Analysis of Volatile Organic Compounds in Soil Samples Using US EPA Method 8260 by CDS 8500 Automated Purge and Trap Concentrator

Application Note
Environmental

Author:

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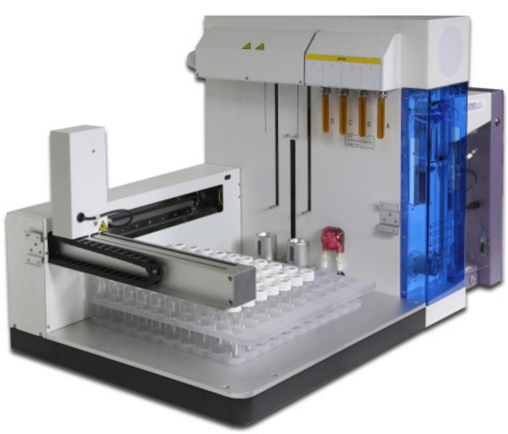
Abstract
The analytical performance of the CDS 8500 Series Purge and Trap is demonstrated for the EPA Method 8260D by GC/MS analysis.

Introduction
CDS Analytical's 8500 Series Purge and Trap System is a new, fully automated Purge and Trap concentrator for the trace measurement of purgeable volatile organic compounds (VOCs) in water. The standard for compliance for water testing is the U.S. EPA method 8260 for VOCs in soil. In this application note, data is presented to demonstrate that the 8500 Series Purge and Trap System meets the soil testing criteria set forth by US EPA method 8260D.

Experimental Setup
The 8500 Series Purge and Trap System was used to process the soil samples. The Purge and Trap method parameters are shown in Table 1, which are standard for the analysis of VOCs defined in the EPA Method 8260D.

Valve Oven Temperature	130°C
Transfer Line Temperature	130°C
Standby Flow	10 mL/min
Trap Ready Temperature	35°C
Wet Trap Ready Temperature	45°C
Water Addition Volume	5 mL
Purge Time	11 min
Purge Flow	40 mL/min
Purge Temperature	40°C
Dry Purge Time	2 min
Dry Purge Flow	200 mL/min
Dry Purge Temperature	35°C
Desorb Parameters:	
Desorb Preheat Temperature	245°C
Desorb Time	4 min
Desorb Drain Flow	250 mL/min
Desorb Temperature	250°C
Bake Parameters:	
Bake Time	4 min
Bake and Vessel Flow	200 mL/min
Trap Bake Temperature	260°C
Wet Trap Bake Temperature	260°C

Table 1. Purge and Trap Method Parameters.



A Shimadzu single quad GCMS-QP 2010 was used. GC/MS conditions are listed in Table 2. Carrier gas was supplied to the 8500 Series Purge and Trap and a heated transfer line from the 8500 Series Purge and Trap concentrator was plumbed into the carrier supply line of the split/spitless inlet.

Analytical Column	RTX-VMS (30 m × 25 mm × 140 µm)
Injector Temperature	240°C
Carrier Gas	He at 1.00 mL/min
Split Ratio	40:1
Oven Program	35°C Hold 4 min 90°C at 5°C/min 100°C at 12°C/min 220°C at 30°C/min Hold 2.67min
Mass Spectrometer:	
Interface Temperature	220°C
Ion Source Temperature	200°C
Scan Mode and Range	Full scan 35-260m/z
Scan Time	0.3 min
Scan Speed	833

Table 2. GCMS Conditions.

The following calibration mixes were purchased from Restek for performing method 8260D on the 8500 series: 8260B MegaMix®, VOA, 502.2, California Oxygenates, and 8260B acetates mixes. From those standards, a stock mixture of 200 µg/L was prepared. All other calibration standards were prepared from this stock solution. In total, six calibration standards were prepared between 5 and 200 µg/L, with the exception of those targets noted in Table 4. The internal standard was a 4 component 8260 internal standard mix, and the 8260 surogates was a 3 component mix. Both were diluted to a concentration of 25 µg/mL. 5 µL of this internal standard was added to each sample by an onboard EPC on the 8500 autosampler (4 total reservoirs supported).

Results and Discussion

Figure 1 depicts the Total Ion Chromatogram (TIC) of a 50 µg/L calibration standard with internal standard and surrogates. All of the analytes are adequately resolved chromatographically. The chromatogram of the 6 gases is shown in Figure 2.

5 µL of the pre-mixed internal standard solution was precisely introduced to each water sample by the autosampler through an onboard EPC. The %RSD for all 4 internal standards was calculated from 6 replicate measurements and are shown in Table 3. The %RSDs are less than 5% showing high reproducibility of the internal standard addition EPC of the 8500 autosampler. Figure 4 shows the overlay of the 6 extracted ion chromatograms for each of the internal standards.

The data summary is shown in Table 4 and lists the results for Retention Time (RT), Average Relative Response Factors (Avg RF), Percent Relative Standard Deviation (% RSD) of the initial calibration, Method Detection Limits (MDL), along with method accuracy as and % RSD. All analytes exceed the EPA 8260D method requirements. The MDL were determined by analyzing replicate samples at a concentration of 5.0 µg/L.

Table 3. Internal standard components and their %RSDs (n=6).

Internal Standard Compound	%RSD
pentafluorobenzene	4.1
1,4-difluorobenzene	4.3
chlorobenzene-d5	4.7
1,4-dichlorobenzene-d4	4.6

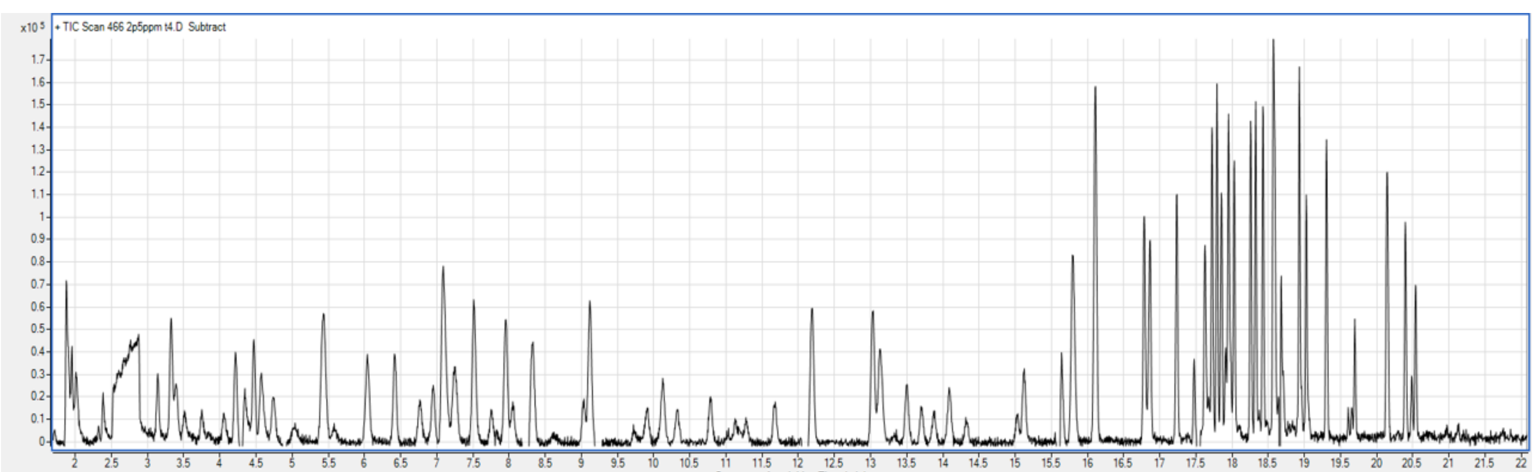


Figure 1. TIC of 8260D volatile organic standard mix at 50 µg/L.

Conclusion

The 8500 Series Purge and Trap System meets and exceeds the EPA Method 8260D over a concentration range from 5 to 200 µg/L. Although the 8500 series supports both water and soil sampling, only the capabilities for soil analysis are demonstrated here. The new 8500 series utilitizes and onboard EPC to deliver internal standard to the sample at RSD <5% for soil samples. This system helps support users in that all EPA regulatory requirements are meet to help save time and money.

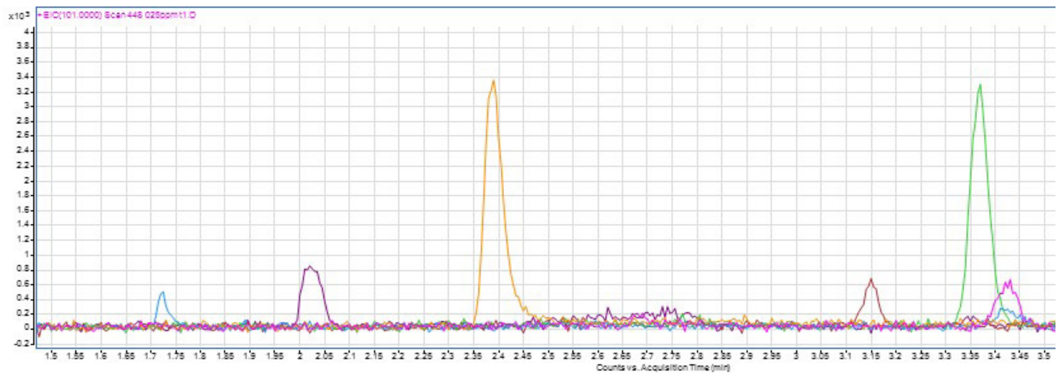


Figure 2. Extracted ion chromatograms of the first 6 gases in the chromatogram at 2 µg/L.

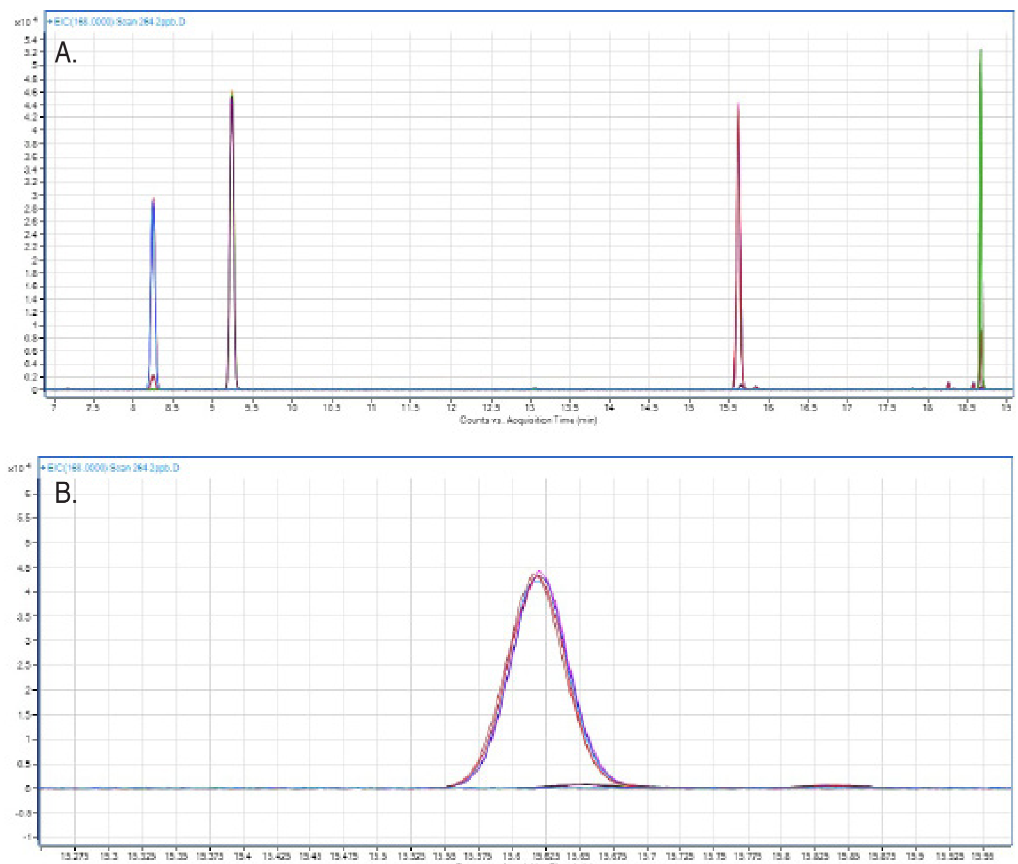


Figure 3. A) Overlay of 6 replicates of the internal standard mixture containing (from left to right) pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4. (B) Magnified chromatogram of chlorobenzene-d5

Table 3. Summary of the performance for each compound. At 5 μg/L, the accuracy and %RSD were also determined. For all compounds, n=4.

Compound	Avg. RF	MDL (μg/L)	Concentration (μg/L)	RSD (%)	Accuracy (%)
dichlorodifluoromethane	0.66	1.36	4.49	9.65	89.82
chloromethane	4.92	4.15	5.49	24.04	109.79
vinyl chloride	2.52	1.07	5.63	6.07	112.6
trichlorofluoromethane	0.36	0.68	5.63	3.84	112.63
diethyl ether	1.51	1.07	5.40	6.33	108.07
carbon disulfide	5.15	0.41	5.64	2.30	112.86
1,1-dichloroethene	2.74	0.77	5.42	4.53	108.41
1,1,2-trichlorotrifluoroethane	1.75	0.97	5.51	5.62	110.17
iodomethane	2.19	2.18	5.81	11.95	116.10
allyl chloride	2.10	3.00	10.35	9.22	103.54
methylene chloride	4.32	1.35	5.43	7.89	108.52
trans-1,2-dichloroethene	2.96	3.09	6.63	14.81	132.63
methyl acetate	3.30	1.15	5.49	6.68	109.83
acetone[1]	7.38	1.80	13.58	4.21	109.50
tert-butanol	5.01	1.28	6.12	6.65	122.47
acetonitrile	3.56	1.89	5.87	10.22	117.47
diisopropyl ether	8.50	0.55	5.51	3.18	110.26
chloroprene	3.88	2.45	5.38	14.51	107.52
1,1-dichloroethane	1.07	1.07	4.82	7.03	96.500
acryonitrile	2.12	1.67	5.93	8.97	118.6
vinyl acetate	0.47	0.98	4.94	6.29	98.80
ethyl-tert-butyl ether	3.18	1.17	5.57	6.66	111.49
cis-1,2-dichloroethene	3.24	0.76	5.47	4.41	109.42
bromochloromethane	1.11	1.46	5.54	8.4	110.85
chloroform	3.88	0.45	5.47	2.61	109.44
carbon tetrachloride	1.26	1.17	6.07	6.14	121.35
methyl acrylate	4.63	2.26	6.03	11.92	120.58
tetrahydrofuran	0.93	1.32	5.51	7.62	110.25
ethyl acetate	2.85	0.53	5.79	2.90	115.90
dibromofluoromethane (SURR)					
2-butanone[1]	9.29	10.75	14.78	23.3	118.16
1,1-dichloropropene	3.74	1.24	5.51	7.19	110.10
benzene	6.72	0.60	5.50	3.48	109.99
propionitrile	0.39	3.61	5.21	22.04	104.18
methacryonitrile	0.99	1.41	5.50	8.17	110.10

[1]Calibration range is bewteen 5 and 500 μg/L

Compound	Avg. RF	MDL (μg/L)	Concentration (μg/L)	RSD (%)	Accuracy (%)
pentafluorobenzene (IS)					
tert-amyl-methyl ether	1.50	2.52	6.09	13.17	121.7
1,2-dichloroethane	1.53	0.48	5.57	2.76	111.34
isopropyl acetate	0.83	1.30	5.54	7.49	110.74
1,4-difluorobenzene (IS)					
trichloroethene	2.75	0.82	5.55	4.72	111.04
dibromomethane	0.68	0.61	5.39	3.61	107.84
1,2-dichloropropane	1.78	0.88	5.49	5.09	109.88
bromodichloromethane	1.46	0.71	5.66	4.00	113.11
mehtyl methacrylate	1.08	1.20	5.67	6.75	113.31
propyl acetate	0.68	1.71	5.49	9.89	109.89
cis-1,3-dichloropropene	1.51	0.52	5.68	2.90	113.59
toluene-d8 (SURR)					
toluene	4.92	0.89	5.55	5.10	110.91
2-nitropropane	0.44	1.54	3.74	13.10	74.80
tetrachloroethene	2.41	0.62	5.53	3.56	110.61
4-methyl-2-pentanone	0.75	5.43	13.98	12.36	111.86
trans-1,3-dichloropropene	0.89	0.92	5.69	5.17	113.75
1,1,2-trichloroethane	1.19	0.96	5.45	5.59	108.92
ethyl methacrylate	1.51	10	5.66	5.61	113.24
dibromochloromethane	1.10	0.34	5.74	1.87	114.82
1,3-dichloropropane	2.23	0.78	5.58	4.46	111.58
1,2-dibromoethane	1.08	0.6	5.57	3.41	111.30
butyl acetate	0.31	1.63	5.67	9.17	113.30
2-hexanone[1]	2.52	2.48	14.93	5.30	119.49
chlorobenzene-d5 (IS)					
chlorobenzene	4.29	0.85	5.53	4.88	110.66
ethylbenzene	7.65	0.69	5.56	3.95	111.10
1,1,1,2-tetrachloroethane	1.24	0.78	5.65	4.4	112.91
m,p-xylene	5.99	0.66	5.56	3.75	111.29
o-xylene	2.96	0.65	5.53	3.73	110.57
bromoform	1.22	1.26	5.71	6.99	114.22
styrene	8.99	0.70	5.63	3.98	112.57
amyl acetate	1.89	2.34	5.53	13.49	110.52
1-bromo-4-fluorobenzene (SURR)					
bromobenzene	3.52	0.99	5.53	5.68	110.61

Compound	Avg. RF	MDL (μg/L)	Concentration (μg/L)	RSD (%)	Accuracy (%)
cis-1,4-dichloro-2-butene	1.14	1.55	5.71	8.62	114.2
isopropylbenzene	11.13	0.53	5.44	3.12	108.84
n-propylbenzene	17.65	0.71	5.55	4.07	111.04
1,1,2,2-tetrachloroethane	1.29	6.22	5.02	39.46	100.37
2-chlorotoluene	10.66	0.69	5.53	3.95	110.66
1,2,3-trichloropropane	2.73	0.66	5.54	3.77	110.73
1,3,5-trimethylbenzene	11.11	0.67	5.42	3.92	108.42
trans-1,4-dichloro-2-butene	1.26	0.96	5.80	5.28	115.95
4-chlorotoluene	11.08	0.54	5.54	3.10	110.74
tert-butylbenzene	10.70	0.67	5.52	3.84	110.49
1,2,4-trimethylbenzene	11.21	0.93	5.55	5.35	110.99
sec-butylbenzene	15.32	0.82	5.51	4.75	110.25
p-isopropyltoluene	13.70	0.62	5.52	3.58	110.36
1,3-dichlorobenzene	7.24	0.61	5.49	3.55	109.79
1,4-dichlorobenzene-d4 (IS)					
1,4-dichlorobenzene	7.67	0.62	5.49	3.62	109.87
n-butylbenzene	12.64	0.76	5.52	4.38	110.38
1,2-dichlorobenzene	6.88	0.64	5.43	3.76	108.63
1,2-dibromo-3-chloropropane	0.61	1.68	5.63	9.51	112.51
nitrobenzene	0.08	6.44	5.10	40.19	102.03
hexachlorobutadiene	2.39	0.84	5.40	4.95	108.03
1,2,4-trichlorobenzene	3.64	0.73	5.35	4.35	107.05
naphthalene	10.42	0.92	5.46	5.39	109.10
1,2,3-trichlorobenzene	3.08	1.55	5.18	9.53	103.62