



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

Application Note

Environmental

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Abstract

Environmental laboratories are always searching for techniques to increase productivity. However, environmental methods demand a large amount of background information in order to ensure sampling and analysis is compliant. The determination of Volatile Organic Compounds (VOCs) in the United States Environmental Protection Agency (USEPA) Method 8260 encompasses many different matrices and in doing so has numerous sampling, quality control, and calibration requirements. The matrices of Method 8260 samples can also vary from air to sludge and from clean to extremely contaminated. Furthermore, the method has an extensive calibration range. All of these factors play into the complexity of sampling and analysis thus, creating ways to streamline sampling while still maintaining sample integrity and method compliance is always of interest to environmental laboratories.

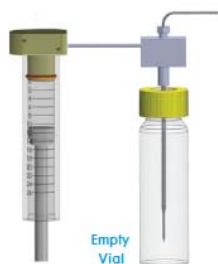
Introduction:

In order to run Method 8260 samples, a bromofluorobenzene (BFB) tune standard, matrix blank, calibration standard, and laboratory control standard all have to be run and pass method requirements every twelve hours. With the exception of the BFB standard, the other standards and blank matrices have to correspond to the samples to be analyzed. Due to this constraint, the analyst is required to run another three samples if the matrix is changed within the twelve hour time window. Moreover, the calibration must also match the sample matrix. These requirements are very time consuming and can limit laboratory profits. 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 and standards for waters and soils.

Discussion:

There are a number of techniques that can enable an analyst to save time in an environmental lab. Analysts can decrease analytical trap bake times, change sample purge time and flow, utilize a dual sampling system for one GC/MS, etc. However, one of the most basic ways to save time is by reducing the number of required curves and standards needed.

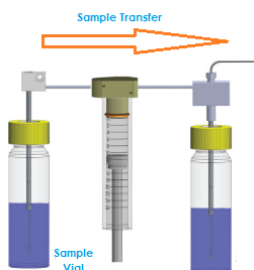
EST Analytical has designed a patent pending, automated water sampling mode that will allow environmental laboratories to run water samples in the soil mode. Thus, water and soil curves, standards, blanks and samples can use the same sampling parameters. (See steps 1, 2, and 3 below). This innovative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



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 Vocab 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 1.4 μ m column. The experiments were run using the extraction mode of the Centurion WS. Refer to Table 1 for the sampling method parameters and Table 2 for GC/MS parameters.

Purge and Trap Concentrator	EST Encon Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	150°C
Transfer Line Temp.	150°C
Trap Temp.	35°C
Moisture Reduction Trap (MoRT) Temp.	39°C
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.	260°C
Moisture Reduction Trap (MoRT) Bake Temp.	230°C
Bake Temp	270°C
Sparge Vessel Bake Temp.	120°C
Bake Time	8
Bake Flow	40mL/min
Purge and Trap Auto-Sampler	EST Centurion WS
Sample Type	Water Extraction
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.	85°C

Table 1: Purge and Trap Experimental Parameters

GC/MS	Agilent 7890A/5975 inert XL
Inlet	Split/Splitless
Inlet Temp.	200°C
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	45°C hold for 1 min., ramp 15°C/min to 220°C, hold for 1.3 min.
Column Flow Rate	1.0mL/min
Gas	Helium
Total Flow	44.00mL/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 35-265
Scans	3.12 scans/sec
Solvent Delay	0.7 min

Table 2: GC/MS Experimental 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. Figure 1 displays a 50ppb Method 8260 standard chromatogram of a water sample run in soil mode and experimental results are listed in Table 3.

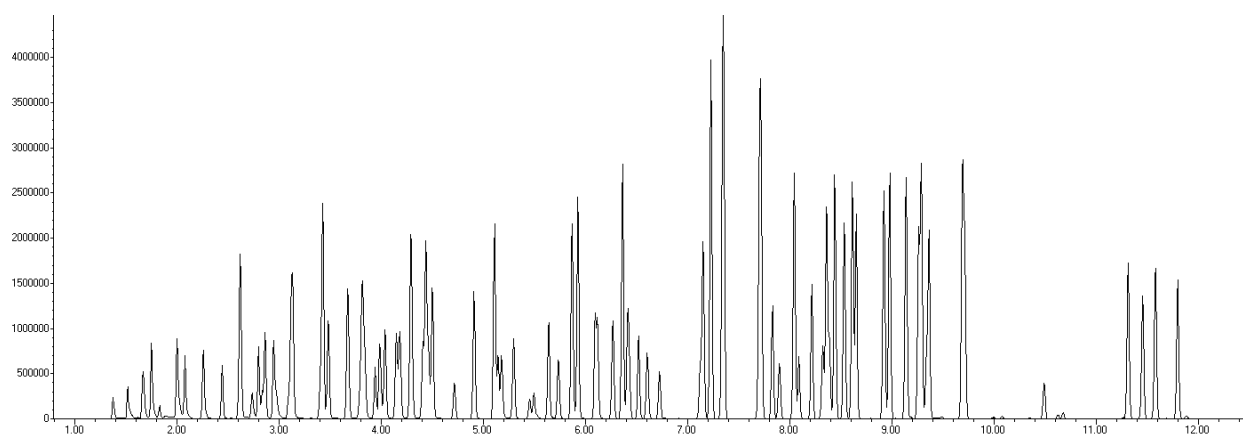


Figure 1: 50ppb Chromatogram of a Water Sample Run in Soil Mode

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

Table 3: Experimental Results

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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