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A Single Calibration Method for Water AND Soil Samples Performing EPA Method 8260

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Introduction:

The United States Environmental Protection Agency (USEPA) Method 8260 is used to determine Volatile Organic Compounds (VOCs) in a variety of matrices. These matrices can vary from water to soil to sludge. In order to determine VOCs in the assorted matrices, the sampling and analysis system needs to be calibrated as closely to the matrix as possible. Therefore, USEPA Method 8260 requires a separate calibration for waters, soils and extractions. This application note will demonstrate a patent pending, automated water sampling mode using the soil sampling station of the autosampler thus eliminating the need to have separate calibrations for waters and soils.

Discussion:

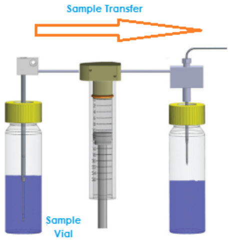
Environmental laboratories are always searching for ways to save time and run more samples per twelve hour increment. In order to save time, the bake time can be reduced, the purge time and flow can be changed, a dual sampling system can be run on one GC/MS, etc. However, one of the most basic ways to save time is by reducing the number of required curves and standards that need to be run. Unfortunately, the USEPA Method 8260 dictates the curves and sample experimental parameters need to be the same, and remain 5035 compliant, until now.

EST Analytical has designed a patent pending, automated water sampling mode that will allow environmental laboratories to run water samples in the soil mode. Thus, water and soil curves, standards, blanks and samples can use the same sampling parameters. (See steps 1, 2, and 3 below). This innovative sampling mode would eliminate the need to have separate curves, standards, etc. for soils and waters, thus saving laboratories time and money.

Step 1:

Empty vials are placed in one tray while water standards or samples are placed in the second tray.

The system transports the empty vial to the soil sampling station and it is moved onto the sample needle.



Step 2:

The arm moves over to the full water vial, the vial is pressurized with helium gas and the prescribed water volume is removed.

Internal standard (IS) is added by injecting IS into the sample as it is transported to the vial in the soil sampling station.

Step 3:

The sample is heated and purged in the same manner as a soil sample, and the analytes are trapped on the analytical trap. Next, the analytes are desorbed onto the GC column.

Experimental:

The sampling system used for this study was the EST Analytical Centurion WS autosampler and Encon Evolution concentrator. The concentrator was affixed with a Vocarb 3000 trap and connected to an Agilent 7890A GC and 5975 inert XL MS. The GC was configured with a Restek Rxi-624 Sil MS 30m x 0.25mm x 1.4µm column. The GC/MS experimental conditions are outlined in Table 1. The experiments were run using the extraction mode of the Centurion WS. Refer to Table 2 for the sampling method parameters.

GC/MS	Agilent 7890A/5975 inert XL
Inlet	Split/Splitless
Inlet Temp.	200ºC
Inlet Head Pressure	12.153 psi
Mode	Split
Split Ratio	40:1
Column	Rxi-624Sil MS 30m x 0.25mm I.D. 1.4µm film thickness
Oven Temp. Program	45ºC hold for 1 min., ramp 15ºC/min to 220ºC, hold for 1.3 min.
Column Flow Rate	1.0mL/min
Gas	Helium
Total Flow	44.00mL/min
Source Temp.	230ºC
Quad Temp.	150ºC
MS Transfer Line Temp.	180ºC
Scan Range	m/z 35-265
Scans	3.12 scans/sec
Solvent Delay	0.7 min

Table 1: GCMS Parameters

Purge and Trap Concentrator	EST Encon Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	150ºC
Transfer Line Temp.	150ºC
Trap Temp.	35ºC
Moisture Reduction Trap (MoRT) Temp.	39ºC
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	40mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	5psi
Desorb Time	0.5 min
Desorb Preheat Delay	5 sec.
Desorb Temp.	260ºC
Moisture Reduction Trap (MoRT) Bake Temp.	230ºC
Bake Temp	270ºC
Sparge Vessel Bake Temp.	120ºC
Bake Time	8
Bake Flow	40mL/min
Purge and Trap Auto-Sampler	**EST Centurion WS**
Sample Type	Water (Extraction Mode)
Sample Fill Mode	Syringe
Sample Volume	10mL
IS Volume	5µl
Needle Rinse Time	20 sec
Needle Sweep Time	20 sec
Concentrator Desorb Time	0.5 min
Syringe Rinse	On/12mL
Number of Syringe Rinses	2
Sparge Rinse Time	Off
Water Heater Temp.	85ºC

Table 2: Purge and Trap Water Parameters

The USEPA Method 8260 standards were acquired from Restek. The linear range of the water extraction experiment was established by running a nine point calibration curve with a range of 0.5 to 200ppb. Method detection limits were also established for each compound by examining seven replicate standards of the low calibration point. Finally, precision and accuracy were determined by running seven replicate midpoint standards. Experimental Results are listed in Table 3.

Compound	Curve %RSD	Curve Ave. RF	MDL	Precision (%RSD 50ppb)	Accuracy Ave. 50ppb Recovery
Dichlorodifluoromethane	8.39	0.681	0.18	3.19	104.57
Chloromethane	9.74	1.436	0.12	2.12	100.67
Vinyl Chloride	8.59	1.529	0.09	1.41	112.97
Bromomethane	10.19	1.429	0.18	1.20	98.16
Chloroethane	6.12	0.622	0.23	4.61	106.65
Trichlorofluoromethane	5.04	1.207	0.13	1.96	102.31
diethyl ether	4.39	0.593	0.15	3.06	103.62
1,1,2-trichlorofluoroethane	3.9	0.731	0.19	1.68	102.41
1,1-Dichloroethene	4.26	0.680	0.10	2.02	102.43
Acetone	12.08	0.329	0.57	3.55	96.00
Iodomethane	*0.999	0.540	0.10	3.31	98.65
Carbon Disulfide	5.92	2.143	0.18	1.35	101.99
allyl chloride	6.59	0.997	0.18	1.87	103.38
Methylene Chloride	5.89	0.800	0.14	1.97	99.36
acetonitrile	11.85	0.092	0.37	5.85	93.77
Tert Butyl Alcohol	9.98	0.144	1.09	3.46	102.11
MTBE	4.96	2.449	0.07	2.78	105.59
cis-1,2-Dichloroethene	5.9	0.909	0.08	2.05	103.58
acrylonitrile	4.62	0.339	0.28	3.58	103.67
Isopropylether	8.98	2.315	0.10	2.03	107.06
Vinyl acetate	9.98	2.712	0.04	2.67	95.55
1,1-Dichloroethane	7.7	1.497	0.10	1.94	105.28
Ethyl Tert Butyl Ether (ETBE)	3.87	2.462	0.09	2.51	104.93
trans-1,2-Dichloroethene	4.26	0.680	0.10	2.02	102.43
ethyl acetate	6.41	0.141	0.23	2.10	96.26
2-Butanone	4.23	1.428	0.14	3.16	100.25
2,2-Dichloropropane	11.39	1.131	0.14	4.08	101.61
Bromochloromethane	5.99	0.503	0.09	1.80	102.76
propionitrile	9.02	0.156	0.21	2.28	104.45
methacrylonitrile	4.23	0.519	0.12	3.00	101.44
THF	11.84	0.326	0.35	3.41	93.23
Chloroform	4.79	1.455	0.18	1.57	104.32
methyl acrylate	2.9	0.873	0.13	2.50	103.84
Dibromofluoromethane SUR	2.74	0.802	0.11	2.02	100.97
1,1,1-Trichloroethane	4.04	1.256	0.08	1.36	105.53
2-Chloroethylvinylether	8.86	0.342	0.18	2.89	120.49
Carbon Tetrachloride	12.96	1.038	0.09	1.84	109.80
1,1-Dichloropropene	8.11	1.145	0.13	2.06	106.95
methyl acetate	6.01	0.859	0.10	2.50	102.39
Tert Amyl Methyl Ether (TAME)	5.67	2.370	0.07	2.80	107.09
Benzene	5.52	3.329	0.05	1.52	104.94
1,2-Dichloroethane	4.52	1.244	0.09	2.06	103.94
propyl acetate	6.23	0.884	0.13	1.63	105.03
Trichloroethene	6.86	0.328	0.10	0.57	103.48
1,2-Dichloropropane	10.27	0.544	0.11	1.25	105.25
methyl methacrylate	9.17	0.474	0.08	1.82	111.39
Dibromomethane	6.64	0.351	0.11	1.40	103.25
Bromodichloromethane	9.86	0.745	0.08	1.07	109.68
2-nitropropane	5.53	0.206	0.23	1.88	105.34
cis-1,3-Dichloropropene	10.18	0.824	0.08	1.05	108.02
4-methyl-2-pentanone	3.11	0.621	0.08	1.98	104.54
Toluene-d8 SUR	9.15	2.026	0.09	1.10	108.73
Toluene	8.6	1.383	0.09	0.94	110.91
ethyl methacrylate	10.93	0.699	0.07	2.13	112.74
trans-1,3-Dichloropropene	11.07	0.803	0.12	0.90	111.66
1,1,2-Trichloroethane	7.42	0.478	0.17	1.77	106.88
Tetrachloroethene	11.34	0.641	0.22	0.96	125.02
1,3-Dichloropropane	6.91	0.833	0.10	1.45	106.40
isopropyl acetate	10.84	0.141	0.23	1.85	109.14
butyl acetate	8.25	0.395	0.22	2.14	104.57
Dibromochloromethane	12.28	0.51	0.11	1.54	114.85
2-Hexanone	4.51	0.469	0.25	2.21	103.87
1,2-Dibromoethane	6.93	0.5	0.12	1.30	106.64
Chlorobenzene	6.12	1.409	0.11	0.99	103.75
1,1,1,2-Tetrachloroethane	12	0.492	0.15	1.62	113.41
Ethylbenzene	8.85	2.499	0.12	1.10	110.51
Xylene (m+p)	11.12	1.945	0.21	1.11	113.70
Styrene	13.39	1.559	0.09	1.27	115.55
Xylene (o)	10.51	1.968	0.08	1.26	112.84
n-amyl acetate	8.03	0.887	0.08	2.12	112.03
Bromoform	12.14	0.348	0.11	1.90	112.12
Isopropylbenzene	11.49	2.391	0.11	1.49	110.51
cis-1,4-dichloro-2-butene	13.82	0.21	0.18	1.74	116.59
BFB SUR	8.61	1.101	0.15	1.95	99.76
Bromobenzene	5.18	1.798	0.16	4.24	103.89
1,2,3-Trichloropropane	7.76	0.818	0.11	5.56	100.86
1,1,2,2-Tetrachloroethane	6.76	0.99	0.07	2.55	100.40
n-Propylbenzene	7.36	4.695	0.09	1.83	105.83
trans-1,4-dichloro-2-butene	6.24	0.355	0.23	2.30	103.67
2-Chlorotoluene	4.95	0.941	0.12	1.76	100.84
4-Chlorotoluene	6.89	1.01	0.15	1.73	101.21
1,3,5-Trimethylbenzene	10.58	3.206	0.09	1.94	106.94
tert-Butylbenzene	11.97	2.827	0.11	1.98	103.40
sec-Butylbenzene	8.54	0.852	0.17	2.32	102.89
1,2,4-Trimethylbenzene	9.52	3.286	0.13	1.88	106.27
nitrobenzene	*0.998	0.068	0.69	4.98	94.35
1,3-Dichlorobenzene	7.31	1.915	0.12	1.64	100.26
1,4-Dichlorobenzene	10.59	1.999	0.18	2.04	96.53
Isopropyltoluene	13.34	3.486	0.11	1.91	107.66
1,2,-Dichlorobenzene	8.27	1.841	0.14	2.09	99.17
n-Butylbenzene	10.94	3.176	0.13	1.88	109.27
1,2-Dibromo-3-chloropropane	11.97	0.219	0.35	3.12	107.17
1,2,4-Trichlorobenzene	10.62	1.275	0.13	2.12	95.00
Naphthalene	10.87	3.271	0.12	2.69	95.76
Hexachlorobutadiene	7.03	0.529	0.13	2.77	100.38
1,2,3-Trichlorobenzene	11.84	1.125	0.10	2.35	97.25
Ave.	8.12	1.194	0.16	2.17	104.86

Table 3: Experimental Results

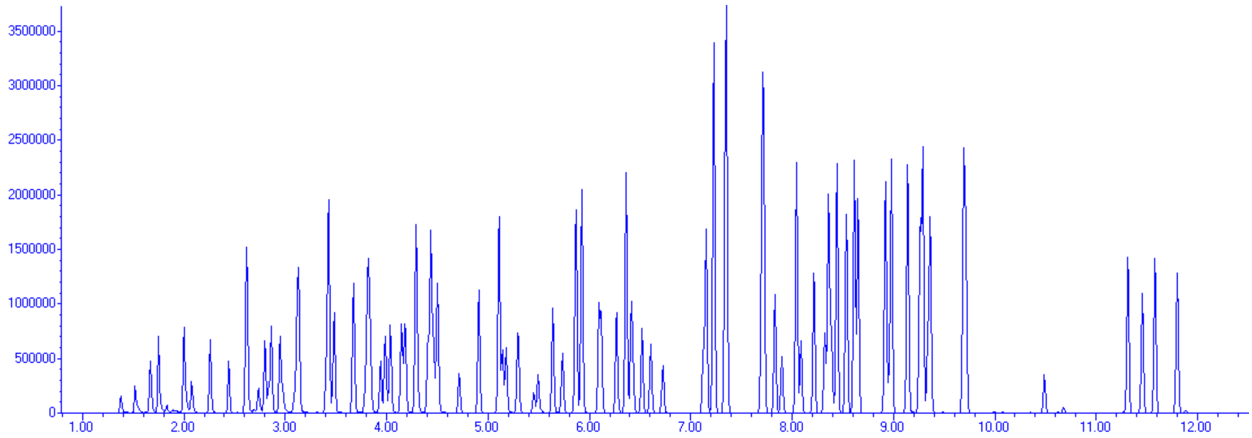


Figure 1: 50ppb Chromatogram of a Water Sample Run in Soil Mode

Conclusion:

The patent pending sampling process of the Centurion WS proved to be a reliable and accurate sampling method for running water samples in the soil mode. The curve linearity and method detection limits both met USEPA Method 8260 requirements, and the precision and accuracy results were excellent. The water extraction option offered with the Centurion WS will save laboratories time and money because only one set of standards, curves, etc. is required for both water and soil samples.

References:

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); United States Environmental Protection Agency Method 8260B, Revision 2, December 1996.

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