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Analysis of Volatile Organic Compounds in Soil Samples by EPA Method 8260 with The Stratum PTC and SOLATek 72 Multi-Matrix Autosampler

Application Note

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Introduction

Purge and Trap concentration (P&T) along with Gas Chromatographic (GC) analysis is a widely used method for the analysis of Volatile Organic Compounds (VOCs). This methodology was developed to achieve the high sensitivity necessary to determine VOCs in drinking water and other matrices according to United States Environmental Protection Agency (USEPA) Method 8260 as well as other SW8000 Methods.

To accommodate the requirements of USEPA Method 8260, Teledyne Tekmar has continually improved P&T technology by introducing new generations of enhanced concentrators into the analytical arena. The new Stratum PTC offers innovative U-shaped analytical and condensate traps, providing improvements over earlier P&T concentrators, and therefore demonstrating excellent data results for many current USEPA methodologies. In addition to introducing the Stratum PTC and U-shaped traps, Teledyne Tekmar has developed a proprietary #9 trap for improved chromatographic performance on early eluting compounds found in many of the aforementioned methods.

In this study soil sample analysis was performed and linear calibration was demonstrated for 95 target analytes over the range of 1.0-200ppb. A 10mL purge volume was utilized, using conditions and specifications outlined in USEPA Method 8260. The Stratum PTC and SOLATek 72 in conjunction with an Agilent 6890 GC and 5973 MSD are excellent tools for the identification and quantification of VOCs in soil matrices.

Experimental-Instrument Conditions

An Agilent 6890/5973 GCMS, Stratum Purge and Trap Concentrator and SOLATek 72 Multi-Matrix Autosampler were used for this analysis. Results and findings were obtained through the use of a 20m X 0.18mm X 1.0µm RTX-VMS fused silica capillary column (Restek Corporation). The Mass Spectrometer Detector (MSD) scanned in the full scan mode from 35-350m/z at 5.27 scan/sec. The GC, MSD and Purge and Trap conditions are shown in Tables 1, 2 and 3 respectively.

GC Parameters	
GC:	Agilent 6890
Column:	Restek RTX-VMS, 20m, 0.18mm ID, 1.0µm
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:	150°C
Column Flow:	1.2mL/min
Gas:	Helium
Split:	30:1
Flow:	1.2mL/min
Pressure:	20psi

MSD Parameters	
MSD:	Agilent 5973
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Column Flow:	1.2mL/min
Scan Range:	mz 35-350
Scans:	5.27 scans/sec
Threshold:	400

Tables 1 & 2: GC and MSD Parameters

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Stratum PTC and SOLATek 72 Parameters			
Variable	**Value**	**Variable**	**Value**
Rinse Water Temp	90°C	Sample Preheat Time	1.00 min
Sample Cup Temp:	40°C	Sample Temp	40°C
Sample Needle Temp	60°C	Purge Temp	0°C
Transfer Line Temp	125°C	Purge Flow	0mL/min
Soil Valve Temp	125°C	Condenser Ready Temp	40°C
Sample Sweep Time	0.50 min	Condenser Purge Temp	20°C
Needle Rinse Volume	7mL	Dry Purge Time	3.00 min
Needle Sweep Time	0.75 min	Dry Purge Temp	20°C
Sample Preheat Time	0.00 min	Dry Purge Flow	50mL/min
Preheat Stir	Off	GC Start	Start of Desorb
Preheat Stir Mode	Spin	Desorb Preheat Temp	245°C
Preheat Stir Speed	1	Desorb Drain	On
Purge Time	11 min	Desorb Time	2.00 min
Purge Stir	On	Desorb Temp	250°C
Purge Stir Mode	Spin	Desorb Flow	300mL/min
Purge Stir Speed	5	Bake Time	4.00 min
Valve Oven Temp	150°C	Bake Temp	270°C
Transfer Line Temp	150°C	Bake Flow	400mL/min
Sample Mount Temp	90°C	Condenser Bake Temp	175°C
Purge Ready Temp	45°C	Focus Temp	-150°C
Standby Flow	25mL/min	Inject Time	1.00 min
Pre-Purge Time	0.50 min	Inject Temp	180°C
Pre-Purge Flow	40mL/min	Standby Temp	100°C
Sample Heater	Off		

Table 3: Stratum PTC/SOLATek 72 Parameters

Stratum PTC Parameters are indicated with the blue background

Calibration

A working standard was prepared in methanol at a final standard concentration of 50ppm. Calibration standards were prepared in 100mL volumetric flasks filled to volume with deionized water over the calibration range of 1.0-200ppb and 10mL aliquots were transferred directly to VOA vials for analysis. Internal Standards (IS) were added at 5µL using the Internal Standards Addition Module of the SOLATek 72 Autosampler to hold at a constant concentration of 25ppb.

Calibration data was processed using Agilent ChemStation software. All analytes and their corresponding calibration data were evaluated using both the %RSD of the relative response factor and by calibration curve linearity. For most compounds the %RSD was <12% over the entire 1.0-200ppb-calibration range. For those target compounds with %RSD >15%, linear regression was employed with acceptance at 0.995 or greater, indicating linear response for all target analytes. The calibration data meets all USEPA Method 8260 performance criteria. Calibration data along with results of a 50ppb Continuing Calibration Verification (CCV) standard are presented in Table 4. Figure 1 shows the total ion chromatogram of a 50ppb standard.

Method Detection Limit (MDL) Determination

A study was performed to statistically determine the Method Detection Limits (MDL's) according to the procedure in USEPA Method 8260. Seven aliquots of a 1.0ppb standard were analyzed and the data processed to determine the MDL's for the compounds listed in Table 4 below. The detection limit results for most of the compounds were 0.5µg/L or less. The data collected met system performance criteria for Method 8260.

MDL according to 40CFR 136, Appendix B, Revision 1.11					
Compound	Spike level (ug/L)	%RSD	MDL	50ppbCCV (%DEV)	% Carryover
Pentafluorobenzene (IS)	25.0		0.000		
Dichlorodifluoromethane	1.00	12.65	0.404	89	0
Chloromethane	1.00	10.77	0.330	89	0.12
Vinyl Chloride	1.00	4.66	0.357	97	0.03
Bromomethane	1.00	10.33	0.330	94	0.20
Chloroethane (Ethyl Chloride)	1.00	11.47	0.471	96	0.03
Trichlorofluoromethane	1.00	9.19	0.400	93	0.02
Diethyl Ether	1.00	3.87	0.381	99	0.00
1,1-Dichloroethene	1.00	5.11	0.484	95	0
Carbon Disulfide	1.00	9.49	0.310	94	0.31
1,1,2-Trichlorofluoroethane	1.00	11.21	0.501	93	0.01
Iodomethane	1.00	14.71	0.228	113	0.28
Allyl Chloride	1.00	13.81	0.404	90	0.08
Methylene Chloride	1.00	8.6	0.357	92	0.06
Acetone	1.00	0.9999	0.710	99	0.30
trans-1,2-Dichloroethene	1.00	7.65	0.388	92	0.16
Methyl Acetate	1.00	7.89	0.431	91	0.13
MTBE	1.00	3.63	0.335	97	0.00
TBA	1.00	9.31	0.384	94	0
Diisopropyl Ether	1.00	7.21	0.313	97	0
Chloroprene	1.00	7.33	0.346	96	0.05
1,1-Dichloroethane	1.00	5.02	0.366	95	0.01
Acrylonitrile	1.00	10.07	0.417	93	0.26
Vinyl acetate	1.00	12.17	0.496	104	0.02
ETBE	1.00	2.64	0.292	98	0
cis-1,2-Dichloroethene	1.00	4.48	0.384	95	0.08
2,2-Dichloropropane	1.00	9.33	0.400	92	0
Bromochloromethane	1.00	6.25	0.303	92	0.05
Chloroform	1.00	4.12	0.435	94	0.01
Carbon Tetrachloride	1.00	2.5	0.228	97	0.00
1,1,1-Trichloroethane	1.00	6.68	0.469	95	0.00
THF	1.00	6.3	0.388	96	0
Dibromofluoromethane	1.00	9.03	0.466	94	0.00
Methyl Acrylate	1.00	5.34	0.356	95	0
1,1-Dichloropropene	1.00	2.71	0.304	98	0.05
2-Butanone (MEK)	1.00	10.2	0.391	88	0
Benzene	1.00	6.97	0.420	94	0.04
Propionitrile	1.00	4.47	0.317	94	0.10
tert Amyl Methyl Ether	1.00	2.84	0.113	98	0.00
1,2-Dichloroethane	1.00	10.7	0.511	92	0.08
Isobutyl Alcohol	1.00	11.11	0.324	83	0
Isopropyl Acetate	1.00	7.96	0.299	106	0.00
Trichloroethene	1.00	5.87	0.388	104	0.11
1,4-Difluorobenzene (IS)	25.00		0.000		
Dibromomethane	1.00	8.63	0.149	95	0.06
1,2-Dichloropropane	1.00	5.06	0.205	96	0.02
Bromodichloromethane	1.00	3.68	0.222	95	0.02
Methyl Methacrylate	1.00	0.9999	0.737	99	0

Stratum PTC Purge and Trap Concentrator

SOLATek 72 Multi-Matrix Autosampler

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MDL according to 40CFR 136, Appendix B, Revision 1.11					
Compound	Spike level (ug/L)	%RSD	MDL	50ppbCCV (%DEV)	% Carryover
n-Propyl Acetate	1.00	13.2	0.132	108	0.00
2-Cleve	1.00	0.9999	0.147	105	0
cis-1,3-Dichloropropene	1.00	10.91	0.127	104	0.04
Toluene-d8 (surr)	1.00	10.94	0.213	100	0.03
Toluene	1.00	13.19	0.164	98	0.04
Tetrachloroethene	1.00	11.93	0.503	100	0.07
4-methyl2-pentanone	1.00	12.07	0.394	104	0.00
1,1,2-Trichloroethane	1.00	8.69	0.404	94	0.00
Ethyl Methacrylate	1.00	9.77	0.779	106	0
Dibromochloromethane	1.00	5.39	0.561	101	0.01
1,3-Dichloropropane	1.00	7.23	0.212	96	0.02
1,2-Dibromoethane	1.00	4.34	0.112	97	0.03
n-Butyl Acetate	1.00	11.56	0.643	107	0.00
2-Hexanone	1.00	11.42	0.521	103	0.1
Chlorobenzene-d5 (IS)	25.00		0.000		
Chlorobenzene	1.00	11.4	0.236	95	0.09
Ethylbenzene	1.00	13.58	0.196	100	0.05
1,1,1,2-Tetrachloroethane	1.00	6	0.269	98	0
M&P Xylene	1.00	10.42	0.129	92	0.05
Ortho Xylene	1.00	13.68	0.165	109	0.03
Styrene	1.00	12.41	0.069	100	0.04
Bromoform	1.00	5.23	0.239	103	0
Isopropylbenzene	1.00	11.72	0.137	105	0.02
n-Amyl Acetate	1.00	9.55	0.149	107	0
BFB (surr)	1.00	8.24	0.090	99	0.08
n-Propylbenzene	1.00	10.28	0.163	97	0.04
cis-1,4-Dichloro-2-Butene	1.00	5.15	0.430	99	0
Bromobenzene	1.00	9.87	0.186	97	0.09
1,1,2,2-Tetrachloroethane	1.00	12.35	0.204	90	0.00
1,3,5-Trimethylbenzene	1.00	0.9999	0.157	128	0.03
2-Chlorotoluene	1.00	11.29	0.173	100	0.04
trans-1,4-Dichloro-2-Butene	1.00	5.15	0.368	99	0.04
4-Chlorotoluene	1.00	12.25	0.134	97	0.09
Tertbutylbenzene	1.00	14.55	0.231	104	0.03
1,2,4-Trimethylbenzene	1.00	13.96	0.175	106	0.02
sec-Butylbenzene	1.00	10.29	0.185	98	0.03
p-Isopropyltoluene	1.00	12.88	0.167	104	0.02
1,3-Dichlorobenzene	1.00	12.17	0.199	93	0.13
1,4-Dichlorobenzene-d4 (IS)	25.00		0.000		
1,4-Dichlorobenzene	1.00	13.18	0.187	93	0.17
n-Butylbenzene	1.00	12.07	0.145	101	0.07
1,2-Dichlorobenzen	1.00	11	0.175	95	0.08
1,2-Dibromo-3-Chloropropane	1.00	14.98	0.302	107	0
Nitrobenzene	1.00	12.12	1.046	107	0
Hexachlorobutadiene	1.00	11.26	0.227	96	0.13
1,2,4-Trichlorobenzene	1.00	9.58	0.160	102	0.16
Napthalene	1.00	10.83	0.235	105	0.09
1,2,3-Trichlorobenzene	1.00	12.34	0.302	94	0.14

Table 4: Calibration %RSDs and Statistically Determined Method Detection Limits for 8260 Target Compounds

Stratum PTC Newly Designed U-shaped Analytical and Condensate Traps

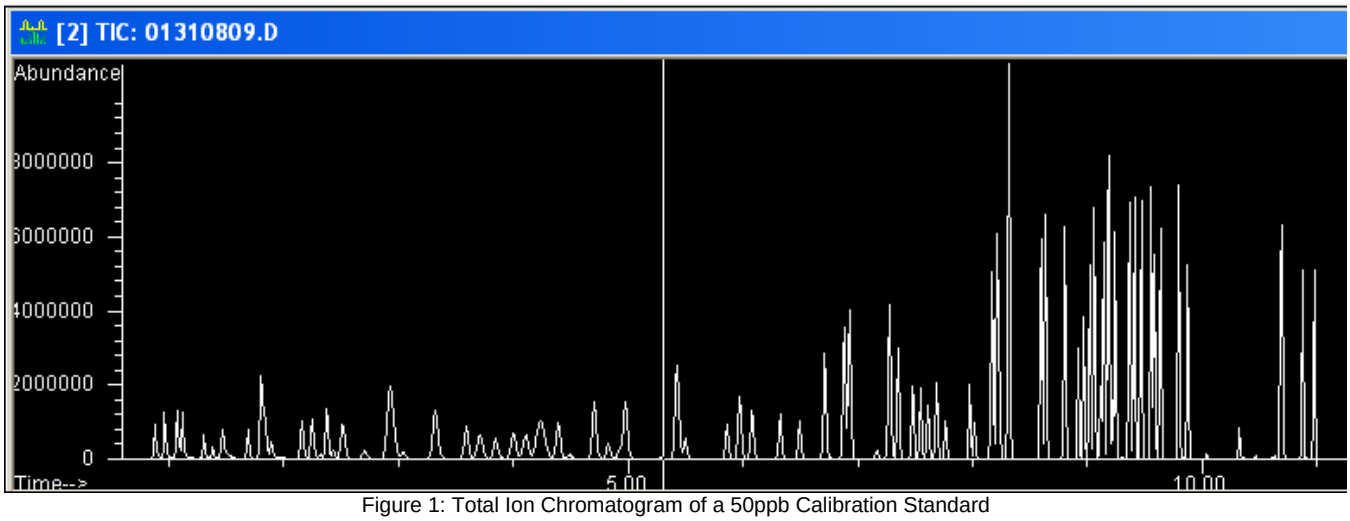

Figure 1: Total Ion Chromatogram of a 50ppb Calibration Standard

Carryover Evaluation

Data was collected and evaluated for carryover of target analytes in subsequent blanks following a 200ppb calibration standard. The Stratum PTC and SOLATek 72 Multi-Matrix Autosampler performed remarkably well. Most target compounds had carryover <0.4% in the first blank for all 95-target analytes evaluated. This data is also presented in Table 4.

Conclusion

The need for water removal from Purge and Trap analysis has been present since the introduction of Purge and Trap technology. The Stratum PTC is equipped with an innovative U-shaped condensate trap. The unique geometry of the trap aids in the removal of water that is typical in Purge and trap analysis. The new condensate trap offers improved water management and therefore a great replacement to early generation concentrators. Increasing demands for low-level sensitivity for VOC analysis has led the need for improved Purge and Trap technology. The new proprietary Tekmar #9 analytical trap performs well on troublesome early eluting compounds as well as difficult to retain compounds like 2-chlorovinyl ether and nitrobenzene. Using the # 9 trap, analysis of volatile organic compounds by USEPA Method 8260 was demonstrated. Linear calibration was also demonstrated over the range of 1.0-200ppb for 95 target compounds. The new Siltek coated sample pathway proves to be the optimal choice for the reduction of carryover for pathway sensitive compounds such as halogenates and others. The new Stratum PTC and SOLATek 72 Multi-Matrix Autosampler prove to be excellent analytical instruments. Teledyne Tekmar once again continues to offer excellent water management, improved analytical performance and a reduction in carryover in subsequent samples all while satisfying the requirements of multiple EPA and other analytical methods.

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