A New Rapid, Simple and Efficient Extraction Method of Semi Volatile Organic Compounds from Soil Matrices

Richard Karbowski [1]; Alan Owens[1]; Nicole Lock[1] ;Ruth Marfil-Vega[1] ;Alicia Stell[2]; Benedict Liu[2] ;Candace Cashman[2]
[1]Shimadzu Scientific Instruments, Inc., Columbia, Maryland; [2] CEM Corporation, Matthews NC

1. Introduction

The analysis of semi volatile organic compounds (SVOCs) in soil matrices and other environmental solid samples encompasses two processes defined under the SW-846 methods: sample extraction and quantification of the analytes of interest. Due to the persistent nature of these compounds there is a constant concern of SVOCs in our soil, and the environment as a whole. The extraction of SVOCs from solid matrices can be challenging given the vast classes of chemicals in this group and sample types. Widely implemented techniques do not offer an extraction method that is rapid, simple, and efficient, which are all crucial to the modern environmental lab. The EDGE extraction system offers a new take on the processes of pressurized fluid extraction in adherence to US EPA 3545A. The Shimadzu GCMS-2020NX is top of the line Single quadrupole GCMS system able to reach detection limits into the low parts per billion levels for most analytes in EPA 8270. This work demonstrates the performance of the system for the analysis of selected solid matrices.

2. Experimental approach

In this study, over 80 SVOCs listed in method EPA 8270D were extracted in different sample types, including sand , sandy loam and clay with the EDGE extraction system and analyzed with Shimadzu's GCMS-QP2020NX. The combination of the two instruments results in a rapid simple and efficient workflow of a variety of compounds from different soil types, all with one method. The Shimadzu GCMS-QP2020NX Single quadrupole mass spectrometer was used, in split mode and Constant Linear Velocity Flow control mode. The general procedure for sample extraction is as follows: 30 g of sample along with 20 g of sodium sulfate were weighed and added to the Edge. Unspiked and spiked samples were extracted (with a combination of hexane:acetone) and further analyzed by GCMS.

Figure 1. The Shimadzu GCMS-QP2020NX.

Figure 2. The CEM EDGE Extraction System.

		Calibration		
		R^2	Mean RF	RF %RSD
1	N-nitrosodimethylamine	0.9993	0.73	8.27
2	Pyridine	0.9988	0.87	36.61
3	2-fluorophenol (surrogate)	0.9995	1.22	5.00
4	Phenol-D6 (surrogate)	0.9998	1.34	6.04
5	Phenol	0.9997	1.44	4.49
6	Aniline	0.9996	1.72	5.86
7	Bis(2-chloroethyl) ether	0.9996	1.23	8.25
8	2-chlorophenol	0.9996	1.01	4.63
9	1,3-Dichlorobenzene	0.9998	1.01	12.39
10	1,4-Dichlorobenzene	0.9997	1.03	11.77
11	Benzyl alcohol	0.9999	0.82	4.15
12	1,2-Dichlorobenzene	0.9997	0.96	10.57
13	2-methylphenol	0.9999	1.26	2.52
14	Bis(2-chloro-1-methylethyl) ether	0.9999	2.40	10.41
15	N-nitrosodi-n-propylamine	0.9999	0.69	7.47
16	3-methylphenol/4-methylphenol	0.9999	1.30	3.99
17	Hexachloroethane	0.9999	0.44	8.29
18	Nitrobenzene-D5 (surrogate)	0.9995	0.30	6.88
19	Nitrobenzene	0.9995	0.32	7.61
20	Isophorone	0.9998	0.70	4.83
21	2-nitrophenol/4-nitrophenol	0.9980	0.09	17.37
22	2,4-dimethylphenol	0.9997	0.46	4.41
23	bis(2-chloroethoxy)methane	0.9998	0.54	9.16
24	2,4-dichlorophenol	0.9996	0.21	8.66
25	1,2,4-trichlorobenzene	0.9999	0.24	11.21
26	Napthalene	0.9999	1.32	9.17
27	4-chloroaniline	0.9997	0.48	6.57
28	Hexachlorobutadiene	0.9996	0.12	8.96
29	4-Chloro-3-methylphenol	0.9998	0.37	7.18
30	1-Methylnapthalene	0.9991	0.75	10.62
31	2-Methylnaphthalene	0.9994	0.66	11.59
32	Hexachlorocyclopentadiene	0.9989	0.17	8.41
33	2,4,6-trichlorophenol	0.9998	0.20	13.20
34	2,4,5-trichlorophenol	0.9996	0.22	15.46
35	2-fluorobiphenyl (surrogate)	0.9994	1.32	11.82
36	2-Chloronaphthalene	0.9998	1.20	13.35
37	2-Nitroaniline	0.9976	0.23	14.46
38	1,4-dinitrobenzene	0.9934	0.11	19.33
39	Dimethyl phthalate	0.9995	1.17	9.79
40	1,3-dinitrobenzene	0.9974	0.12	20.88
41	2,6-dinitrotoluene	0.9945	0.15	24.16
42	Acenaphthylene	0.9991	1.82	6.10
43	3-nitroaniline	0.9993	0.35	10.17
44	Acenaphthene	0.9992	1.34	13.87
45	2,4-dinitrophenol	0.9997	0.03	40.91
46	Dibenzofuran	0.9995	1.69	12.10
47	2,4-Dinitrotoluene	0.9995	0.17	22.37
48	2,3,4,5-tetrachlorophenol	0.9990	0.13	21.74
49	2,3,4,6-tetrachlorophenol	0.9986	0.13	19.81
50	Diethyl Phthalate	0.9996	1.15	5.98
51	Fluorene	0.9996	1.35	11.88
52	4-Chlorophenylphenyl ether	0.9984	0.61	12.78
53	4-Nitroaniline	0.9996	0.25	19.24
54	2-methyl, 4,6-dinitrophenol	0.9999	0.38	28.22
55	Diphenylamine	0.9997	0.71	5.57
56	Azobenzene	0.9993	1.43	4.76
57	Benzophenone	0.9989	0.49	2.91
58	2,4,6-Tribromophenol (surrogate)	0.9995	0.07	17.21
59	4-bromophenyl phenyl ether	0.9995	0.23	11.82
60	Hexachlorobenzene	0.9999	0.22	15.90
61	Pentachlorophenol	0.9894	0.03	27.75
62	Phenanthrene	0.9997	1.22	17.04
63	Anthracene	0.9992	1.12	8.59
64	Carbazole	0.9993	1.06	5.60
65	Dibutyl phthalate	0.9997	1.14	9.46
66	Fluoranthene	0.9999	1.10	5.79
67	Pyrene	0.9994	1.40	6.30
68	p-Terphenyl-d14	0.9993	0.96	8.92
69	Butyl benzyl phthalate	0.9977	0.44	19.15
70	bis (2-ethylhexyl) adipate	0.9980	0.52	24.05
71	di-n-octyl phthalate	0.9984	0.61	25.57
72	Benzo(a)anthracene	0.9998	1.11	4.09
73	Chrysene	0.9996	1.20	12.81
74	bis(2-ethylhexyl) phthalate	0.9950	0.90	31.78
75	Benzo(b)fluoranthene	0.9988	1.02	4.80
76	Benzo(k)fluoranthene	0.9997	1.13	2.47
77	Benzo(a)pyrene	0.9995	0.91	10.40
78	Indeno[1,2,3-cd]fluoranthene	0.9990	0.75	13.79
79	Dibenz[a,h]anthracene	0.9995	0.92	4.38
80	Benzo[g,h,i]perylene	0.9987	0.94	3.51

3. Results

The results from the calibration in accordance to EPA 8270D are included in Table 1. The calibration curve results for most compounds allow for average response factor quantification. Quantification can be performed if %RSD of the response factors are <20%. Any above this limit must be calculated with a linear or quadratic fit, and in these cases the R2 is above 0.99. The recoveries of 10 ppb CCVs for most compounds are within 80-120% (6 replicates analyzed during the performance demonstration experiments). The vast majority of CCVs ran at a 2ppb level are also between 80-120%. These are both plotted in Figure 3, 10ppb with green and 2 ppb with orange.

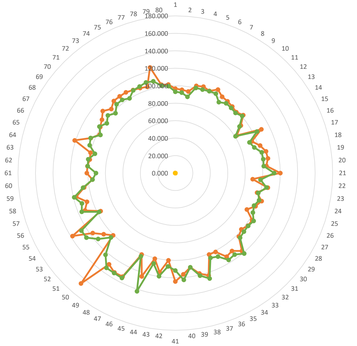

Figure 3. % recovery data for CCVs, 6 replicates

Table 1. Calibration curve results EPA 8270.

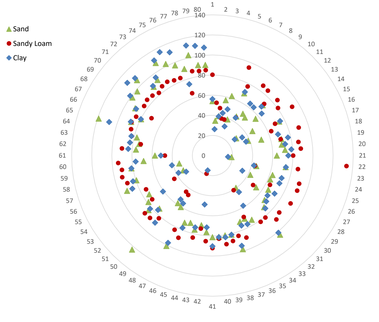

Figure 4. % recovery data for 3 different matrices (n=6)

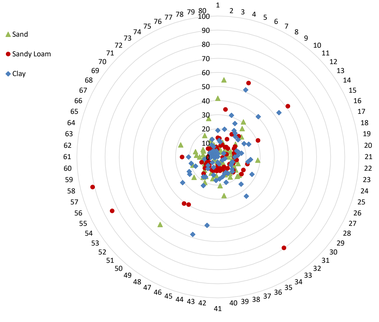

Figure 5. % RSD data for 3 different matrices (n=6)

Figures 4 and 5 summarize the results from a mid level spike calculated at 5ppm in the final extract in both sand, sandy loam and clay matrices. Both the recovery and reproducibility results from all targets are consistent with the acceptable ranges included in EPA 8270D.

4. Conclusions

The combination of the CEM automated extraction system with the Shimadzu GCMS-QP2020NX is the ideal solution for rapid and efficient extraction and analysis of various soil matrices. The data shows that the GCMS produces stable calibration and data, and the CEM EDGE produces good data for various oil matrices.

–The EDGE Automated Extraction System is able to extract up to 12 samples in one extraction batch and is able to perform these procedures 3 times faster than other pressurized fluid extractors, which includes filtering, cooling and washing. The Shimadzu GCMS- QP2020NX has excellent calibrations and reproducibility, as well as an improved DFTPP tuning algorithm to consistently pass DFTPP specifications for EPA 8270.. Further study on more challenging samples is expected in the future.