Evaluating USEPA Method 524.3 Utilizing Newly Permissible Method Modifications to Purge and Trap Techniques

Abstract

Purge and Trap (P&T) concentration in conjunction with Gas Chromatography/Mass Spectrometry (GC/MS) is a widely used technique for the determination of Volatile Organic Compounds (VOCs). Due to the sensitivity requirements required with drinking waters, P&T is the recommended technique for VOC extraction. Recently, the United States Environmental Protection Agency (USEPA) approved an updated version to their drinking water method, USEPA Method 524.3. This new revision allows more flexibility in method operating parameters that take advantage of current P&T capabilities and have the possibility of increasing lab productivity. This application note investigates purge extraction volume variations and their respective affects on compound response.

Introduction

In June of 2009, the USEPA released an updated version of their drinking water method. The latest version includes a revised analyte list, a change in the preservation scheme, a required 5mL sample volume and most significantly more flexibility when choosing experimental purge and trap options1.

In this study, data was collected to evaluate and optimize purge and trap parameters. An Initial Demonstration of Capability (IDC) was performed in order to show the system's effectiveness utilizing the recommended P&T parameters. The final purge volume chosen was 350mL as this purge volume displayed the best recoveries for all of the required 76 analytes. Finally, an IDC was demonstrated for the new P&T parameters.

Experimental-Instrument Conditions

The Stratum PTC and Aquatek 70 Autosampler were coupled to an Agilent 7890A GC and a 5975 inert XL MS system for analysis. The split/splitless inlet of the GC was equipped with a Restek 1.0ID x 6.3OD x 78.5 inlet split liner. Teledyne Tekmar's proprietary #9 trap was the analytical trap used. In order to hold the temperature of the samples at or below the required 10°C, the Aquatek 70 was outfitted with a refrigerated recirculating bath. The GC was configured with an Agilent J&W DB-VRX 30m x 0.250mm x 1.4µm column. The GC/MS parameters are outlined in Tables 1 and 2 respectively while Table 3 outlines the 440mL purge volume P&T conditions and Table 4 outlines the chosen P&T conditions.

GC Parameters	
GC:	Agilent 7890A
Column:	Agilent J&W DB-VRX 30m x 0.250mm x1.4um
Oven Program:	45°C for 4.5 min; 12°C/min to 100°C for 0 min; 25°C /min to 240°C for 1.32 min, 16.003 min runtime
Inlet:	220°C
Column Flow	0.9mL/min
Gas:	Helium
Split:	30:1
Pressure:	11.196 psi
Inlet:	Split/Splitless

MSD Parameters	
MSD:	5975C Inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Scan Range:	m/z 47-300 from 0 to 2.9 min m/z 35-300 from 2.9-16.0 min
Scans:	2.85 scans/sec from 0 to 2.9 min 2.73 scans/sec from 2.9 to 16.0 min
Threshold:	400
Transfer Line Temp:	240°C

Tables 1 & 2: GC and MSD Parameters

Calibration

A 50ppm working calibration stock standard was prepared in methanol. Calibration standards were prepared in a 50mL volumetric flask and filled to volume with preserved de-ionized water. The calibration range was 0.5 – 40ppb. The standards were transferred to headspace free 40mL vials for analysis.

The calibration data was analyzed using Agilent Chemstation software. The analytes of interest were evaluated and quadratic regression was used to fit the calibration points. The quadratic regression values for both purge volumes studied are listed in Table 5.

Stratum PTC and Aquatek 70 Parameters for 440mL Purge Volume			
Variable	Value	Variable	Value
Pressurize Time	0.25 min	Purge Time	11.00
Fill IS Time	0.04 min	Purge Temp	0°C
Sample Transfer Time	0.50 min	Purge Flow	40mL/min
Rinse Loop Time	0.50 min	Dry Purge Time	0.5 min
Purge Loop Time	0.50 min	Dry Purge Temp	30°C
Bake Rinse	On	Dry Purge Flow	100mL/min
Number of Bake Rinses	2	GC Start	Start of Desorb
Bake Drain Time	0.50 min	Desorb Preheat Temp	245°C
Bake Drain Flow	400mL/min	Desorb Drain	On
Valve Oven Temp	150°C	Desorb Time	1.00 min
Transfer Line Temp	150°C	Desorb Temp	250°C
Sample Mount Temp	90°C	Desorb Flow	300mL/min
Purge ready Temp	45°C	Bake Time	2.00 min
Condenser Ready Temp	40°C	Bake Temp	260°C
Condenser Purge Temp	20°C	Bake Flow	400mL/min
Standby Flow	10mL/min	Condenser Bake Temp	175°C
Pre-Purge Time	0.5 min		
Pre-Purge Flow	40.0mL/min		
Sample Heater	Off		
Sample Preheat Time	1.00 min		
Sample Temp	40°C		

Table 3: Stratum PTC and Aquatek Parameters for 440mL Purge Volume
Stratum PTC Parameters are in Blue

Stratum PTC and Aquatek 70 Parameters for 350mL Purge Volume, 0.5min Desorb and 50°C Sample Temp			
Variable	Value	Variable	Value
Pressurize Time	0.25 min	Purge Time	5.00
Fill IS Time	0.04 min	Purge Temp	0°C
Sample Transfer Time	0.35 min	Purge Flow	70mL/min
Rinse Loop Time	0.25 min	Dry Purge Time	0.5 min
Purge Loop Time	0.25 min	Dry Purge Temp	30°C
Bake Rinse	On	Dry Purge Flow	100mL/min
Number of Bake Rinses	2	GC Start	Start of Desorb
Bake Drain Time	0.25 min	Desorb Preheat Temp	245°C
Bake Drain Flow	400mL/min	Desorb Drain	On
Valve Oven Temp	150°C	Desorb Time	0.50 min
Transfer Line Temp	150°C	Desorb Temp	250°C
Sample Mount Temp	90°C	Desorb Flow	300mL/min
Purge ready Temp	45°C	Bake Time	2.00 min
Condenser Ready Temp	40°C	Bake Temp	260°C
Condenser Purge Temp	20°C	Bake Flow	400mL/min
Standby Flow	10mL/min	Condenser Bake Temp	175°C
Pre-Purge Time	0.1 min		
Pre-Purge Flow	40.0mL/min		
Sample Heater	On		
Sample Preheat Time	0.00 min		
Sample Temp	50°C		

Table 4: Stratum PTC and Aquatek Parameters for 350mL Purge Volume, 0.5min Desorb and 50°C Sample Temp
Stratum PTC Parameters are in Blue

Minimum Reporting Level (MRL) and Precision and Accuracy Determination

EPA method 524.3 requires the establishment of MRLs for the 76 analytes in order to prove the IDC. This was done by running 7 replicate standards of the low calibration point and calculating the half range for the prediction interval of results (HRPIR) using the equation given in the method1. The established upper PIR and lower PIR must be between 50% and 150% of the fortified concentration. After the IDC was established with the required

440mL purge volume, several different purge volumes were tested and compared in order to evaluate purge volumes that would best extract the required analytes. The MRL results for the IDC are listed in Table 6 for the 440mL purge volume and for the 350mL purge volume.

Precision and Accuracy was established by the analysis of 7 replicate standards of a 10ppb calibration point. The percent recovery for the 76 analytes was within +20% of the fortified concentration for accuracy. For precision, the percent Relative Standard Deviation (%RSD) of the analyte recovery was +10% for all of the analytes. The precision and accuracy results for both purge volumes are listed in Table 5.

Curve and Precision and Accuracy Summary						
Compound	Curve Quadratic Regression Results (440mL Purge Volume)	Precision % Recovery (440mL Purge Volume)	Accuracy %RSD (440mL Purge Volume)	Curve Quadratic Regression Results (350mL Purge Volume)	Precision % Recovery (350mL Purge Volume)	Accuracy %RSD (350mL Purge Volume)
1,4-Difluorobenzene (IS)	N/A	N/A	N/A	N/A	N/A	N/A
Dichlorodifluoromethane	0.9999	100.40	8.31	0.9999	87.43	8.02
Chlorodifluoromethane	0.9999	104.63	6.40	0.9999	87.36	8.21
Chloromethane	1.0000	102.13	4.34	0.9996	96.13	6.60
vinyl chloride	0.9979	86.10	4.69	0.9998	102.16	7.83
1,3-Butadiene	0.9999	105.13	6.85	0.9998	86.17	8.34
Bromomethane	0.9998	103.50	4.13	1.0000	83.01	6.34
Trichlorofluoromethane	1.0000	99.60	6.17	0.9999	98.80	8.19
Diethyl Ether	0.9999	101.06	3.08	0.9999	101.09	2.86
1,1-Dichloroethene	1.0000	98.70	6.69	0.9999	98.57	6.81
Carbon Disulfide	1.0000	98.27	7.02	0.9997	93.19	6.48
Methyl Iodide	0.9997	96.20	6.04	0.9993	107.39	6.74
Allyl Chloride	1.0000	99.96	4.72	0.9996	92.33	3.98
Methylene Chloride	1.0000	103.63	2.39	1.0000	100.67	2.57
trans-1,2-Dichloroethene	0.9999	101.73	4.10	0.9998	95.39	4.75
Methyl Acetate	0.9999	105.97	2.43	0.9999	96.60	3.59
MTBE-d3 (Surr)	2.40	N/A	N/A	2.00	N/A	N/A
MTBE	1.0000	100.56	1.77	0.9999	99.51	1.53
TBA	0.9999	106.62	4.08	0.9998	89.73	7.45
Diisopropylether	1.0000	100.86	2.34	0.9997	96.66	2.79
1,1-Dichloroethane	1.0000	102.50	4.10	0.9997	97.97	4.14
ETBE	1.0000	102.73	2.32	0.9999	101.41	1.83
cis-1,2-Dichloroethene	1.0000	100.36	6.93	0.9996	96.69	4.22
Bromochloromethane	0.9999	96.23	1.69	0.9997	103.93	4.72
Tetrahydrofuran	0.9999	109.90	3.95	0.9998	94.20	5.49
Chloroform	1.0000	100.84	3.36	0.9996	98.39	3.85
1,1,1-Trichloroethane	1.0000	100.60	5.00	0.9998	98.37	6.68
Carbon Tetrachloride	1.0000	97.84	6.49	0.9997	95.67	7.12
1-Chlorobutane	1.0000	102.39	5.88	0.9999	94.26	6.34
1,1-Dichloropropene	0.9999	100.16	6.33	0.9996	94.73	7.56
Benzene	0.9999	100.76	3.70	0.9998	97.91	4.54
TAME	1.0000	102.29	1.26	0.9998	100.99	1.82
1,2-Dichloroethane	0.9998	102.00	4.70	0.9996	98.00	4.78
Trichloroethene	1.0000	99.81	4.73	0.9998	105.21	5.09
t-Amyl Ethyl Ether	1.0000	102.17	2.41	0.9998	98.99	2.61
1,2-Dichloropropane	1.0000	101.96	2.13	0.9997	95.46	3.25
Dibromomethane	0.9999	102.13	2.55	0.9997	103.79	1.76
Bromodichloromethane	1.0000	100.30	1.76	0.9996	98.04	3.19
cis-1,3-Dichloropropene	1.0000	99.14	2.47	0.9994	98.79	3.48
Chlorobenzene-d5 (IS)	N/A	N/A	N/A	N/A	N/A	N/A
Toluene	1.0000	106.14	5.96	0.9998	98.99	3.50
trans-1,3-Dichloropropene	1.0000	104.09	4.50	0.9999	97.66	2.24
1,1,2-Trichloroethane	0.9999	109.34	2.42	0.9998	100.86	1.98
Ethyl Methacrylate	0.9999	107.36	2.80	0.9999	99.09	4.34
Tetrachloroethene	0.9999	102.81	6.04	0.9998	116.54	3.64
1,3-Dichloropropane	0.9998	108.00	3.12	0.9999	99.96	2.62
Dibromochloromethane	0.9999	103.11	3.51	0.9999	105.19	2.82
1,2-Dibromoethane	0.9997	105.90	3.31	0.9999	104.79	2.90
Chlorobenzene	0.9999	103.21	4.77	0.9999	102.73	3.95
ethylbenzene	0.9999	104.91	6.31	0.9999	98.43	4.70
1,1,1,2-Tetrachloroethane	0.9999	100.70	5.02	0.9997	102.94	2.33
M & P Xylene	0.9998	105.46	4.74	0.9998	100.64	3.61
O Xylene	1.0000	105.61	4.68	0.9998	101.47	2.83
Styrene	0.9999	104.70	5.35	0.9999	101.90	1.03
Bromoform	0.9997	99.51	4.04	0.9997	105.50	2.91
isopropylbenzene	0.9999	103.47	6.51	0.9999	100.87	4.29
1,4-Dichlorobenzene-d4	N/A	N/A	N/A	N/A	N/A	N/A
4-BFB(surr)	2.40	N/A	N/A	3.90	N/A	N/A
Bromobenzene	0.9999	100.60	2.64	0.9997	102.90	3.99
1,1,2,2-Tetrachloroethane	0.9997	106.61	2.06	0.9995	92.17	2.64
n-Propylbenzene	0.9999	103.51	6.50	0.9997	94.11	5.17
1,2,3-Trichloropropane	0.9999	107.66	1.53	0.9995	93.87	3.94
2-Chlorotoluene	0.9999	103.07	4.23	0.9998	96.47	4.07
1,3,5-Trimethylbenzene	0.9999	101.36	5.06	0.9997	97.07	4.93
Pentachloroethane	0.9999	95.40	3.89	0.9993	85.27	6.93
4-Chlorotoluene	0.9999	101.30	3.58	0.9996	97.67	4.05

**%RSD*

Table 5: Curve and Precision and Accuracy Summary

Curve and Precision and Accuracy Summary						
Compound	Curve Quadratic Regression Results (440mL Purge Volume)	Precision % Recovery (440mL Purge Volume)	Accuracy %RSD (440mL Purge Volume)	Curve Quadratic Regression Results (350mL Purge Volume)	Precision % Recovery (350mL Purge Volume)	Accuracy %RSD (350mL Purge Volume)
tert Butyl Benzene	1.0000	101.37	6.71	0.9998	98.54	5.20
1,2,4-Trimethylbenzene	0.9999	102.93	4.49	0.9995	96.81	4.54
sec-Butylbenzene	0.9999	96.83	6.51	0.9997	100.89	4.72
4-Isopropyltoluene	1.0000	98.91	6.34	0.9994	98.01	5.88
1,2-Dichlorobenzene	0.9999	100.01	2.96	0.9994	103.53	2.92
1,3-Dichlorobenzene	0.9999	98.74	3.40	0.9997	103.97	3.58
1,4-Dichlorobenzene	0.9999	97.56	3.45	0.9995	103.80	3.99
n-Butylbenzene	1.0000	98.13	6.16	0.9995	93.70	6.55
1,2-Dichlorobenzene-d4(surr)	7.46	N/A	N/A	8.70	N/A	N/A
Hexachloroethane	1.0000	98.76	4.47	0.9995	96.14	4.55
1,2-Dibromo-3-Chloropropane	0.9997	98.71	3.61	0.9990	99.56	5.45
1,2,4-Trichlorobenzene	1.0000	96.03	3.74	0.9995	106.99	3.80
Hexachlorobutadiene	1.0000	92.57	6.35	0.9993	103.70	6.15
Naphthalene	0.9999	101.91	1.66	0.9996	104.69	4.47
1,2,3-Trichlorobenzene	0.9999	97.97	1.61	0.9995	106.66	3.22

*%RSD

Table 5: Curve and Precision and Accuracy Summary (Continued)

		MRLs for 350mL Purge Volume							MRLs for 440mL Purge Volume						
Compound	Spike Conc. (ppb)	Std. Dev. (ppb)	Mean Compound Response (ppb)	Half Range of Prediction Interval (ppb)	Low PIR (% Recovery)	Pass/ Fail	Upper PIR (% Recovery)	Pass/ Fail	Std. Dev. (ppb)	Mean Compound Response (ppb)	Half Range of Prediction Interval (ppb)	Low PIR (% Recovery)	Pass/ Fail	Upper PIR (% Recovery)	Pass/ Fail
1,4-Difluorobenzene (IS)	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Dichlorodifluoromethane	0.50	0.04	0.44	0.16	56.30	pass	119.70	pass	0.03	0.48	0.12	71.63	pass	121.52	pass
Chlorodifluoromethane	0.50	0.02	0.49	0.08	82.15	pass	113.85	pass	0.04	0.50	0.18	63.77	pass	135.09	pass
Chloromethane	0.50	0.04	0.53	0.16	74.30	pass	137.70	pass	0.03	0.48	0.10	76.99	pass	116.73	pass
vinyl chloride	0.50	0.04	0.51	0.16	70.30	pass	133.70	pass	0.03	0.58	0.11	94.10	pass	136.75	pass
1,3-Butadiene	0.50	0.02	0.55	0.08	94.15	pass	125.85	pass	0.03	0.62	0.12	99.43	pass	148.57	pass
Bromomethane	0.50	0.04	0.41	0.16	50.30	pass	113.70	pass	0.03	0.62	0.11	101.67	pass	146.90	pass
Trichlorofluoromethane	0.50	0.03	0.48	0.12	72.22	pass	119.78	pass	0.03	0.50	0.13	72.86	pass	126.56	pass
Diethyl Ether	0.50	0.02	0.52	0.08	88.15	pass	119.85	pass	0.05	0.53	0.22	63.01	pass	149.56	pass
1,1-Dichloroethene	0.50	0.04	0.51	0.16	70.30	pass	133.70	pass	0.03	0.53	0.14	78.09	pass	133.33	pass
Carbon Disulfide	0.50	0.02	0.60	0.08	104.15	pass	135.85	pass	0.02	0.51	0.08	86.91	pass	118.23	pass
Methyl Iodide	0.50	0.01	0.64	0.04	120.07	pass	135.93	pass	0.01	0.50	0.05	89.49	pass	109.36	pass
Allyl Chloride	0.50	0.02	0.54	0.08	92.15	pass	123.85	pass	0.03	0.50	0.11	78.53	pass	120.90	pass
Methylene Chloride	0.50	0.01	0.54	0.04	100.07	pass	115.93	pass	0.03	0.49	0.11	76.51	pass	121.21	pass
trans-1,2-Dichloroethene	0.50	0.03	0.55	0.12	86.22	pass	133.78	pass	0.03	0.50	0.13	74.52	pass	125.48	pass
Methyl Acetate	0.50	0.03	0.56	0.12	88.22	pass	135.78	pass	0.03	0.58	0.10	96.05	pass	135.95	pass
MTBE-d3 (Surr)	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MTBE	0.50	0.01	0.51	0.04	94.07	pass	109.93	pass	0.04	0.55	0.16	78.15	pass	140.85	pass
TBA	2.50	0.07	2.63	0.28	94.10	pass	116.30	pass	0.21	2.83	0.83	79.91	pass	146.49	pass
Diisopropylether	0.50	0.01	0.55	0.04	102.07	pass	117.93	pass	0.03	0.52	0.11	81.40	pass	124.60	pass
1,1-Dichloroethane	0.50	0.03	0.52	0.12	80.22	pass	127.78	pass	0.04	0.52	0.16	72.58	pass	135.42	pass
ETBE	0.50	0.02	0.47	0.08	78.15	pass	109.85	pass	0.02	0.54	0.09	88.67	pass	126.33	pass
cis-1,2-Dichloroethene	0.50	0.03	0.60	0.12	96.22	pass	143.78	pass	0.03	0.48	0.14	68.48	pass	123.02	pass
Bromochloromethane	0.50	0.03	0.56	0.12	88.22	pass	135.78	pass	0.03	0.57	0.12	90.22	pass	137.78	pass
Tetrahydrofuran	0.50	0.03	0.48	0.12	72.22	pass	119.78	pass	0.09	0.94	0.36	58.33	pass	129.67	pass
Chloroform	0.50	0.02	0.54	0.08	92.15	pass	123.85	pass	0.03	0.53	0.13	79.14	pass	132.36	pass
1,1,1-Trichloroethane	0.50	0.03	0.51	0.12	78.22	pass	125.78	pass	0.05	0.52	0.21	61.49	pass	144.51	pass
Carbon Tetrachloride	0.50	0.02	0.54	0.08	92.15	pass	123.85	pass	0.03	0.49	0.10	78.13	pass	117.87	pass
1-Chlorobutane	0.50	0.04	0.50	0.16	68.30	pass	131.70	pass	0.02	0.55	0.09	91.65	pass	127.35	pass
1,1-Dichloropropene	0.50	0.03	0.56	0.12	88.22	pass	135.78	pass	0.04	0.54	0.16	76.87	pass	140.13	pass
Benzene	0.50	0.02	0.53	0.08	90.15	pass	121.85	pass	0.03	0.52	0.12	79.25	pass	128.25	pass
TAME	0.50	0.02	0.50	0.08	84.15	pass	115.85	pass	0.02	0.50	0.07	85.61	pass	113.39	pass
1,2-Dichloroethane	0.50	0.02	0.58	0.08	100.15	pass	131.85	pass	0.04	0.59	0.16	86.30	pass	149.70	pass
Trichloroethene	0.50	0.02	0.57	0.08	98.15	pass	129.85	pass	0.03	0.56	0.13	87.03	pass	137.47	pass
t-Amyl Ethyl Ether	0.50	0.02	0.50	0.08	84.15	pass	115.85	pass	0.05	0.53	0.22	62.35	pass	148.65	pass
1,2-Dichloropropane	0.50	0.02	0.60	0.08	104.15	pass	135.85	pass	0.04	0.55	0.14	82.10	pass	139.04	pass
Dibromomethane	0.50	0.04	0.42	0.16	52.30	pass	115.70	pass	0.04	0.58	0.17	82.64	pass	149.36	pass
Bromodichloromethane	0.50	0.01	0.57	0.04	106.07	pass	121.93	pass	0.04	0.54	0.17	73.48	pass	142.02	pass
cis-1,3-Dichloropropene	0.50	0.02	0.62	0.08	108.15	pass	139.85	pass	0.04	0.53	0.14	77.16	pass	133.84	pass
Chlorobenzene-d5 (IS)	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Toluene	0.50	0.03	0.45	0.12	66.22	pass	113.78	pass	0.04	0.44	0.14	59.48	pass	115.52	pass
trans-1,3-Dichloropropene	0.50	0.03	0.47	0.12	70.22	pass	117.78	pass	0.03	0.47	0.10	72.81	pass	114.69	pass
1,1,2-Trichloroethane	0.50	0.04	0.47	0.16	62.30	pass	125.70	pass	0.06	0.49	0.24	51.08	pass	145.49	pass
Ethyl Methacrylate	0.50	0.03	0.50	0.12	76.22	pass	123.78	pass	0.06	0.49	0.23	51.63	pass	142.87	pass
Tetrachloroethene	0.50	0.05	0.48	0.20	56.37	pass	135.63	pass	0.03	0.52	0.13	78.63	pass	129.94	pass
1,3-Dichloropropane	0.50	0.04	0.48	0.16	64.30	pass	127.70	pass	0.03	0.54	0.13	81.64	pass	134.86	pass
Dibromochloromethane	0.50	0.03	0.51	0.12	78.22	pass	125.78	pass	0.03	0.49	0.11	76.18	pass	120.82	pass
1,2-Dibromoethane	0.50	0.03	0.48	0.12	72.22	pass	119.78	pass	0.05	0.49	0.18	62.36	pass	134.64	pass
Chlorobenzene	0.50	0.03	0.49	0.12	74.22	pass	121.78	pass	0.02	0.42	0.07	71.23	pass	97.27	pass
ethylbenzene	0.50	0.03	0.47	0.12	70.22	pass	117.78	pass	0.03	0.46	0.12	67.30	pass	116.70	pass
1,1,1,2-Tetrachloroethane	0.50	0.03	0.53	0.12	82.22	pass	129.78	pass	0.04	0.46	0.16	60.15	pass	122.85	pass
M & P Xylene	1.00	0.04	0.93	0.16	77.15	pass	108.85	pass	0.07	0.99	0.27	72.21	pass	125.79	pass
O Xylene	0.50	0.02	0.47	0.08	78.15	pass	109.85	pass	0.02	0.46	0.08	76.37	pass	106.63	pass

Table 6: MRL Results for the 350mL and 440mL Purge Volume

Compound	Spike Conc. (ppb)	MRLs for 350mL Purge Volume							MRLs for 440mL Purge Volume						
		Std. Dev. (ppb)	Mean Compound Response (ppb)	Half Range of Prediction Interval (ppb)	Low PIR (% Recovery)	Pass/ Fail	Upper PIR (% Recovery)	Pass/ Fail	Std. Dev. (ppb)	Mean Compound Response (ppb)	Half Range of Prediction Interval (ppb)	Low PIR (% Recovery)	Pass/ Fail	Upper PIR (% Recovery)	Pass/ Fail
Styrene	0.50	0.04	0.53	0.16	74.30	pass	137.70	pass	0.03	0.49	0.13	72.13	pass	124.87	pass
Bromoform	0.50	0.03	0.58	0.12	92.22	pass	139.78	pass	0.05	0.53	0.20	66.27	pass	144.73	pass
isopropylbenzene	0.50	0.03	0.49	0.12	74.22	pass	121.78	pass	0.02	0.46	0.09	73.56	pass	109.94	pass
1,4-Dichlorobenzene-d4	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
4-BFB(surr)	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Bromobenzene	0.50	0.04	0.56	0.16	80.30	pass	143.70	pass	0.03	0.39	0.13	53.03	pass	103.47	pass
1,1,2,2-Tetrachloroethane	0.50	0.04	0.43	0.16	54.30	pass	117.70	pass	0.03	0.50	0.11	77.59	pass	121.41	pass
n-Propylbenzene	0.50	0.02	0.47	0.08	78.15	pass	109.85	pass	0.02	0.39	0.09	60.31	pass	97.19	pass
1,2,3-Trichloropropane	0.50	0.03	0.44	0.12	64.22	pass	111.78	pass	0.04	0.56	0.15	81.07	pass	142.43	pass
2-Chlorotoluene	0.50	0.02	0.42	0.08	68.15	pass	99.85	pass	0.04	0.45	0.14	61.16	pass	117.84	pass
1,3,5-Trimethylbenzene	0.50	0.02	0.47	0.08	78.15	pass	109.85	pass	0.03	0.42	0.11	62.47	pass	106.03	pass
Pentachloroethane	0.50	0.04	0.56	0.16	80.30	pass	143.70	pass	0.03	0.38	0.12	52.22	pass	99.78	pass
4-Chlorotoluene	0.50	0.03	0.44	0.12	64.22	pass	111.78	pass	0.03	0.41	0.13	55.58	pass	106.42	pass
tert Butyl Benzene	0.50	0.04	0.44	0.16	56.30	pass	119.70	pass	0.04	0.43	0.15	55.65	pass	114.35	pass
1,2,4-Trimethylbenzene	0.50	0.04	0.50	0.16	68.30	pass	131.70	pass	0.03	0.46	0.13	64.48	pass	118.02	pass
sec-Butylbenzene	0.50	0.03	0.41	0.12	58.22	pass	105.78	pass	0.03	0.42	0.12	59.89	pass	109.11	pass
4-Isopropyltoluene	0.50	0.03	0.51	0.12	78.22	pass	125.78	pass	0.04	0.43	0.14	58.48	pass	114.52	pass
1,2-Dichlorobenzene	0.50	0.05	0.51	0.20	62.37	pass	141.63	pass	0.03	0.39	0.12	54.03	pass	101.97	pass
1,3-Dichlorobenzene	0.50	0.02	0.41	0.08	66.15	pass	97.85	pass	0.03	0.45	0.13	63.82	pass	115.18	pass
1,4-Dichlorobenzene	0.50	0.04	0.43	0.16	54.30	pass	117.70	pass	0.03	0.42	0.12	60.19	pass	107.24	pass
n-Butylbenzene	0.50	0.04	0.48	0.16	64.30	pass	127.70	pass	0.03	0.50	0.14	71.80	pass	127.20	pass
1,2-Dichlorobenzene-d4(surr)	5.00	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Hexachloroethane	0.50	0.06	0.50	0.24	52.44	pass	147.56	pass	0.04	0.44	0.15	59.11	pass	118.39	pass
1,2-Dibromo-3-Chloropropane	0.50	0.01	0.63	0.04	118.07	pass	133.93	pass	0.06	0.50	0.24	52.30	pass	147.20	pass
1,2,4-Trichlorobenzene	0.50	0.05	0.48	0.20	56.37	pass	135.63	pass	0.02	0.44	0.10	68.20	pass	106.80	pass
Hexachlorobutadiene	0.50	0.04	0.56	0.16	80.30	pass	143.70	pass	0.04	0.41	0.15	53.61	pass	112.11	pass
Naphthalene	0.50	0.05	0.49	0.20	58.37	pass	137.63	pass	0.03	0.43	0.11	65.59	pass	107.91	pass
1,2,3-Trichlorobenzene	0.50	0.05	0.54	0.20	68.37	pass	147.63	pass	0.03	0.46	0.13	66.47	pass	118.53	pass

Table 6: MRL Results for the 350mL and 440mL Purge Volume (Continued)

Drinking Water Study Using Preserved Tap Water		
Compound	Average Compound Recovery Batch 1	Average Compound Recovery Batch 2
Chloroform	4.71	4.19
Bromodichloromethane	10.48	9.78
Dibromochloromethane	18.07	17.17
Bromoform	9.80	9.67
Compound	Average %Surrogate Recovery Batch 1	Average %Surrogate Recovery Batch 2
MTBE-d3 (Surr)	99.00	99.00
4-BFB(surr)	100.00	99.10
1,2-Dichlorobenzene-d4(surr)	100.00	100.00

Table 9: Tap Water Summary

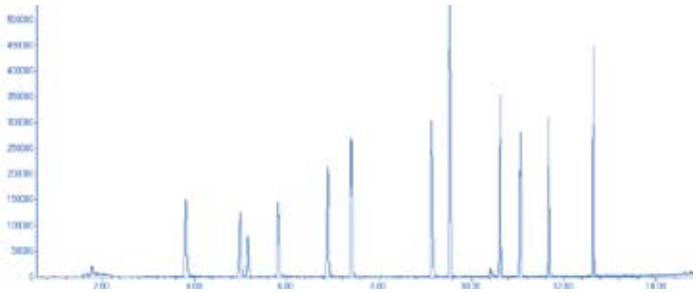

Figure 2: Chromatogram of Tap Water

Tap Water Study

Two batches of 20 tap water samples were preserved and tested with the newly established P&T method modifications. The samples were analyzed and quantified and a summary of the results are listed in Table 9. A chromatogram of a preserved tap water sample is displayed in Figure 2.

Conclusions

USEPA Method 524.3 has many noteworthy changes for the analysis of drinking waters and the Stratum PTC and Aquatek 70 system proved to be an excellent purge and trap system for this analysis. The option to modify purge and trap parameters and shorten experimental time is very important when trying to optimize sample throughput in a drinking water lab. In this study, a five minute purge time at 70mL/min along with a 30 second desorb time and a 50°C sample temperature proved to produce consistent results and meet all of the quality control specifications set by the new method. These new parameters can shorten experimental time by more than six minutes, thus improving laboratory productivity.

References

1. USEPA Method 524.3, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Version 1.0, June 2009.