Decreasing Purge and Trap Cycle Times for USEPA Method 8260

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ABSTRACT

The United State Environmental Protection Agency (USEPA) Method 8260 is prescriptive in its purge and trap parameters. The method requires an eleven minute purge at 40ml/min purge flow. Due to this restriction, environmental analysts need to investigate other means to decrease cycle times without sacrificing analytical results. This application note will examine decreasing cycle times within the USEPA Method requirements.

INTRODUCTION

Environmental laboratories are always searching for new ways to decrease cycle times and run more samples. According to USEPA Method 8260D, a calibration verification standard, laboratory control standard and a blank need to be verified every 12 hours, these requirements not only limit the amount of samples that can be run, but also limits the time samples may be run between the required standards. Recent Gas Chromatograph/Mass Spectrometer (GC/MS) improvements have enabled analysts to decrease the amount of time it takes to examine the analytes outlined the Method 8260, however, the purge and trap sampling requirements have limited the ability to take advantage of these enhancements.

Method 8260 requires an eleven minute purge time thus other sampling parameters need to be explored in order to make cycle times shorter. However, when changing a parameter, consideration has to be taken into account as to how it affects the overall experimental results. This application will outline the optimum parameters for the EST Analytical Evolution purge and trap concentrator for the enhancement of sample cycle times.

EXPERIMENTAL

The EST Analytical Evolution purge and trap concentrator was configured to the Centurion WS Autosampler. The system was set up to run water samples. A Vocarb 3000 was employed for the analytical trap. A 30m x 0.25mm x 1.4µm Rxi Sil MS 624 column was installed in the GC for analyte separation while the MS was set to run in scan mode with a range of 35 to 300m/z. The parameters used for the experiment are outlined in Tables 1 through 3.

Table 1: GC/MS Parameters

Parameter	Setting
Inlet	Split/Splitless
Inlet Temp.	220°C
Inlet Head Pressure	7.77 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 20°C/min to 220°C, hold for 1.25 min, 11 min run time
Column Flow Rate	1mL/min
Gas	Helium
Total Flow	44mL/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 35-300
Scans	5.2 scans/sec
Solvent Delay	0.7 min

Table 2: Purge and Trap Concentrator Parameters

Purge and Trap Concentrator	EST Analytical Evolution
Trap Type	Vocarb 3000
Valve Oven Temp.	130°C
Transfer Line Temp.	130°C
Auto Sample Prep Time	0.4min
Trap Temp.	40°C
Moisture Reduction Trap (MoRT) Temp.	40°C
Purge Time	11 min
Purge Flow	40mL/min
Dry Purge Temp.	ambient
Dry Purge Flow	NA
Dry Purge Time	0.0 min
Desorb Pressure Control	On
Desorb Pressure	5psi
Desorb Time	0.5 min
Desorb Preheat Delay	5 sec
Desorb Temp.	250°C
Moisture Reduction Trap (MoRT) Bake Temp.	210°C
Bake Temp	260°C
Sparge Vessel Bake Temp.	110°C
Bake Time	3 min
Bake Flow	85mL/min

Table 3: Autosampler Parameters

Purge and Trap Auto-Sampler	EST Analytical Centurion WS
Sample Type	Water
Sample Fill Mode	Syringe
Sample Volume	5mL
Sample Prime Time	7 sec
Loop Equilibration Time	5 sec
Sample Transfer Time	10 sec
Syringe Rinse	On/20mL
Number of Syringe Rinses	1
Sample Loop Rinse	On/10 sec
Sample Loop Sweep Time	5 sec
Number of Sparge Rinses	Syringe/1
Rinse Volume	5mL
Rinse Transfer Time	15 sec
Rinse Drain Time	20 sec
Number of Foam Rinse Cycles	3
Water Heater Temp.	85°C
Internal Standard Vol.	5µl

USEPA Method 8260 standards were procured and used to make stock standard mixes. Next a nine point calibration curve was prepared with a range of 0.5 to 200µg/L. The calibration curve was run and once established, a method detection limit and precision and accuracy study was performed. The seven method detection samples were run over the course of three days using blank and low level calibration standards. A concentration of 50µg/L was employed for the precision and accuracy samples. The precision and accuracy study was also run over the course of three days with seven standards. All of the results were compiled and are listed in Table 4. A chromatogram of a 50µg/L standard is displayed in Figure 1.

Figure 1: Chromatogram of 50µg/L Standard

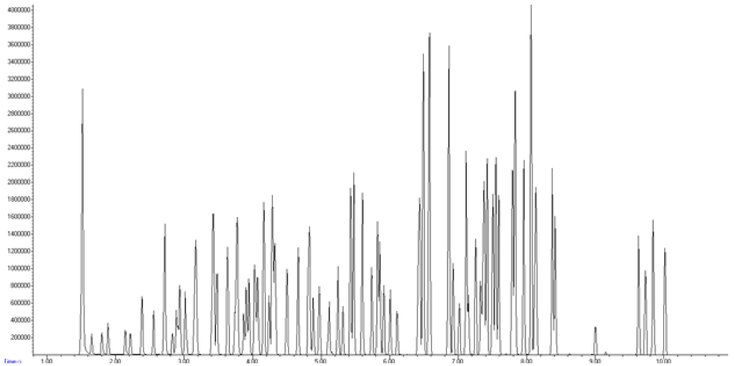

Table 4: Experimental Results Summary

Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry
Dichlorodifluoromethane	0.447	10.51	0.07	3.14	92.70
Chloromethane	0.591	4.89	0.11	1.43	86.92
Vinyl Chloride	0.701	5.74	0.07	2.52	93.84
Bromomethane	0.410	13.78	0.21	2.89	92.77
Chloroethane	0.465	5.49	0.07	2.35	94.07
Trichlorofluoromethane	0.652	8.35	0.11	1.78	98.71
diethyl ether	0.520	5.46	0.11	1.59	94.87
1,1,2-trichlorofluoroethane	0.652	8.35	0.11	1.78	98.71
1,1-Dichloroethene	0.622	6.29	0.09	1.95	97.85
Acetone	0.217	8.94	0.43	2.17	85.06
Iodomethane	0.565	14.09	0.09	3.97	104.68
Carbon Disulfide	1.613	6.03	0.07	1.82	97.17
allyl chloride	0.690	6.73	0.10	2.17	97.43
Methylene Chloride	0.689	5.76	0.07	1.79	92.02
Tert Butyl Alcohol	0.069	9.52	0.40	5.56	81.24
MTBE	2.098	4.51	0.03	1.42	92.78
cis-1,2-Dichloroethene	0.822	4.45	0.08	1.76	93.05
acrylonitrile	0.287	6.29	0.16	2.46	89.13
Vinyl acetate	2.205	4.04	0.02	1.59	95.25
Isopropyl Ether	1.941	3.45	0.04	1.85	94.33
1,1-Dichloroethane	1.257	4.21	0.08	1.68	96.78
Ethyl Tert Butyl Ether	2.049	4.24	0.03	1.67	92.18
Ethyl Acetate	0.109	5.70	0.17	1.13	90.77
trans-1,2-Dichloroethene	0.714	4.64	0.11	1.29	97.01
2-Butanone	1.061	6.26	0.07	1.68	87.16
2,2-Dichloropropane	1.003	6.12	0.08	3.16	96.93
Bromochloromethane	0.487	9.51	0.08	2.92	93.71
propionitrile	0.112	8.15	0.28	3.24	86.25
methacrylonitrile	0.457	9.38	0.08	1.93	86.11
THF	0.222	6.29	0.37	2.05	86.27
Chloroform	1.340	5.79	0.02	1.78	91.42
methyl acrylate	0.724	5.99	0.10	1.47	88.44
Dibromofluoromethane	0.767	11.69	0.07	1.74	87.56
1,1,1-Trichloroethane	1.225	5.99	0.09	1.62	95.73
2-Chloroethylvinylether	0.318	3.55	0.10	1.93	98.38
Carbon Tetrachloride	1.023	9.27	0.08	1.89	100.12
1,1-Dichloropropene	1.027	5.41	0.06	2.07	95.85
methyl acetate	0.641	6.51	0.06	1.65	90.30
isobutyl alcohol	0.026	13.92	1.68	5.29	84.02
Tert Amyl Methyl Ether	2.096	4.94	0.05	1.46	92.12
Benzene	2.885	4.43	0.02	1.51	93.57
1,2-Dichloroethane	1.069	12.55	0.05	1.56	86.91
propyl acetate	0.676	4.48	0.06	1.24	94.33
Trichloroethene	0.574	5.71	0.05	1.97	97.60
1,2-Dichloropropane	0.443	4.83	0.09	2.31	96.97
methyl methacrylate	0.380	6.17	0.05	1.37	94.91
Dibromomethane	0.370	8.04	0.06	1.73	93.95
Bromodichloromethane	0.635	4.41	0.07	2.02	100.49
2-nitropropane	0.152	9.29	0.14	2.02	95.18
cis-1,3-Dichloropropene	0.745	3.75	0.10	1.97	99.83
4-methyl-2-pentanone	0.471	5.35	0.06	1.78	91.71
Toluene-d8	1.764	5.10	0.03	2.07	97.27
Toluene	1.251	5.52	0.10	2.13	97.07

Table 4: Experimental Results Summary Continued

Compound	Curve RF	Curve %RSD	MDL	%RSD Precision	% Rec'ry
ethyl methacrylate	0.648	4.87	0.07	1.60	99.95
trans-1,3-Dichloropropene	0.688	3.89	0.04	2.28	99.58
1,1,2-Trichloroethane	0.445	6.41	0.07	1.86	94.01
Tetrachloroethene	0.523	5.15	0.09	1.81	97.42
1,3-Dichloropropane	0.734	5.88	0.04	2.25	95.51
isopropyl acetate	0.034	5.79	0.77	2.10	99.94
butyl acetate	0.330	6.92	0.07	1.55	94.21
Dibromochloromethane	0.508	6.26	0.04	1.91	102.13
2-Hexanone	0.334	3.98	0.15	1.65	92.55
1,2-Dibromoethane	0.482	7.52	0.04	1.44	95.08
Chlorobenzene	1.613	8.62	0.05	1.40	94.18
1,1,1,2-Tetrachloroethane	0.564	3.55	0.06	1.44	95.08
Ethylbenzene	2.652	5.45	0.05	1.55	98.97
Xylene (m+p)	2.033	6.48	0.06	1.59	98.36
Styrene	1.671	3.85	0.05	1.69	99.39
Xylene (o)	2.086	5.76	0.08	1.46	97.55
n-amyl acetate	0.834	3.91	0.06	1.13	97.42
Bromoform	0.403	10.49	0.06	1.18	106.07
Isopropylbenzene	2.651	6.11	0.08	1.76	99.00
cis-1,4-dichloro-2-butene	0.215	10.74	0.09	1.86	106.77
BFB	1.463	11.13	0.08	1.87	93.11
Bromobenzene	2.379	8.64	0.07	1.81	94.84
1,2,3-Trichloropropane	1.183	7.15	0.09	1.68	92.21
1,1,2,2-Tetrachloroethane	1.303	6.03	0.06	1.58	96.18
n-Propylbenzene	5.836	6.29	0.04	2.00	99.25
trans-1,4-dichloro-2-butene	.381	4.61	0.13	1.90	98.56
2-Chlorotoluene	1.260	7.89	0.05	1.77	95.21
4-Chlorotoluene	1.256	5.71	0.07	1.71	97.28
1,3,5-Trimethylbenzene	4.167	5.42	0.08	1.92	98.75
tert-Butylbenzene	3.859	6.40	0.06	1.89	98.77
sec-Butylbenzene	1.223	4.35	0.10	2.38	100.06
1,2,4-Trimethylbenzene	4.290	6.04	0.05	1.94	97.69
1,3-Dichlorobenzene	2.481	8.93	0.07	2.05	93.94
1,4-Dichlorobenzene	2.444	8.14	0.05	1.93	94.39
Isopropyltoluene	4.496	4.14	0.07	2.06	101.30
1,2,-Dichlorobenzene	2.271	7.79	0.07	2.13	94.67
n-Butylbenzene	3.718	4.63	0.07	2.18	102.07
1,2-Dibromo-3-chloroprop	0.261	8.51	0.09	2.31	97.43
1,2,4-Trichlorobenzene	1.425	6.79	0.06	2.01	99.88
Naphthalene	3.971	6.25	0.06	1.75	98.84
Hexachlorobutadiene	0.610	10.23	0.15	1.72	105.65
1,2,3-Trichlorobenzene	1.328	5.25	0.06	1.60	101.18

CONCLUSIONS:

Using the EST Analytical Evolution purge and trap concentrator, the cycle time for a water sample following the USEPA Method 8260D method was reduced to less than 17 minutes. The results met all of the method requirements and with the water management system unique to the Evolution, there were no problems with water even though the dry purge was not utilized. Furthermore, the bake time for the analytical trap was set to three minutes and displayed no issues with carryover due to the patented bake of the sparge vessel during the trap bake cycle. The parameters outlined in this application proved that sample cycle times can be reduced without sacrificing quality thus enabling environmental laboratories more leeway to run more samples during a twelve hour period and increasing lab profitability.

REFERENCES:

1. 1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); United States Environmental Protection Agency Method 8260D, Revision 4, June 2018.

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