

Analysis of Volatile Organic Compounds Using USEPA Method 8260 and the 4760 Purge and Trap and the 4100 Autosampler

Introduction

Although analysis of VOCs by purge and trap is considered a mature technique, advances in methodology and refinement of instrumentation are still being developed. This application note presents suggested operating conditions for the 4760 purge and trap and the 4100 autosampler.

Methodology

A multi-point calibration from 2 to 200 ppb was run using the conditions listed below.



Purge-and-Trap	Eclipse 4760 P&T Sample Concentrator
Trap	#10 trap; Tenax® / Silica gel / CMS
Purge Gas	Zero grade Helium at 40 mL/min
Purge Time	11 min
Sparge Mount Temperature	45 °C
Sample Temperature (purge)	45 °C
Sample Temperature (bake)	45 °C
Desorb Time	0.5 min
Bake Time	4 min
OI #10 Trap Temperature	Ambient during purge 180 °C during desorb pre-heat 190 °C during desorb 210 °C during bake
Water Management	120 °C during purge Ambient during desorb 240 °C during bake
Transfer Line Temperature	140 °C
Six-port Valve Temperature	140 °C

Autosampler	4100 Water/Soil Sample Processor
System Gas	Zero grade nitrogen
Purge Gas	Zero grade helium
LV20 Pressure	8.0 psi
Loop-based Time Settings	Default
Rinse Water	80 °C
Soil Sample Transfer	150 °C
Soil Oven	150 °C
Soil Lift Station	45 °C

4100 Sample Processor Methods			
Sample Type	Waters Only	Soils Only	Blanks Only
Vial Cap Color	Blue	Yellow	Green
Needle Rinses	1	1	0
SAM A (µL)	5	5	5
SAM B (µL)	0	0	0
SAM C (µL)	0	0	0
SAM D (µL)	0	0	0
Purge Time (min)	11.0	11.0	11.0
Desorb Time (min)	0.5	0.5	0.5
P&T Rinses	2	1	0
Rinse Water	Hot	Hot	Hot
Water Stir Time (min)	0.0		
Water Settle Time (sec)	0		
Soil Add Water to Vial (#loops)		* 1 x 5 mL	
Soil Pre-Heat Stir		Yes	
Soil Pre-Heat/Purge Temp (°C)		45.0	
Soil Stir During Purge		Yes	

* Suggested initial volume in vial should be 5 mL and final volume 10 mL.

Gas Chromatograph	Agilent 7890A
Column	Restek Rxi 624 Sil MS 30 meter, 0.25 mm ID, 1.4 µm df
Carrier Gas	Zero grade helium
Inlet Temperature	250 °C
Inlet Liner	Agilent Ultra Inert, 1 mm straight taper
Column Flow Rate	0.8 mL/min
Split Ratio	150:1
Oven Program	Hold at 40 °C for 1.5 min 16 °C/minute to 180 °C 40 °C/minute to 220 °C Hold at 220 °C for 1.75 min Total GC Run is 13 min
Mass Spectrometer	Agilent 5975C
Mode	Scan 35 - 300 amu
Scans/Second	5.19
Solvent Delay	1.05 min
Transfer Line Temperature	250 °C
Source Temperature	300 °C
Quadrupole Temperature	200 °C
Draw Out Plate	6 mm

Results

Results are listed in Table 1.

Table 1. Calibration Data

Analyte	Compound	Liquid RF	% RSD	Solid RF	% RSD
1	pentafluorobenzene (IS)				
2	dichlorodifluoromethane	0.401	3.75	0.392	9.58
3	chloromethane	0.352	4.58	0.323	6.51
4	vinyl chloride	0.386	4.11	0.354	6.94
5	bromomethane	0.313	9.54	0.290	8.33
6	chloroethane	0.168	6.97	0.155	7.79
7	trichlorofluoromethane	0.537	5.51	0.601	7.61
8	ethyl ether	0.158	5.15	0.142	5.03
9	1,1-dichloroethene	0.338	2.44	0.342	7.06
10	carbon disulfide	0.740	6.22	0.867	6.30
11	1,1,2-trichloro-1,2,2-trifluoroethane	0.356	2.94	0.353	10.12
12	methyl iodide	0.640	4.73	0.714	8.20
13	allyl chloride	0.170	4.80	0.161	7.11
14	methylene chloride	0.336	2.64	0.370	8.25
15	acetone	0.042	14.66	0.023	6.35
16	trans-1,2-dichloroethene	0.403	1.58	0.389	7.48
17	methyl tert-butyl ether	0.907	4.80	0.799	6.85

Analyte	Compound	Liquid RF	% RSD	Solid RF	% RSD
18	acetonitrile	0.023	14.19	0.020	11.03
19	chloroprene	0.621	5.28	0.603	7.74
20	1,1-dichloroethane	0.687	2.92	0.671	7.23
21	acrylonitrile	0.185	12.25	0.111	10.79
22	cis-1,2-dichloroethene	0.465	2.10	0.454	7.71
23	2,2-dichloropropane	0.228	3.85	0.264	6.57
24	bromochloromethane	0.243	3.87	0.233	7.67
25	chloroform	0.792	2.36	0.774	7.35
26	methyl acrylate	0.471	5.23	0.267	6.79
27	carbon tetrachloride	0.559	2.11	0.562	6.66
28	dibromofluoromethane (SS)	0.512	1.71	0.536	1.40
29	1,1,1-trichloroethane	0.555	3.05	0.562	5.45
30	2-butanone	0.054	7.83	0.026	6.82
31	1,1-dichloropropene	0.528	1.38	0.502	7.47
32	1,4-difluorobenzene (IS)				
33	benzene	0.994	1.24	0.937	13.41
34	methacrylonitrile	0.186	6.84	0.107	9.40
35	1,2-dichloroethane-d4 (SS)	0.049	2.03	0.045	4.82
36	1,2-dichloroethane	0.424	4.20	0.375	14.86
37	trichloroethene	0.316	2.27	0.288	8.24
38	dibromomethane	0.228	1.75	0.184	7.79
39	bromodichloromethane	0.378	4.71	0.353	5.92
40	1,2-dichloropropane	0.257	3.17	0.239	7.06
41	methyl methacrylate	0.192	3.45	0.109	6.81
42	2-chloroethyl-vinyl-ether	0.197	5.82	0.117	6.70
43	cis-1,3-dichloropropene	0.365	8.68	0.322	7.36
44	chlorobenzene-d5 (IS)				
45	toluene-d8(ss)	1.311	1.50	1.362	1.33
46	toluene	0.794	1.50	0.779	8.01
47	2-nitropropane	0.122	13.55	0.061	9.80
48	4-methyl-2-pentanone	0.042	4.64	0.022	5.03
49	tetrachloroethene	0.357	5.18	0.330	5.83
50	trans-1,3-dichloropropene	0.392	12.20	0.337	10.61
51	ethyl methacrylate	0.352	7.57	0.245	7.63
52	1,1,2-trichloroethane	0.286	2.54	0.237	6.97
53	chlorodibromomethane	0.394	7.02	0.350	6.90
54	1,3-dichloropropane	0.414	1.35	0.339	5.98
55	1,2-dibromoethane	0.420	2.33	0.306	5.49
56	2-hexanone	0.358	2.99	0.157	4.80
57	chlorobenzene	1.001	1.00	0.928	7.64
58	ethylbenzene	1.546	1.70	1.435	6.83
59	1,1,1,2-tetrachloroethane	0.288	3.61	0.286	5.96
60	m,p-xylenes	0.616	2.18	0.577	6.30

Analyte	Compound	Liquid RF	% RSD	Solid RF	% RSD
61	o-xylene	0.590	3.60	0.560	6.36
62	styrene	1.004	4.24	0.929	4.49
63	bromoform	0.283	11.99	0.210	7.89
64	isopropylbenzene	1.452	4.26	1.374	5.03
65	cis-1,4-dichloro-2-butene	0.160	6.81	0.089	5.45
66	1,4-dichlorobenzene-d4 (IS)				
67	4-bromofluorobenzene (SS)	1.066	2.22	1.087	3.23
68	bromobenzene	0.936	2.79	0.893	9.38
69	n-propylbenzene	3.831	2.58	3.739	7.87
70	1,1,2,2-tetrachloroethane	1.048	2.43	0.700	6.51
71	2-chlorotoluene	2.213	2.34	2.146	7.98
72	1,3,5-trimethylbenzene	2.695	3.00	2.657	5.87
73	1,2,3-trichloropropane	1.150	6.62	0.660	7.72
74	trans-1,4-dichloro-2-butene	0.323	4.85	0.187	5.19
75	4-chlorotoluene	2.606	3.19	2.545	8.58
76	tert-butylbenzene	2.552	2.65	2.531	7.63
77	pentachloroethane	0.382	7.85	0.415	7.73
78	1,2,4-trimethylbenzene	2.728	3.31	2.658	5.89
79	sec-butylbenzene	3.321	2.77	3.250	5.77
80	p-isopropyltoluene	2.686	4.08	2.620	5.32
81	1,3-dichlorobenzene	1.521	2.37	1.468	8.27
82	1,4-dichlorobenzene	1.530	1.06	1.468	8.67
83	n-butylbenzene	2.521	6.02	2.461	5.66
84	1,2-dichlorobenzene	1.429	1.98	1.343	6.52
85	1,2-Dibromo-3-chloropropane	0.204	12.57	0.096	9.28
86	hexachlorobutadiene	0.526	5.22	0.573	5.60
87	1,2,4-trichlorobenzene	0.983	6.87	0.941	6.18
88	naphthalene	2.866	13.02	1.872	8.90
89	1,2,3-trichlorobenzene	0.940	7.24	0.872	6.66

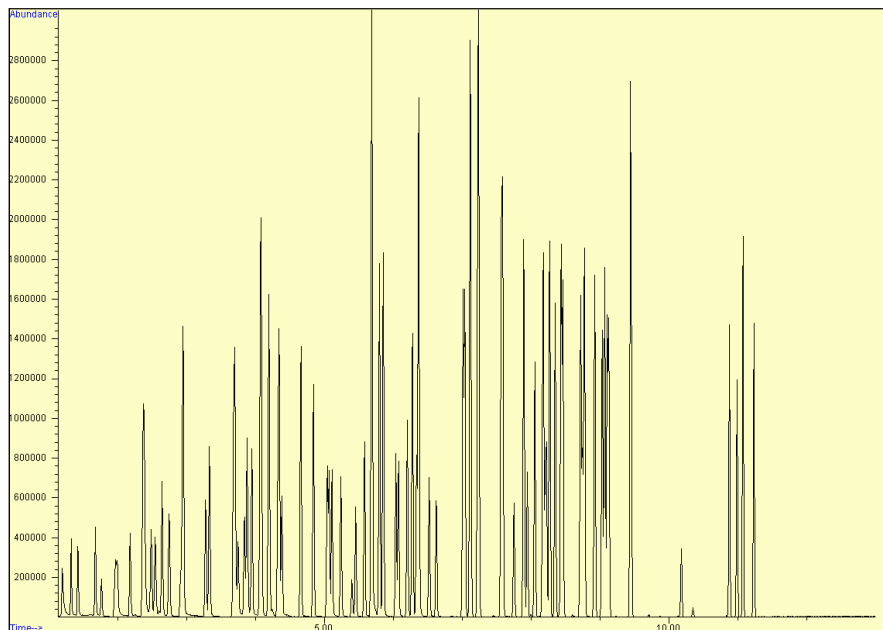


Figure 1. Liquid Standard (50 ppb)

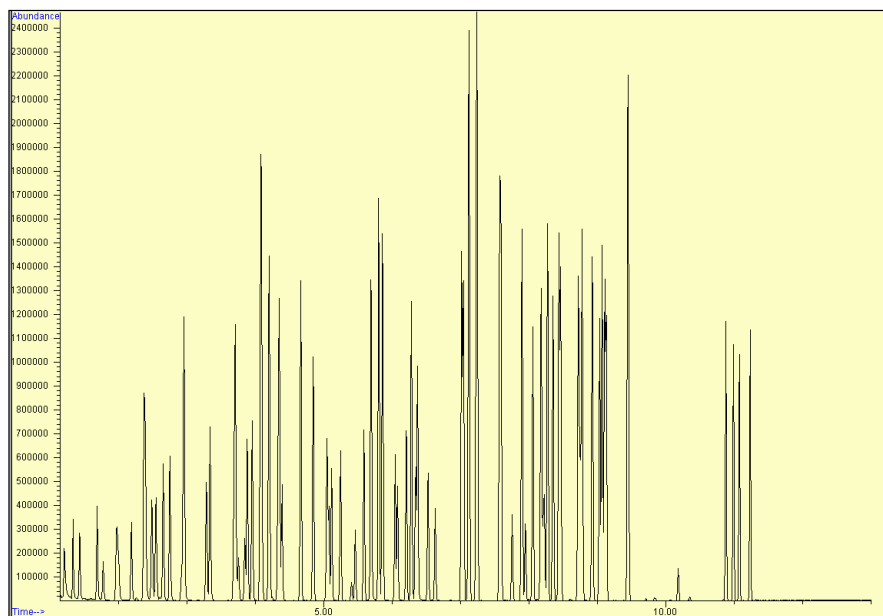


Figure 2. Solid Standard (50 ppb)

Conclusions

Calibrations were generated using USEPA Method 8260. All method criteria were met.