve to be run from 5 to 200ppb.

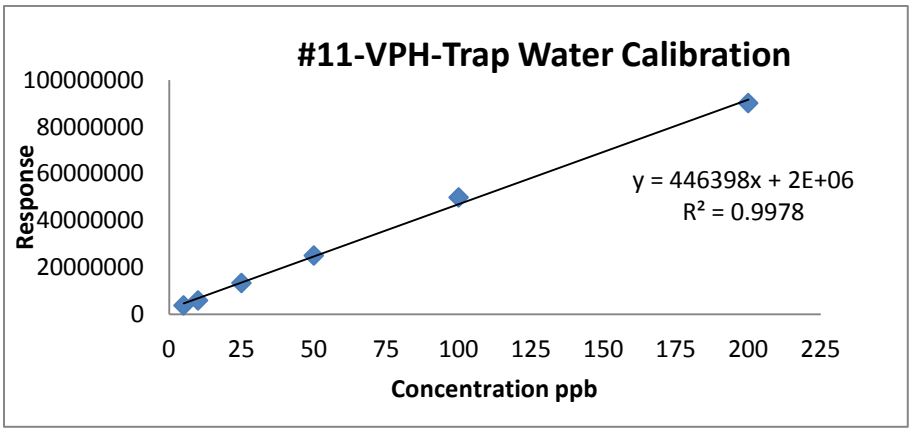

Figure 1: Wisconsin GRO water calibration on a range from 5-200ppb using #11-VPH-Trap on Teledyne Tekmar's Atomx

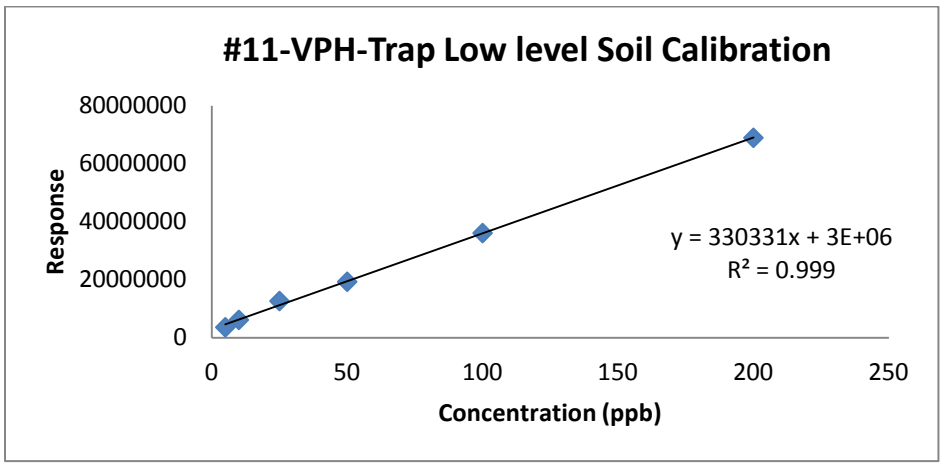

Figure 2: Wisconsin GRO low level soil calibration on a range from 5-200ppb using #11-VPH-Trap on Teledyne Tekmar's Atomx

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The Wisconsin GRO method requires for Quality Control (QC) checks to demonstrate the capabilty to generate acceptable accuracy and precision.[1] For the water/soil, each matrix must run a RLCS, DLCS, LOD and LOQ. Each of these test listed above must fall between required limits that are set forth in the Wisconsion GRO method. Results of these test can be found in **Table 5-6**. **Table 5** shows the capabilty of the Atomx to generate acceptable accuracy and precision for both water and soil matrices using the #11-VPH-Trap. **Table 6** shows that the LOQs for both matrices are below the required limits set by the method.

Water Quality Control				
	Theoretical (ppb)	Experimental (ppb)	%Recovery	Experimental %RSD
RLCS	100 +/-20%	95.73*	95.73	2.47
DLCS	100+/-20%	89.0	89.0	
Low Level Soil Quality Control				
RLCS	100 +/-20%	105.08*	105.08	1.59
DLCS	100+/-20%	97.48	97.48	
High Soil Quality Control (Methanol Extracted)				
	Theoretical (ppm)	Experimental (ppm)	%Recovery	Experimental %RSD
RLCS	10+/-120-75%	8.75*	87.5	3.96
DLCS	10+/-120-75%	8.61	86.1	

Table 5: Replicate Laboratory Control Spike (RLCS) and Duplicate Laboratory Control Spike (DLCS) Results for water and soil. (*n= 5 replicates)

Water/Soil Quality Control		
Matrix	LOD* (ppb)	LOQ*(ppb)
Water	1.56	4.96
Low Level Soil	1.854	5.89
High level Soil	1.16	5.14

Table 6: Results for for the Lower Limit of Detection (LOD) and Limit of Quantitation LOQ for both water and soils. (*n=7 replicates)

All the experimental values fall with in the acceptable range set forth by the Wisconsin GRO Method. The method states the LOQ for soil should be less then 10ppm and for groundwater it should be 0.1ppm or less.[1]

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Velocity XPT (straight trap) with an AQUATek 100 Autosampler

Instrument Parameters

The second part of the #11-VPH-Trap study was to validate the trap on a system that requires a straight analytical trap. A Velocity XPT concentrator with an AQUATek 100 autosampler was used in tandem with a GC/FID. The Agilent 6890 GC/FID parameters can be found in **Table 1** while the Velocity XPT and AQUATek 100 instrument parameters can be found in **Table 7**.

Velocity P&T and AQUATek 100 Parameters			
Variable	**Value**	**Variable**	**Value**
Valve Oven Temp	150˚C	Desorb Time	2.00 min
Transfer Line Temp	150˚C	Desorb Temp	250˚C
Purge Ready Temp	35˚C	Drain Flow	300 mL/min
Dry Flow Standby Temp	40˚C	Bake Time	2.00 min
Standby Flow	5 mL/min	Bake Temp	280˚C
Sparge Vessel Heater	On	Dry Flow Bake Temp	270˚C
Pre-Purge Time	0.00 min	Bake Flow	200 mL/min
Pre-Purge Flow	40 mL/min	Pressurize Time	0.35 min
Preheat Time	1.00 min	Sample Transfer Time	0.35 min
Sample Temp	40˚C	Rinse Loop Time	0.30 min
Purge Temp	20˚C	Sweep Needle Time	0.30 min
Purge Flow	40 mL/min	Bake Rinse	On
Dry Purge Time	2.00 min	Bake Rinse Cycle	1
Dry purge Temp	20˚C	Bake Rinse Drain Time	0.35 min
Dry Purge Flow	200 mL/min	Presweep Time	0.25 min
GC Start	Start of Desorb	Water Temp	90˚C
Desorb Preheat Temp	245˚C		

Table 7: Velocity XPT and AQUATek 100 parameters. (AQUATek 100 parameters are listed in blue)

Calibration / Results

A 50ppm working calibration standard was prepared in methanol using a 1000ppm Wisconsin GRO/PRVO standard from Restek. Calibration standards were then serially diluted with de-ionized water over a range of 5-200ppb. A 25ppm surrogate of fluorobenzene was prepared in methanol and transferred to one of the two standard addition vessels on the AQUATek100. The autosampler delivered the surrogate in 5µL aliquots to the sample for a final concentration of 25ppb.

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Using Agilent Chemstation software, the summations of each standard from methyl-tert-butyl-ether to naphthalene were used to generate the water calibration curves seen in **Figure 3**. The area of the surrogate fluorobenzene was subtracted from the total area to give the correct value required for the Wisconsin GRO calibration.The calibration pass method criteria with a correlation coefficient (r^2) of 0.9998.

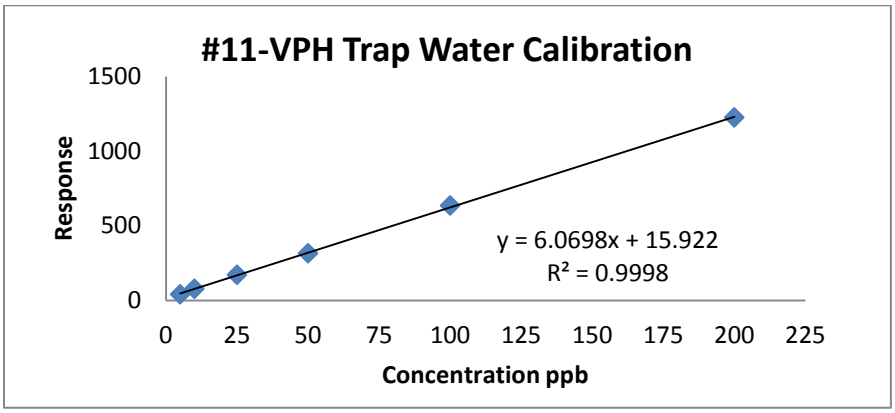

Figure 3: Wisconsin GRO water calibration on a range from 5-200ppb using #11-VPH-Trap on Teledyne Tekmar's Velocity XPT Purge and Trap Concentrator AQUATek 100 Autosampler.

Since the AQUATek 100 is a water autosampler, only the water portion of the Wisconsin GRO method could be evaluated. RLCS, DLCS, LOD and, LOQ were preformed. Results of these tests can be found in **Table 8-9**. **Table 7** shows the capabilty of the Velocity XPT with a AQUATek 100 to generate acceptable accuracy and precision for water samples using the #11-VPH-Trap. **Table 9** shows that the LOQs for both matrices are below the required limits set by the method.

Water Quality Control				
	Theoretical (ppb)	Experimental (ppb)	%Recovery	Experimental %RSD
RLCS	100 +/-20%	98.44*	98.44	4.20
DLCS	100+/-20%	97.10	97.10	

Table 8: Replicate Laboratory Control Spike (RLCS) and Duplicate Laboratory Control Spike (DLCS) Results for water.
(*n= 5 replicates)

Water Quality Control		
Matrix	LOD* (ppb)	LOQ*(ppb)
Water	1.22	3.90

Table 9: Results for for the LOD and LOQ for both water.
(*n=7 replicates)

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All the experimental values fall with in the acceptable range set forth by the Wisconsin GRO Method. The method states for LOQ the groundwater should be 0.1ppm or less.[1]

HT3™ Automated Headspace Vial Sampler

Instrument Parameters

The dynamic (trap) mode of the HT3™ headspace system was used to evaluate the GRO analysis. The HT3™ was connected to an Agilent 6890 GC/FID. HT3™ parameters are listed in **Table 10**. The Agilent 6890 GC/FID system parameters are listed in **Table 1**.

Dynamic (Trap)			
Variable	**Value**	**Variable**	**Value**
Platen/Sample Temp	75°C	Dry Purge Time	2.00 min
Valve Oven Temp	150°C	Dry Purge Flow	100mL/min
Transfer Line Temp	150°C	Dry Purge Temp	25°C
Standby Flow Rate	50mL/min	Desorb Preheat	245°C
Sample Preheat Time	0.00 min	Desorb Temp	250°C
Sweep Flow Rate	200mL/min	Desorb Time	1.00 min
Sweep Flow Time	5.00 min	Trap Bake Temp	270°C
Preheat Mixer	0.00 min	Trap Bake Time	3.00 min
Trap Material	#11	Trap Bake Flow	200mL/min

Table 10: Dynamic HT3™ Headspace parameters.

Standard and Sample Preparation

A 100ppb stock Internal Standard (IS) solution was prepared by adding 50 µL of a 2000ppm fluorobenzene standard to 1 L of de-ionized water. Using a Wisconsin GRO/PVOC mix a 50ppm stock standard was made. Calibration standards were then serially diluted with de-ionized water containing a 100ppb IS of fluorobenzene. The working range of the calibration ranged from 5-200ppb for Wisconsin GRO. 5 mL of the working standards were transferred to 22 mL headspace vials with a glass syringe and capped for the GRO analysis.

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Calibration / Results

The peak areas of the Wisconsin GRO standards from 5 to 200ppb were used to calculate the Response Factors (RF) by both external and internal standard calculations. The method requires %RSD less than 20% and correlation coefficients (r^2) greater than 0.99, these values are shown in **Table 11**.

Calibration Data		
5 mL Sample Data	%RSD of the Compound Response Factor	Correlation Coefficient of the Compound
Compound	#11-VPH-Trap	#11-VPH-Trap
MtBE	8.59	0.9999
Benzene	3.92	0.9996
Toluene	2.32	0.9993
Ethylbenzene	2.50	0.9995
m-, p-Xylene	1.02	0.9995
o-Xylene	2.39	0.9998
1,3,5-Trimethylbenzene	4.60	0.9999
1,2,4-Trimethylbenzene	4.00	0.9999
Naphthalene	5.82	0.9998

Table 11: Calibration data showing the %RSD and correlation coefficient for the calibration curve (5-200ppb)

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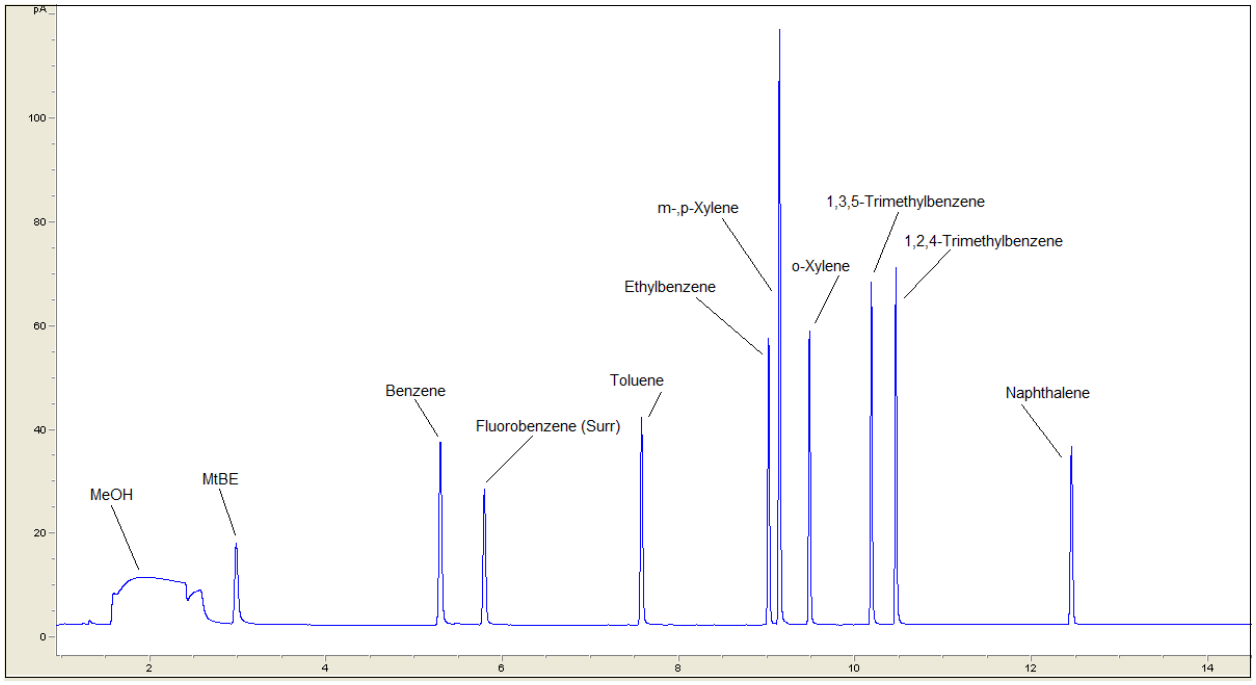

Figure 4: Chromatogram showing the separation 100ppb Wisconsin GRO standard from Methanol (MeOH) on the Tekmar HT3™

Using the #11-VPH-Trap enabled resolution of MtBE from the methanol peak seen in **Figure 4.** For all ten compounds listed in **Table 11** all the %RSD were below 10%, while the correlation coefficient were all greater than the requirements of 0.99 or better.

Trap Comparison

Throughout this study, the #11-VPH-Trap was compared to other traps for differences in performing this method. The first difference is the ability of the #11-VPH-Trap to resolve MtBE down to 5ppb while the M-Trap can only resolve MtBE down to 10ppb. **Figure 5** shows an overlay comparing chromatograms of a 5ppb Wisconsin GRO water sample on the #11-VPH-Trap and the M-Trap.

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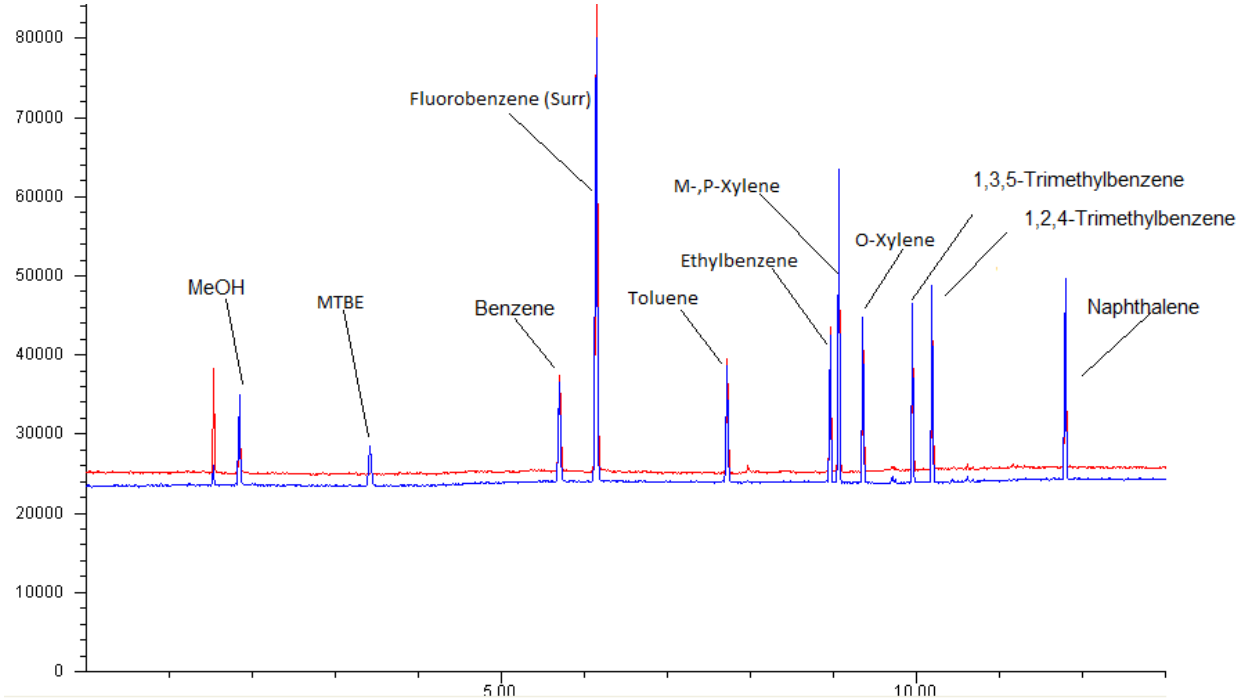

Figure 5: 5ppb chromatogram showing an overlay of Wisconsin GRO standard on the #11-VPH-Trap (Blue) and M-Trap (Red).

Using the #11-VPH-Trap will allow the analyst to generate a calibration curve for all compounds over a range of 5-200ppb for the Atomx automated VOC sample prep system, the Velocity XPT concentrator, and the HT3™ automated headspace vial sampler.

The second difference uncovered in the trap comparison was each trap's methanol retention. If the trap retains too much methanol, the associated solvent peak could affect, or in some cases completely mask the MtBE due to the similar retention times. Figure 6 shows an overlay of the solvent peak of three traps: the #11-VPH-Trap, The #9 Trap, and the M-Trap.

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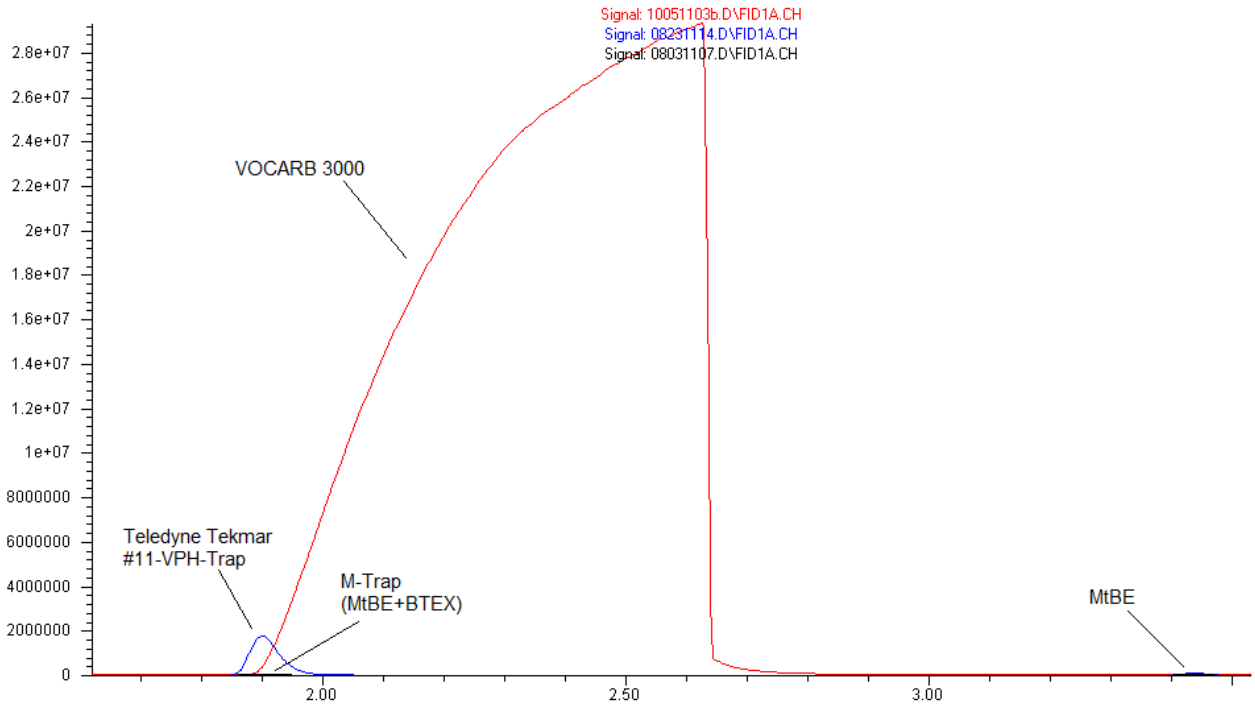

Figure 6: An overlay of the solvent peaks for all three traps.

As **Figure 6** demonstrates the VOCARB 3000 Trap retains the most amount of methanol due to the fact that it is typically used in full VOC list which require stronger sorbent materials in order to trap lighter volatiles such as Freon for example. The #11-VPH-Trap and M-Trap are designed for the analysis of Wisconsin GRO and BTEX compounds, which do not contain many polar compounds. Since these two traps do not require full analyte list the sorbent beds can be modified so that each trap retains a smaller amounts of methanol. The methanol does not interfere with the first analyte of interest (MtBE), making the #11-VPH-Trap an idea analytical trap for GRO analysis.

The third and final difference while noticed between the #11-VPH-Trap and the M-Trap was the bake back pressure. The #11-VPH-Trap behaved similarly to the the Tekmar #9 trap, the M-Trap showed evidence of increasing bake back pressure. This increasing pressure can lead to early degradation of the trap resulting in more frequent replacement of the analytical trap.

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Conclusion

The #11-VPH-Trap is the latest advancement in analytical traps by Teledyne Tekmar. This analytical trap provides the features of the Tekmar #9 analytical trap, but also reduces the sorbent bed so that lesser amount of methanol is retained. The trap keeps its ability to trap the polar and non-polar compounds found in gasoline range organics (ie: Wisconsin GRO and BTEX methods).

This study validates the use of the #11-VPH-Trap in three of Tekmar products the Atomx, Velocity XPT with an AQUATek 100 autosampler, and the HT3™ headspace analyzer. By using the #11-VPH-Trap the solvent peak is greatly reduced and does not interfere with the first analyte, MtBE. The #11-VPH-Trap also has a reduced bake pressure than the M-Trap which translates to a longer life cycle of the trap and reduces overall operation costs.

References

1. Modified GRO Method for Determine Gasoline Range Organics Wisconsin DNR September 1995 PUBL-SW-140

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