USEPA 8260D Determination Using the New Evolution 2 Purge and Trap Concentrator

Application Note

Environmental

Author

Anne Jurek
Applications Chemist
EST Analytical
Cincinnati, OH

Abstract

USEPA Method 8260D is a procedure that uses Gas Chromatography and Mass Spectrometry in order to determine Volatile Organic Compounds (VOCs). There are several preparative methods used in conjunction with this practice. USEPA Method 5030, for waters, and Method 5035, for soils and waste, are the purge and trap preparative methods for volatile organic analysis. This application will examine purge and trap sampling of VOCs in water and soil matrices.

Introduction:

Purge and trap concentrators have evolved over the years since they were introduced. Innovations include more efficient desorption to the GC, better moisture control, lower carryover, etc. The EST Analytical Evolution incorporated features and functionality that delivered better purge and trap concentration of VOCs. These include an 8-port valve for moisture control, patented desorb flow and pressure control for efficient desorption and the patented ability to bake the sparge vessel of the concentrator to limit carryover.

The Evolution 2 (EV2) is designed from the Evolution's framework and is a second-generation purge & trap concentrator. The EV2 features new enhancements for lab productivity while maintaining features that made its predecessor, the Evolution, a preferred purge and trap concentrator around the world. New enhancements on the EV2 include a smaller footprint for more lab bench space, better air circulation for shorter cool down times, and new software that can be run from a benchtop computer with an enhanced interface for easier day to day use. The architecture of the EV2 enables improved accessibility for easier maintenance and new electronics to ensure product sustainability and future connectivity.

This application will examine USEPA Method 8260D using the new Evolution 2 purge and trap concentrator for both water and soil matrices. Linearity, precision and accuracy, detection limits, carryover and Internal Standard stability will be among the results demonstrated using this new system.

Experimental:

A Vocarb 3000 trap was installed into the EST Analytical Evolution 2 purge and trap concentrator. The trap was conditioned per manufacturer requirements prior to the initiation of the experiments. The Centurion WS autosampler was used for sampling automation for both the water and soil analyses. The Gas Chromatograph (GC) was configured with a Rxi 624 Sil MS 30m x 0.25mm ID x 1.4µm film thickness column for analyte separation while the Mass Spectrometer (MS) was set in scan mode with a range of m/z 35 to 300 for compound analysis. Experimental parameters are listed in Tables 1 and 2.

Purge and Trap Concentrator	EST Analytical Evolution 2 (Water)	EST Analytical Evolution 2 (Soil)
Trap Type	Vocarb 3000	Vocarb 3000
Valve Oven Temp.	140°C	140°C
Transfer Line Temp.	140°C	140°C
Trap Temp.	35°C	35°C
Moisture Reduction Trap (MoRT) Temp.	39°C	39°C
Purge Time	11 min	11 min
Purge Flow	40mL/min	40mL/min
Dry Purge Temp.	Off	Off
Dry Purge Flow	40mL/min	40mL/min
Dry Purge Time	1.0 min	1.0 min
Desorb Pressure Control	On	On
Desorb Pressure	5psi	5psi
Desorb Time	0.5 min	0.5 min
Desorb Preheat Delay	5 sec	5 sec
Desorb Temp.	250°C	250°C
Moisture Reduction Trap (MoRT) Bake Temp.	210°C	210°C
Bake Temp	255°C	255°C
Sparge Vessel Bake Temp.	110°C	110°C
Bake Time	6 min	6 min
Bake Flow	40mL/min	40mL/min
Purge and Trap Auto-Sampler	**EST Analytical Centurion WS**	**EST Analytical Centurion WS**
Sample Type	Water	Soil
Sample Fill Mode	Syringe	Syringe
Sample Volume	5mL	5mL
Sample Prime Time	7 sec	7 sec
Loop Equilibration Time	5 sec	5 sec
Sample Transfer Time	5 sec	5 sec
Syringe Rinse	On/20mL	Off
Number of Syringe Rinses	2	NA
Sample Loop Rinse	On/10 sec	Off
Sample Loop Sweep Time	5 sec	NA
Number of Sparge Rinses	Syringe/2	NA
Rinse Volume	5mL	NA
Rinse Transfer Time	10 sec	NA
Rinse Drain Time	20 sec	NA
Number of Foam Rinse Cycles	3	NA
Water Heater Temp.	85°C	85°C
Internal Standard Vol.	5µl	5µl
Sample Preheat Temp.	NA	40°C
Soil Valve Temp.	NA	85°C
Soil Transfer Line Temp.	NA	150°C

Table 1: Evolution 2 and Centurion WS Water and Soil Experimental Parameters

GC/MS	Parameter
Inlet	Split/Splitless
Inlet Temp.	220°C
Inlet Head Pressure	7.774 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 Parameters

The GC/MS system was first checked for any air leaks or moisture contamination. Next, a Bromofluorobenzene (BFB) sample was examined in order to establish the GC/MS passed the method requirements for BFB before initiating the study. The linear range of the system was established by analyzing a nine-point calibration curve from 0.5 to 200µg/L according to the outlined procedures in the method, while the internal standard concentration was held constant at 50µg/L. The data were analyzed in order to determine the linearity of the calibration. The percent Relative Standard Deviation (%RSD) for most of the compounds was below the method criteria of <20%. For the one compound with a %RSD >20%, a weighted linear regression curve fit was used. Blank samples and low-level standard samples were run over the course of three days in order to determine the Method Detection limits (MDLs). For the precision and accuracy study, 50ppb standards were prepared and analyzed in seven replicates for the determination of the precision (% RSD) and % recovery of the analytes. These calibration, MDL and precision and accuracy results for both matrices are listed in Tables 3 and 4. This procedure was followed for both the water and soil matrices. Furthermore, internal standard stability was established by running 30 water blanks sequentially and examining the consistency of the internal standard compound responses. See Figure 1. Carryover was determined by the examination of the first blank sample after a 50µg/L and a 200µg/L water standard. This study was repeated three times with the averaged carryover results summarized in Table 5 and 6. Finally, chromatograms of a 50µg/L standard of both the water and the soil matrices are shown in Figures 2 and 3.

Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry	Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry
Dichlorodifluoromethane	0.374	8.23	0.15	5.75	81.19	Dibromomethane	0.183	10.88	0.17	1.73	101.67
Chloromethane	0.497	7.45	0.09	4.04	93.82	Bromodichloromethane	0.410	3.27	0.07	2.29	99.68
Vinyl Chloride	0.507	4.45	0.09	4.25	93.64	2-nitropropane	0.139	7.17	0.11	1.83	107.80
Bromomethane	0.396	11.94	0.24	2.60	90.87	cis-1,3-Dichloropropene	0.448	4.09	0.08	2.48	99.12
Chloroethane	0.348	5.96	0.17	4.49	94.71	4-methyl-2-pentanone	0.421	5.80	0.11	1.50	106.28
Trichlorofluoromethane	0.722	4.51	0.17	4.95	87.36	Toluene-d8 SUR	0.943	3.45	0.11	3.27	101.31
Ethanol	0.008	11.03	7.74	5.93	105.27	Toluene	0.683	4.83	0.09	3.36	101.64
diethyl ether	0.377	2.34	0.10	2.00	102.37	ethyl methacrylate	0.446	9.15	0.15	1.74	108.38
1,1,2-trichlorofluoroethane	0.382	7.64	0.14	6.22	86.44	trans-1,3-Dichloropropene	0.428	6.54	0.07	2.32	101.85
1,1-Dichloroethene	0.373	5.82	0.15	4.91	95.28	1,1,2-Trichloroethane	0.274	3.62	0.13	2.04	100.37
Acetone	0.280	8.82	0.52	1.48	103.17	Tetrachloroethene	0.266	8.50	0.15	3.02	101.78
Iodomethane	0.443	8.61	0.28	5.53	103.40	1,3-Dichloropropane	0.474	2.92	0.15	2.14	101.34
Carbon Disulfide	1.048	3.34	0.12	5.05	95.29	butyl acetate	0.574	9.54	0.16	1.91	107.76
methyl acetate	0.594	8.65	0.18	3.23	105.33	Dibromochloromethane	0.313	7.90	0.11	1.97	102.76
Acetonitrile	0.067	10.63	1.36	9.65	102.28	2-Hexanone	0.322	7.60	0.12	1.83	108.79
allyl chloride	0.585	3.55	0.17	3.32	100.23	1,2-Dibromoethane	0.298	3.32	0.09	1.66	100.80
Methylene Chloride	0.452	6.06	0.14	2.77	97.70	Chlorobenzene	0.844	3.71	0.10	3.33	94.48
MTBE	1.404	1.83	0.06	1.98	103.17	1,1,1,2-Tetrachloroethane	0.312	3.80	0.13	2.90	96.55
acrylonitrile	0.274	2.45	0.06	1.96	105.77	Ethylbenzene	1.412	3.50	0.11	3.51	97.74
Isopropylether	1.377	2.66	0.08	2.57	105.52	Xylene (m+p)	1.078	4.04	0.24	3.52	98.99
Vinyl acetate	1.469	7.71	0.22	2.22	108.58	Xylene (o)	1.104	5.30	0.08	3.25	100.53
1,1-Dichloroethane	0.799	2.61	0.07	3.64	103.52	Styrene	0.893	7.46	0.11	2.88	102.73
Ethyl Tert Butyl Ether	1.376	2.32	0.07	1.94	104.78	n-amyl acetate	0.600	8.90	0.16	1.98	105.45
Chloroprene	0.695	4.02	0.13	4.51	101.00	Bromoform	0.259	10.94	0.18	2.21	99.05
trans-1,2-Dichloroethene	0.426	2.61	0.14	4.34	99.61	Isopropylbenzene	1.336	6.44	0.14	3.56	98.83
ethyl acetate	0.103	6.06	0.22	2.00	102.78	cis-1,4-dichloro-2-butene	0.165	8.22	0.10	1.90	100.16
2-Butanone	1.059	1.87	0.10	1.79	105.30	BFB SUR	0.772	4.76	0.10	3.48	93.05
cis-1,2-Dichoroethene	0.491	2.91	0.09	3.36	101.41	Bromobenzene	1.434	5.18	0.06	2.82	95.66
2,2-Dichloropropane	0.509	5.25	0.50	4.23	82.00	1,2,3-Trichloropropane	0.809	3.84	0.14	2.74	94.79
Bromochloromethane	0.301	2.21	0.13	3.17	99.71	1,1,2,2-Tetrachloroethane	0.932	4.31	0.10	1.89	93.03
propionitrile	0.123	2.70	0.16	2.43	106.09	n-Propylbenzene	3.020	4.97	0.12	4.11	96.27
methacrylonitrile	0.398	2.15	0.11	1.71	105.14	trans-1,4-dichloro-2-butene	0.302	3.48	0.20	2.04	93.59
THF	0.256	10.17	0.18	1.56	99.43	2-Chlorotoluene	0.603	5.50	0.14	4.26	96.04
Chloroform	0.860	3.59	0.06	3.25	99.84	4-Chlorotoluene	0.628	5.03	0.15	4.08	96.11
methyl acrylate	0.637	3.18	0.09	1.65	107.86	1,3,5-Trimethylbenzene	2.044	6.50	0.12	4.00	98.21
Dibromofluoromethane	0.474	9.37	0.12	3.11	96.03	tert-Butylbenzene	1.801	7.54	0.15	3.76	95.84
1,1,1-Trichloroethane	0.743	4.88	0.14	4.34	98.12	Pentachloroethane	0.311	8.00	0.27	4.30	85.52
Carbon Tetrachloride	0.619	6.19	0.20	4.94	95.75	sec-Butylbenzene	0.519	9.08	0.18	3.92	94.32
1,1-Dichloropropene	0.616	4.96	0.14	4.72	97.01	1,2,4-Trimethylbenzene	2.087	6.61	0.10	3.92	99.26
isobutyl alcohol	0.043	7.47	0.50	1.68	105.82	1,3-Dichlorobenzene	1.209	2.94	0.10	3.73	93.48
isopropyl acetate	1.127	3.57	0.09	1.99	107.18	1,4-Dichlorobenzene	1.265	5.84	0.09	3.25	90.94
Benzene	1.779	2.24	0.07	3.57	103.29	Isopropyltoluene	2.131	9.14	0.16	3.90	98.18
1,2-Dichloroethane	0.695	3.25	0.09	2.31	102.16	1,2,-Dichlorobenzene	1.172	5.91	0.06	3.26	94.52
Tert Amyl Methyl Ether	1.358	3.87	0.06	2.15	105.04	n-Butylbenzene	1.909	8.32	0.15	4.13	97.57
1,4-Dioxane	0.010	4.60	3.95	5.62	108.46	Nitrobenzene	0.079	11.84	0.71	1.84	103.93
tert-butlyl alcohol	1.344	9.85	0.61	3.18	86.55	1,2-Dibromo-3-chloroprop	0.249	4.63	0.15	1.74	94.79
propyl acetate	0.577	8.43	0.10	1.75	107.42	1,2,4-Trichlorobenzene	0.721	6.26	0.09	2.98	93.92
Trichloroethene	0.285	3.17	0.15	3.43	97.11	Naphthalene	2.450	6.13	0.06	1.98	98.84
1,2-Dichloropropane	0.282	3.33	0.07	2.66	100.78	Hexachlorobutadiene	0.259	13.50	0.27	3.89	92.89
methyl methacrylate	0.274	7.19	0.09	1.70	106.17	1,2,3-Trichlorobenzene	0.696	5.52	0.12	2.45	95.19

Table 3: Data Summary of 8260 Water

Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry	Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry
Dichlorodifluoromethane	0.289	11.92	0.19	3.89	89.78	Dibromomethane	0.194	3.58	0.20	3.27	92.83
Chloromethane	0.463	11.57	0.05	4.17	86.44	Bromodichloromethane	0.436	3.76	0.10	3.50	94.28
Vinyl Chloride	0.502	7.66	0.12	3.72	88.59	2-nitropropane	0.076	13.77	0.14	2.76	114.93
Bromomethane	0.278	11.29	0.23	2.31	111.89	cis-1,3-Dichloropropene	0.494	4.66	0.08	3.72	96.05
Chloroethane	0.340	7.40	0.22	5.07	88.10	4-methyl-2-pentanone	0.324	6.98	0.10	3.69	95.54
Trichlorofluoromethane	0.730	9.56	0.17	2.77	91.14	Toluene-d8 SUR	1.029	6.55	0.13	3.79	93.34
Ethanol	0.007	*0.999	4.62	5.97	96.89	Toluene	0.781	9.27	0.09	3.70	90.48
diethyl ether	0.371	5.65	0.13	3.14	92.63	ethyl methacrylate	0.408	5.82	0.08	3.38	100.12
1,1,2-trichlorofluoroethane	0.400	10.81	0.16	3.08	92.67	trans-1,3-Dichloropropene	0.452	6.61	0.10	3.42	97.55
1,1-Dichloroethene	0.394	6.05	0.13	3.35	90.18	1,1,2-Trichloroethane	0.273	8.91	0.11	3.36	91.15
Acetone	0.228	14.62	1.20	2.20	87.86	Tetrachloroethene	0.353	6.55	0.15	3.83	86.13
Iodomethane	0.417	14.00	0.16	2.81	107.22	1,3-Dichloropropane	0.473	4.49	0.13	3.32	94.37
Carbon Disulfide	1.399	14.38	0.23	2.78	87.13	butyl acetate	0.471	6.44	0.15	2.97	100.06
methyl acetate	0.437	10.25	0.10	2.41	85.33	Dibromochloromethane	0.322	3.69	0.08	3.18	96.51
Acetonitrile	0.034	6.39	2.71	9.51	113.18	2-Hexanone	0.239	6.45	0.12	3.47	96.55
allyl chloride	0.659	8.25	0.09	4.22	92.43	1,2-Dibromoethane	0.282	2.51	0.05	3.12	95.17
Methylene Chloride	0.493	13.21	0.20	2.69	88.31	Chlorobenzene	0.933	8.49	0.10	4.16	90.25
MTBE	1.382	5.85	0.12	2.38	94.20	1,1,1,2-Tetrachloroethane	0.344	4.42	0.11	4.08	92.89
acrylonitrile	0.201	9.19	0.13	2.61	94.55	Ethylbenzene	1.631	11.39	0.13	3.84	90.05
Isopropylether	1.424	4.33	0.13	2.67	96.20	Xylene (m+p)	1.242	11.26	0.25	3.79	90.35
Vinyl acetate	1.252	6.11	0.10	2.51	114.33	Xylene (o)	1.243	7.66	0.10	4.02	93.37
1,1-Dichloroethane	0.870	5.18	0.12	2.96	92.79	Styrene	0.993	4.67	0.07	4.01	96.91
Ethyl Tert Butyl Ether	1.425	4.27	0.13	2.73	95.25	n-amyl acetate	0.511	5.28	0.06	3.53	104.34
Chloroprene	0.759	5.51	0.10	3.02	94.14	Bromoform	0.244	7.77	0.23	3.40	98.99
trans-1,2-Dichloroethene	0.482	6.57	0.14	3.35	92.67	Isopropylbenzene	1.493	6.67	0.10	3.64	95.68
ethyl acetate	0.078	7.24	0.29	2.59	94.65	cis-1,4-dichloro-2-butene	0.142	7.41	0.15	3.57	103.67
2-Butanone	0.828	11.79	0.05	2.89	90.90	BFB SUR	0.833	10.35	0.11	4.72	90.38
cis-1,2-Dichoroethene	0.547	5.84	0.16	3.14	93.23	Bromobenzene	1.455	7.26	0.12	4.13	92.96
2,2-Dichloropropane	0.739	6.67	0.15	2.50	94.79	1,2,3-Trichloropropane	0.674	9.44	0.09	5.30	91.22
Bromochloromethane	0.334	3.42	0.22	2.40	91.63	1,1,2,2-Tetrachloroethane	0.776	7.73	0.06	3.58	93.66
propionitrile	0.086	7.09	0.71	2.09	96.46	n-Propylbenzene	0.346	7.36	0.10	3.98	94.07
methacrylonitrile	0.317	7.31	0.08	2.29	93.91	trans-1,4-dichloro-2-butene	0.247	8.98	0.05	3.70	98.89
THF	0.180	9.49	1.39	3.82	90.75	2-Chlorotoluene	0.667	6.66	0.06	3.88	92.96
Chloroform	0.937	8.07	0.08	2.71	90.64	4-Chlorotoluene	0.702	7.88	0.11	4.92	92.87
methyl acrylate	0.492	3.37	0.17	2.16	97.72	1,3,5-Trimethylbenzene	2.293	6.82	0.07	4.38	94.74
Dibromofluoromethane	0.495	8.00	0.10	2.62	90.97	tert-Butylbenzene	2.064	8.33	0.12	4.06	94.06
1,1,1-Trichloroethane	0.821	6.87	0.11	2.60	91.18	Pentachloroethane	0.223	11.65	0.30	6.59	119.27
Carbon Tetrachloride	0.691	9.64	0.14	2.48	91.37	sec-Butylbenzene	0.609	10.32	0.14	3.87	93.99
1,1-Dichloropropene	0.694	6.27	0.09	2.68	90.71	1,2,4-Trimethylbenzene	2.302	5.36	0.09	4.54	95.94
isobutyl alcohol	0.030	10.29	1.64	3.60	93.91	1,3-Dichlorobenzene	1.317	8.33	0.11	4.78	93.65
isopropyl acetate	0.942	5.44	0.09	2.19	96.53	1,4-Dichlorobenzene	1.369	10.31	0.12	5.02	90.93
Benzene	1.958	8.64	0.11	2.60	91.97	Isopropyltoluene	2.501	8.68	0.12	4.18	95.59
1,2-Dichloroethane	0.730	10.04	0.11	1.84	91.51	1,2,-Dichlorobenzene	1.258	6.44	0.11	4.32	93.28
Tert Amyl Methyl Ether	1.375	4.52	0.12	2.18	95.08	n-Butylbenzene	2.332	8.74	0.09	4.55	96.82
1,4-Dioxane	0.007	7.48	3.01	4.89	103.21	Nitrobenzene	0.055	9.88	0.79	3.62	101.32
tert-butlyl alcohol	1.242	10.66	0.39	6.06	86.96	1,2-Dibromo-3-chloroprop	0.184	5.00	0.22	4.60	94.06
propyl acetate	0.450	6.14	0.08	3.86	100.08	1,2,4-Trichlorobenzene	0.827	10.10	0.18	5.57	94.58
Trichloroethene	0.334	8.55	0.10	3.47	88.93	Naphthalene	2.245	7.28	0.10	4.30	96.04
1,2-Dichloropropane	0.299	5.63	0.10	3.64	93.25	Hexachlorobutadiene	0.448	11.88	0.26	3.90	93.03
methyl methacrylate	0.236	5.83	0.12	3.61	95.25	1,2,3-Trichlorobenzene	0.789	7.31	0.12	4.69	95.17

Table 4: Data Summary of 8260 Soil

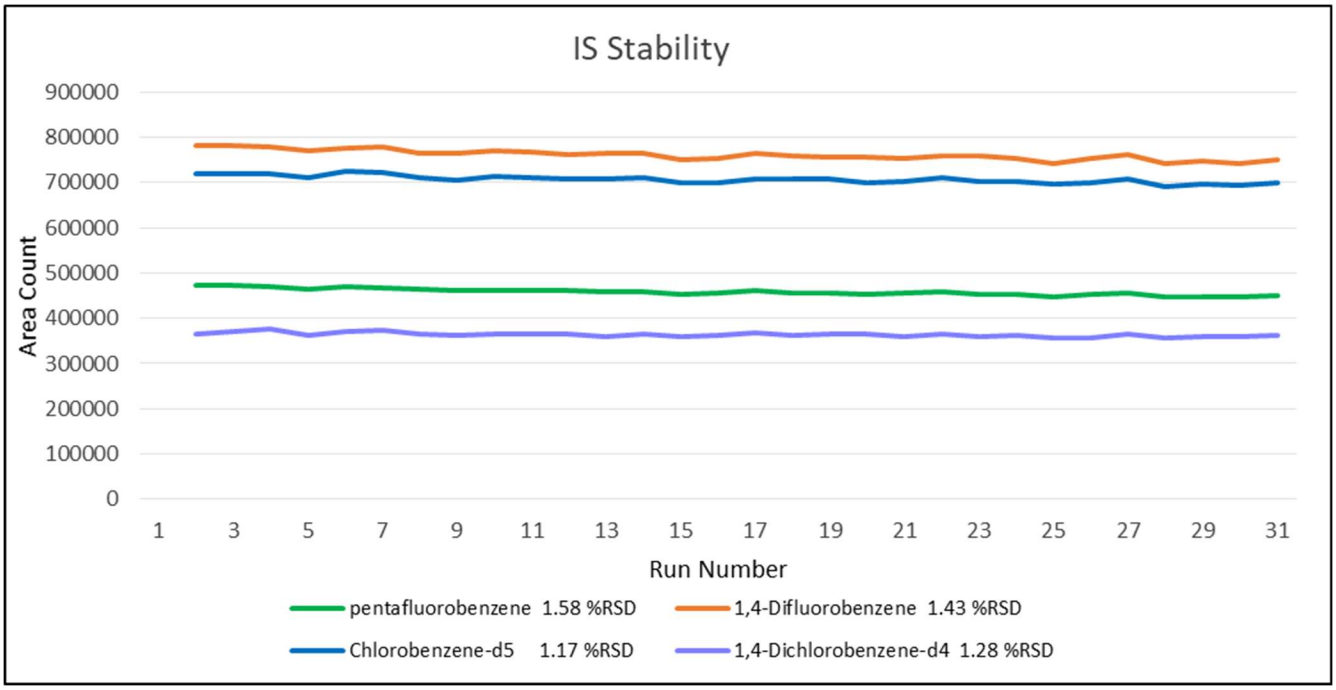

Figure 1: IS Reproducibility

50ppb Carryover First Blank				
Compound	**% Carryover 1**	**% Carryover 2**	**% Carryover 3**	**Average**
Benzene	0.089	0.091	0.098	0.093
Toluene	0.076	0.093	0.066	0.078
Ethylbenzene	0.076	0.090	0.081	0.083
p&m-Xylene	0.071	0.090	0.086	0.083
o-Xylene	0.072	0.067	0.064	0.068
1,2,4-Trichlorobenzene	0.190	0.346	0.278	0.272
Naphthalene	0.188	0.214	0.239	0.214
Hexachlorobutadiene	0.000	0.000	0.213	0.071
1,2,3-Trichlorobenzene	0.187	0.220	0.263	0.223

Table 5: % Carryover after a 50ppb Standard

200ppb Carryover First Blank				
Compound	% Carryover 1	% Carryover 2	% Carryover 3	Average
Benzene	0.070	0.076	0.078	0.074
Toluene	0.055	0.060	0.064	0.060
Ethylbenzene	0.064	0.065	0.071	0.067
p&m-Xylene	0.062	0.064	0.067	0.064
o-Xylene	0.049	0.052	0.058	0.053
1,2,4-Trichlorobenzene	0.224	0.254	0.228	0.235
Naphthalene	0.192	0.185	0.191	0.189
Hexachlorobutadiene	0.000	0.000	0.176	0.059
1,2,3-Trichlorobenzene	0.213	0.255	0.235	0.234

Table 6: % Carryover after a 200ppb Standard

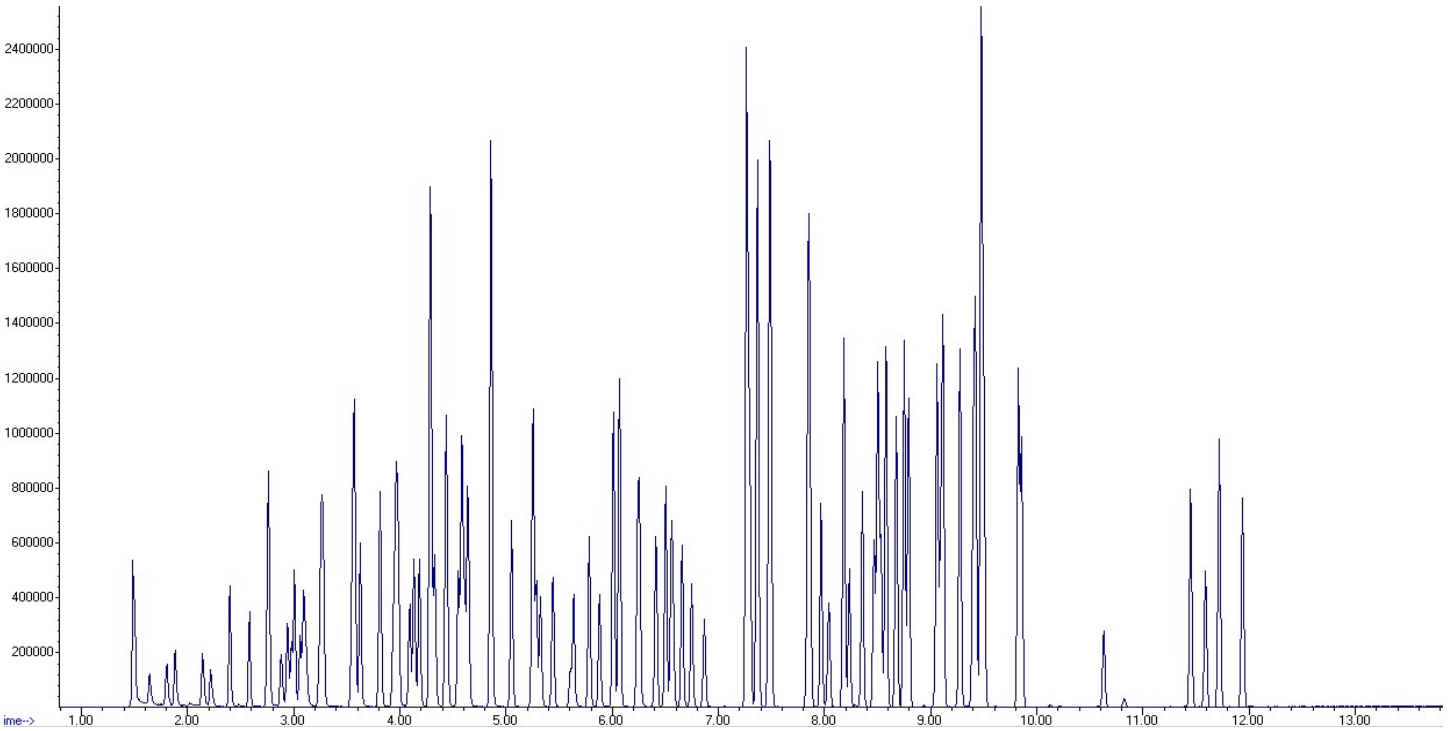

Figure 2: 50µg/L Water Chromatogram

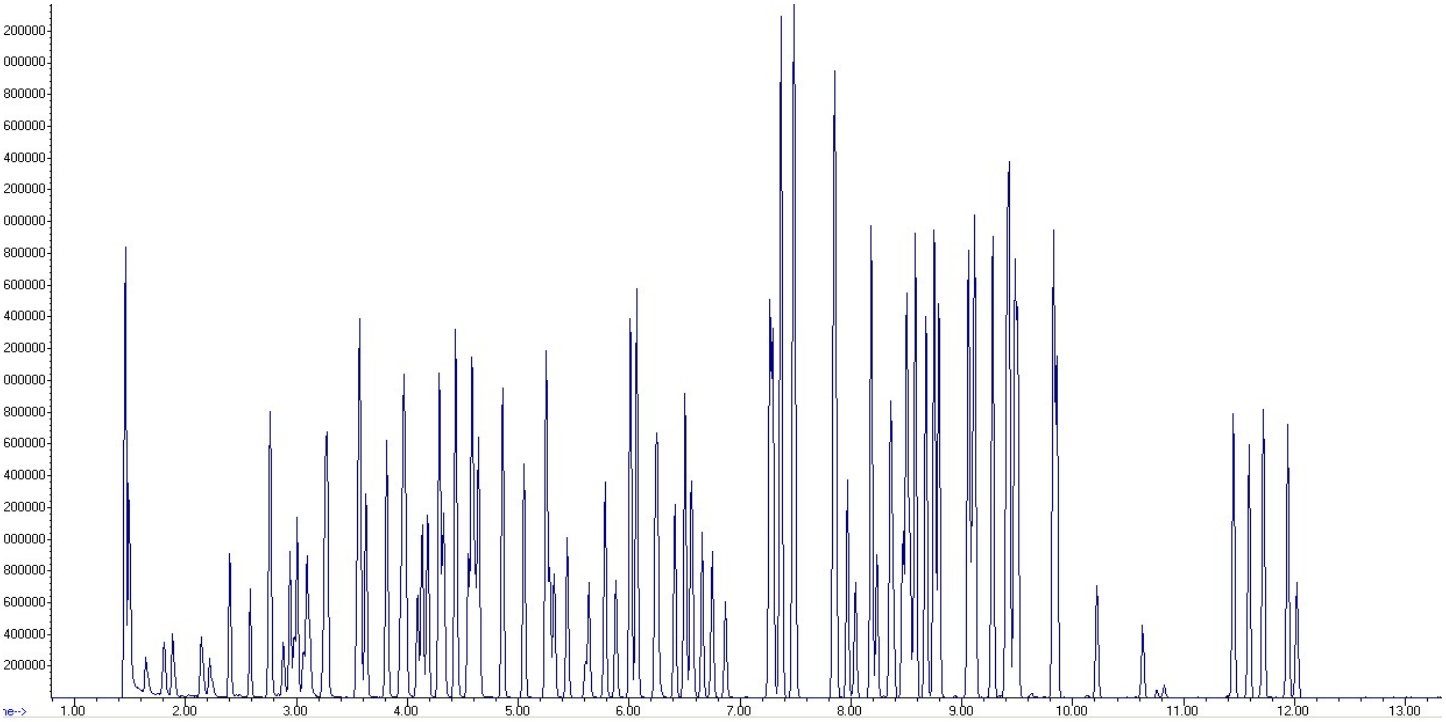

Figure 3: 50µg/L Soil Chromatogram

Conclusions:

The new Evolution 2 is the most advanced purge and trap concentrator from EST analytical. In conjunction with the Centurion Water/Soil autosampler it provided results that met all of the USEPA Method 8260D criteria. The linearity of both curves showed less than 15% RSD for a nine-point calibration with a linear range of 0.5 to 200µg/L. The precision and accuracy at 50µg/L averaged less than 5% for precision with a 95% average recovery. Furthermore, carryover after a 200µg/L sample was minimal and the internal standard stability over 30 blanks was less than 2% RSD for all four internal standards. This Evolution 2 with its smaller footprint, better circulation and new software interface is an excellent advancement in the EST Analytical product line.

References:

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); United States Environmental Protection Agency Method 8260D, Revision 4, June 2018.

For More Information

For more information on our products and services, visit our website www.estanalytical.com/products