

HT3 Application Note

Introduction

Identifying and quantifying Volatile Organic Compounds (VOCs) in blood, breath and urine samples at sub-ppb levels has been a challenge for forensic laboratories. In addition, data must be able to stand up in court, so showing accuracy and repeatability in analytical results is critical. The new Teledyne Tekmar HT3™ Static and Dynamic Headspace system improves sensitivity by up to 100 times and offers new technology to ensure data is accurate and consistent.

Low-end sensitivity is the critical factor when developing an automated headspace GC method for blood volatiles. The HT3 Headspace system is the only system on the market today to offer the rugged dependability of the traditional fixed volume loop sampling technique, while dramatically increasing sensitivity with the new patent-pending dynamic trapping option.

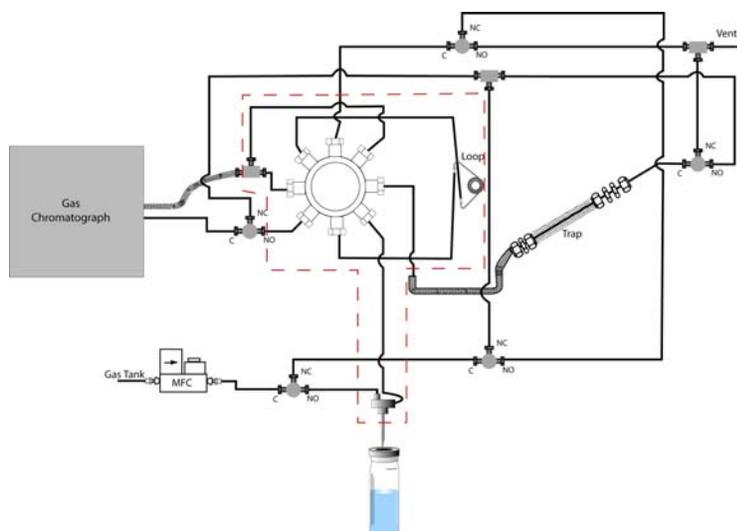
The fixed volume loop sampling method is for those samples that need to be prescreened or are known to contain large amounts of VOCs. This method transfers a single volume of headspace sample to the GCMS for detection.

The dynamic trapping method is for samples that demand greater sensitivity. This method sweeps the headspace of the sample vial for a fixed amount of time while simultaneously concentrating the volatiles onto an adsorbent trap. Once the trapping is completed, the analytes are transferred from the trap to the GCMS for detection. In the case of blood volatiles, this dynamic trapping method provides excellent low-end recovery and precision required for forensic applications.

The HT3 offers unprecedented headspace capabilities never before utilized in forensic laboratories:

- **Sample Pathway:** The HT3 system is plumbed with an inert sample pathway to achieve the quality control and cleanliness specifications set forth by the method.
- **System Temperature Control:** The entire sample pathway is temperature controlled up to 300 degrees Celsius in one degree increments ensuring reproducibility of sample collection and transfer.
- **Pressure/Flow Control:** The HT3 system utilizes a Mass Flow Controller/Pressure Transducer module that is capable of unmatched repeatability of gas flows, pressures, and volumes.

HT3 Sample Flow Path

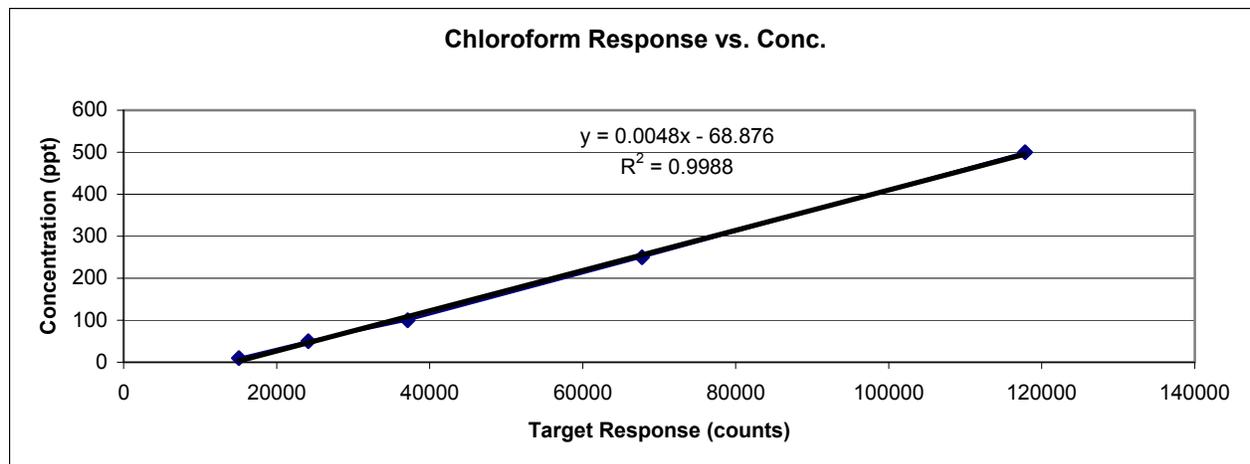


Experimental

Headspace Autosampler Parameters	
Platen Temp	70°C
Sample Preheat	5 min.
Sample Equilibration	14 min.
Transfer Line Temperature	175°C
Valve Oven Temperature	175°C
Vial Size	22 ml
Preheat Mixer	On / level 5 / 2 min.
Trap Standby Temp	30°C
Sweep Flow Rate	75 mL/min.
Sweep Flow Time	5. min
Dry Purge Time	1 min
Dry Purge Flow	50 mL/min
Dry Purge Temp	30°C
Desorb Preheat	245°C
Desorb Temp	260°C
Desorb Time	2 min
Trap Bake Temp	300°C
Trap Bake Time	8 min
Trap Bake Flow	450 mL/min

Target Compound	R ² Value
Vinyl chloride	.9978
Ethyl alcohol	.9983
Trans-1,2-dichlorethene	.9970
Cis-1,2-dichloroethene	.9986
Chloroform	.9988
1,1,1-trichloroethane	.9931
1,2-dichloroethane	.9969
Trichloroethylene	.9968
Dichloropropane	.9985
Bromodichloromethane	.9976
Toluene	.9955
Tetrachloroethylene	.9958
Dibromochloromethane	.9988
Chlorobenzene	.9969
Ethylbenzene	.9965
M,p-xylene	.9967
o-xylene	.9971
Styrene	.9962
Bromoform	.9952
1,4-dichlorobenzene	.9934
1,2-dichlorobenzene	.9932
1,2,4-trichlorobenzene	.9921

Calibration Curve



Target compounds were calibrated using 10, 50, 100, 250 and 500ppt solutions.

Conclusion

This data clearly demonstrates the HT3 Static and Dynamic Headspace system's capabilities in offering forensic laboratories a simpler, faster, and more repeatable tool for performing blood, breath and urine volatile analysis. By achieving superior sub-ppb data, the HT3 Headspace sampler is the first of its kind to match and surpass antiquated SPME methodology.

Teledyne Tekmar would like to thank Patrick Ayres, Cynthia Burbach, Ashley Demander, and C. Michael Smith of the Colorado Department of Public Health and Environment in Denver, Colorado, U.S.A. for their support and cooperation.