Chromatography
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Demonstration of Drinking Water Volatiles Analysis by Purge and Trap Utilizing Hydrogen as the Carrier Gas with Agilent's HydroInert® Source

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Abstract

As helium supplies become scarcer and more expensive, laboratories have been seeking alternative carrier gases. Hydrogen is a low-cost, renewable gas suitable for several gas chromatograph (GC)/mass spectrometer (MS) (GC/MS) applications. The United States Environmental Protection Agency (US EPA) has not evaluated hydrogen as a carrier gas for drinking water analysis yet, therefore, this is a demonstration of hydrogen carrier gas for the quantitation of volatile organic compounds (VOCs) with the method requirements of US EPA Method 524.4 in mind. The Tekmar Lumin Purge and Trap (P&T) concentrator with the AQUATek LVA autosampler was paired with an Agilent 7890B GC and 5977B MS and the HydroInert ion source to demonstrate the method requirements: a working linear (r^2) calibration curve, method detection limits (MDL), a mid-point calibration check with accuracy and precision, and Minimum Reporting Level (MRL) confirmation for target compounds. The calibration curve was also validated with the lowest level standard within ±50% of the true value and all other calibration standards within ±30% of the true value.

Introduction

The Agilent HydroInert source is a GC/MS ion source that improves chromatographic performance when using hydrogen as carrier gas. Its inertness minimizes hydrogenation and dechlorination reactions in the MS source, avoiding loss of sensitivity and spectral anomalies while offering a high-boiler peak shape. The Tekmar Lumin P&T is compatible with hydrogen carrier gas and allows the use of nitrogen as purge gas, avoiding the need for a helium supply. The AQUATek LVA autosampler utilizes a fixed volume loop that transfers the liquid sample, internal standards, and surrogate standards to the Lumin P&T concentrator. Then it initiates a clean-up cycle where the sample loop and sparger are cleaned with 90 °C water, achieving method required carryover compliance.

Sample Preparation

A 50 parts per million (ppm) calibration working standard was prepared in methanol from the following Restek® standards: 524.3 VOA Mega Mix and 524.3 Gas Calibration Mix. In total, the standards contained 75 compounds.

A seven-point linear (r^2) calibration curve was prepared from 0.2 ppb to 50 parts per billion (ppb) for all compounds with regression value (r^2) ≥0.995. The relative response factor (RRF) was calculated for each compound using three internal standards: 1,4-difluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4. Surrogate standards consisted of: methyl-tert-butyl ether-d3, 4-bromofluorobenzene, and 1,2-dichlorobenzene-d4. Internal and surrogate standards were prepared in methanol from Restek standards at a concentration of 10 ppm, after which 5 µL was then mixed with each 5 mL sample for a resulting concentration of 10 ppb.

Seven 0.5 ppb standards were prepared to calculate the MDL and MRL confirmation calculations. Also, seven 10 ppb standards were prepared for the accuracy and precision calculations of the mid-point calibration check. All calibration, MDL, MRL, and mid-point calibration check standards were analyzed with the Tekmar Lumin P&T and AQUATek LVA conditions in Table I. GC-MS conditions are shown in Table II.

Experimental Instrument Conditions

Table I Tekmar Lumin P&T and AQUATek LVA Water Method Conditions

Standby	Variable	Desorb	Variable
Valve Oven Temp	150 °C	Desorb Preheat Temp	245 °C
Transfer Line Temp	150 °C	Desorb Temp	250 °C
Sample Mount Temp	90 °C	Desorb Time	1.00 min
Standby Flow	10 mL/min	Drain Flow	300 mL/min
Purge Ready Temp	35 °C	GC Start Signal	Begin Desorb
MCS Purge Temp	20 °C	**Bake**	**Variable**
Purge	**Variable**	Bake Time	2.00 min
Purge Temp	20 °C	Bake Temp	270 °C
Purge Time	9.00 min	MCS Bake Temp	180 °C
Purge Flow	50 mL/min	Bake Flow	200 mL/min
Dry Purge Temp	20 °C	**AQUATek LVA**	**Variable**
Dry Purge Time	1.00 min	Sample Loop Time	0.35 min
Dry Purge Flow	100 mL/min	Sample Transfer Time	0.35 min
Sparge Vessel Heater	Off	Rinse Loop Time	0.35 min
		Sweep Needle Time	0.30 min
		Presweep Time	0.25 min
Trap	9	Water Temp	90 °C
Chiller Tray	On	Bake Rinse Cycles	1
Purge Gas	Nitrogen	Bake Rinse Drain Time	0.30 min

Table II Agilent 7890B GC/5977B MS System Conditions

Agilent 7890B GC Conditions	
Column	DB-624 Ultra Inert, 30 m x 0.25 mm, 1.4µm Film, Column Flow –0.7 mL/min
Oven Profile	35 °C, 2 min, 15 °C/min to 100 °C, 30 °C/min to 230 °C, 1 min Hold, Run Time 11.67 min
Inlet	200 °C, 100:1 Split, Septum Purge Flow 0.5 mL/min, 4.715 psi, Carrier Gas - Hydrogen
Agilent 5977B MS Conditions	
Temp	Transfer Line 250 °C; Source 250 °C; Quad 200 °C
Scan	Range 35 *m/z* to 270 *m/z*, Solvent Delay 0.50 min, Normal Scanning
Current	Gain Factor 3.00, Extraction Source Tune

Results

The linear correlation coefficient of the calibration curve (r^2), MDL, and MRL confirmation data are shown in Table III. The mid-point calibration check with precision and accuracy data are down in Table IV. Table V displays the validation of the initial calibration curve. Figure 1 displays an overlay of the calibration standards, with an inset demonstrating excellent peak shape and separation across all concentrations with minimal water interference for all target compounds.

Table III **US EPA Method 524.4 Calibration, MDL, and MRL Confirmation Data (cont**

Compound	Calibration (0.2-50 ppb)					Method Detection Limits (n=7, 0.5 ppb)		Minimum Reporting Level (n=7, 0.5 ppb)	
	Ret. Time	Confirm. Ion	RRF (%rsd)	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	LPIR (≥50%)	UPIR (≤150%)
Dichlorodifluoromethane	0.94	85	4.72	1.00	0.540	0.07	4.68	77	112
Chlorodifluoromethane	0.96	51	12.8	0.996	0.675	0.09	6.43	69	116
Chloromethane	1.07	50	12.1	1.00	0.411	0.14	8.41	69	138
Vinyl Chloride	1.13	62	2.88	1.00	0.426	0.09	5.84	75	120
1,3-Butadiene	1.16	54	6.60	1.00	0.428	0.13	8.33	66	130
Bromomethane	1.35	94	15.7	0.995	0.401	0.09	5.53	78	121
Trichlorofluoromethane	1.62	101	5.06	1.00	0.512	0.11	7.58	67	124
Diethyl Ether	1.86	59	8.02	1.00	0.312	0.08	5.10	80	120
1,1-Dichloroethene	2.03	96	11.6	1.00	0.425	0.12	7.58	71	131
Iodomethane	2.13	142	12.7	0.998	0.563	0.08	5.82	69	110
Carbon Disulfide	2.18	76	2.16	1.00	1.07	0.11	6.89	75	127
Allyl Chloride	2.32	41	10.1	1.00	0.720	0.13	8.22	69	135
Methyl Acetate	2.36	43	9.02	1.00	0.387	0.06	3.75	87	117
Methylene Chloride[1]	2.41	84	1.00	1.00	0.546	0.07	4.00	89	122
tert-Butyl Alcohol	2.57	59	6.14	0.999	0.047	0.06	4.14	82	114
trans-1,2-Dichloroethene	2.65	96	8.45	1.00	0.461	0.12	7.63	73	136
Methyl-tert-Butyl Ether-d3 (SURR)	2.66	76	2.58		1.22		1.50	96	109
Methyl-tert-Butyl Ether	2.68	73	12.2	1.00	1.68	0.04	2.53	92	112
1,1-Dichloroethane	2.99	63	8.75	1.00	0.937	0.09	5.84	78	125
Diisopropyl Ether	3.10	45	12.4	1.00	1.58	0.06	4.01	82	114
tert-Butyl Ethyl Ether	3.40	59	9.25	1.00	1.79	0.05	3.36	86	113
cis-1,2-Dichloroethene	3.47	96	11.7	1.00	0.554	0.10	5.98	79	128
Bromochloromethane	3.66	128	9.31	1.00	0.257	0.12	7.69	71	134
Tetrahydrofuran	3.73	72	8.56	0.999	0.069	0.11	6.93	75	133
Chloroform	3.74	83	9.75	1.00	0.955	0.09	5.82	76	121
1,1,1-Trichloroehane	3.89	97	6.83	1.00	0.803	0.12	7.47	69	128

Table III **US EPA Method 524.4 Calibration, MDL, and MRL Confirmation Data (continued)**

Compound	Calibration (0.2-50 ppb)					Method Detection Limits (n=7, 0.5 ppb)		Minimum Reporting Level (n=7, 0.5 ppb)	
	Ret. Time	Confirm. Ion	RRF (%rsd)	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	LPIR (≥50%)	UPIR (≤150%)
1-Chlorobutane	3.98	56	10.2	1.00	1.03	0.13	7.77	71	135
1,1-Dichloropropene	4.03	75	2.91	1.00	0.700	0.09	5.84	78	125
Carbon Tetrachloride	4.03	117	7.35	1.00	0.630	0.07	4.65	83	120
Benzene	4.19	78	11.3	1.00	2.17	0.07	4.60	84	121
1,2-Dichloroethane	4.19	62	9.82	1.00	0.811	0.06	3.93	88	120
tert-Amyl Methyl Ether	4.33	73	7.58	1.00	1.62	0.08	4.89	80	119
1,4-Difluorobenzene (IS)	4.53	114							
Trichloroethylene	4.74	132	5.41	1.00	0.589	0.07	4.36	88	124
1,2-Dichloropropane	4.90	63	10.1	1.00	0.565	0.12	7.52	74	136
tert-Amyl Ethyl Ether	5.00	59	10.4	1.00	1.42	0.06	3.66	85	114
Dibromomethane	5.00	93	9.30	1.00	0.336	0.09	5.18	86	131
Bromodichloromethane	5.15	83	11.8	1.00	0.680	0.09	5.58	77	121
cis-1,3-Dichloropropene	5.54	75	9.07	1.00	0.913	0.11	6.76	74	128
Toluene	5.83	92	11.7	1.00	1.29	0.11	6.35	79	133
trans-1,3-Dichloropropene	6.02	75	10.4	1.00	0.796	0.07	4.49	82	117
Ethyl Methacrylate	6.14	69	9.71	1.00	0.700	0.12	8.05	66	128
1,1,2-Trichloroethane	6.17	83	12.5	1.00	0.406	0.10	6.52	72	123
1,3-Dichloropropane	6.31	76	7.59	0.999	0.786	0.09	5.56	77	120
Tetrachloroethylene	6.32	166	11.5	0.999	0.938	0.10	5.88	80	128
Dibromochloromethane	6.50	129	5.80	1.00	0.458	0.10	6.72	71	122
1,2-Dibromoethane	6.59	107	9.85	1.00	0.472	0.09	5.81	77	123
Chlorobenzene-d5 (IS)	6.98	117							
Chlorobenzene	7.00	112	9.75	0.999	1.39	0.05	3.36	86	113
1,1,1,2-Tetrachloroethane	7.06	131	6.67	1.00	0.428	0.12	7.30	72	130
Ethylbenzene	7.06	91	10.7	0.999	2.40	0.09	5.39	84	130
m,p-Xylene	7.18	106	10.3	0.999	0.937	0.19	6.08	76	124
o-Xylene	7.46	106	9.72	1.00	0.952	0.09	5.58	79	123
Styrene	7.47	104	12.1	1.00	1.60	0.10	5.99	83	135
Bromoform	7.58	173	4.68	1.00	0.289	0.07	4.96	77	115
Isopropylbenzene	7.72	105	7.88	0.999	2.32	0.12	7.56	69	128
4-Bromofluorobenzene (SURR)	7.81	95	1.39		0.684		1.11	95	104

Table III **US EPA Method 524.4 Calibration, MDL, and MRL Confirmation Data (continued)**

Compound	Calibration (0.2-50 ppb)					Method Detection Limits (n=7, 0.5 ppb)		Minimum Reporting Level (n=7, 0.5 ppb)	
	Ret. Time	Confirm. Ion	RRF (%rsd)	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	LPIR (≥50%)	UPIR (≤150%)
Bromobenzene	7.90	156	9.64	1.00	0.917	0.10	6.26	76	127
1,1,2,2-Tetrachloroethane	7.90	83	5.53	1.00	0.611	0.14	9.16	63	135
1,2,3-Trichloropropane	7.92	110	9.84	0.999	0.279	0.13	7.60	73	137
n-Propylbenzene	7.99	91	10.1	0.998	4.40	0.09	5.55	80	124
2-Chlorotoluene	8.04	91	9.03	0.999	2.77	0.11	7.04	75	132
1,3,5-Trimethylbenzene	8.11	105	11.9	0.998	3.43	0.09	5.47	79	123
4-Chlorotoluene	8.11	91	9.93	0.998	3.22	0.11	7.16	73	131
tert-Butylbenzene	8.31	119	5.52	0.999	2.80	0.07	4.30	86	122
Pentachloroethane[2]	8.31	167	1.00	1.00	0.089	0.09	6.00	76	123
1,2,4-Trimethylbenzene	8.34	105	10.7	0.998	3.43	0.07	4.47	85	121
sec-Butylbenzene	8.44	105	10.5	0.998	4.01	0.11	7.17	70	126
1,3-Dichlorobenzene	8.50	146	10.5	0.999	1.73	0.08	5.04	79	118
4-Isopropyltoluene	8.53	119	8.34	0.999	3.41	0.08	5.35	79	122
1,4-Dichlorobenzene-d4 (IS)	8.54	152							
1,4-Dichlorobenzene	8.55	146	9.23	0.999	1.69	0.07	4.37	83	118
1,2-Dichlorobenzene-d4 (SURR)	8.76	152	1.34		0.998		0.460	98	101
1,2-Dichlorobenzene	8.77	146	8.41	0.999	1.65	0.07	4.40	93	118
n-Butylbenzene	8.77	91	11.3	0.998	3.28	0.13	8.80	63	131
Hexachloroethane	8.93	201	7.36	0.999	0.334	0.04	2.92	82	103
1,2-Dibromo-3-Chloropropane	9.20	157	13.8	1.00	0.163	0.13	8.22	67	131
1,2,4-Trichlorobenzene	9.68	180	7.44	1.00	0.983	0.09	6.35	69	115
Hexachlorobutadiene	9.78	225	4.55	1.00	0.390	0.07	4.95	77	114
Naphthalene	9.80	128	9.04	0.998	2.80	0.06	3.89	85	116
1,2,3-Trichlorobenzene	9.94	180	9.29	1.00	0.903	0.07	4.59	76	109

1. Compound used a linear regression calibration fit.
2. Compound used a quadratic regression calibration fit.

Table IV **US EPA 524.4 Mid-Point Calibration Check Data**

Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
Dichlorodifluoromethane	10.2	5.12	102
Chlorodifluoromethane	9.79	4.12	98
Chloromethane	9.46	3.79	95
Vinyl Chloride	10.1	4.30	101
1,3-Butadiene	10.4	5.06	104
Bromomethane	9.54	2.89	95
Trichlorofluoromethane	10.7	4.45	107
Diethyl Ether	9.66	3.08	97
1,1-Dichloroethene	9.56	4.06	96
Iodomethane	10.3	4.28	103
Carbon Disulfide	10.1	4.77	101
Allyl Chloride	9.34	2.96	93
Methyl Acetate	8.96	3.18	90
Methylene Chloride	10.2	3.37	102
tert-Butyl Alcohol	9.96	4.69	100
trans-1,2-Dichloroethene	9.64	4.03	96
Methyl-tert-Butyl Ether-d3 (SURR)	10.1	1.20	101
Methyl-tert-Butyl Ether	9.63	2.80	96
1,1-Dichloroethane	9.54	3.32	95
Diisopropyl Ether	9.53	2.95	95
tert-Butyl Ethyl Ether	9.66	2.44	97
cis-1,2-Dichloroethene	9.30	3.47	9.
Bromochloromethane	9.38	3.23	94
Tetrahydrofuran	9.80	4.30	98
Chloroform	9.55	2.97	95
1,1,1-Trichloroehane	10.0	3.90	100
1-Chlorobutane	9.66	4.23	97
1,1-Dichloropropene	10.1	4.17	101
Carbon Tetrachloride	10.1	4.40	101
Benzene	9.45	3.42	95
1,2-Dichloroethane	9.65	2.85	96
tert-Amyl Methyl Ether	9.79	2.85	98
1,4-Difluorobenzene (IS)			

Table IV **US EPA 524.4 Mid-Point Calibration Check Data (cont)**

Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
Trichloroethylene	10.2	3.70	102
1,2-Dichloropropane	9.55	2.79	95
tert-Amyl Ethyl Ether	9.56	2.88	96
Dibromomethane	9.79	2.86	98
Bromodichloromethane	9.47	2.96	95
cis-1,3-Dichloropropene	9.57	2.70	96
Toluene	9.44	2.94	94
trans-1,3-Dichloropropene	9.50	1.96	95
Ethyl Methacrylate	9.66	2.29	97
1,1,2-Trichloroethane	9.26	2.52	93
1,3-Dichloropropane	9.77	2.64	98
Tetrachloroethylene	9.88	3.45	99
Dibromochloromethane	9.64	2.19	96
1,2-Dibromoethane	9.63	2.75	96
Chlorobenzene-d5 (IS)			
Chlorobenzene	9.47	2.79	95
1,1,1,2-Tetrachloroethane	9.70	2.69	97
Ethylbenzene	9.60	3.00	96
m,p-Xylene	19.3	2.85	96
o-Xylene	9.56	3.15	96
Styrene	9.36	2.91	94
Bromoform	10.2	2.34	102
Isopropylbenzene	9.84	3.29	98
4-Bromofluorobenzene (SURR)	10.2	0.580	102
Bromobenzene	9.32	2.55	93
1,1,2,2-Tetrachloroethane	9.56	2.32	96
1,2,3-Trichloropropane	9.43	2.76	94
n-Propylbenzene	9.50	3.55	95
2-Chlorotoluene	9.48	2.86	95
1,3,5-Trimethylbenzene	9.33	3.13	93
4-Chlorotoluene	9.50	2.97	95
tert-Butylbenzene	9.64	3.44	96
Pentachloroethane	8.16	7.52	82
1,2,4-Trimethylbenzene	9.41	3.16	94

Table IV US EPA 524.4 Mid-Point Calibration Check Data (cont)			
Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
sec-Butylbenzene	9.58	3.91	96
1,3-Dichlorobenzene	9.41	2.49	94
4-Isopropyltoluene	9.60	3.69	96
1,4-Dichlorobenzene-d4 (IS)			
1,4-Dichlorobenzene	9.44	2.50	94
1,2-Dichlorobenzene-d4 (SURR)	10.1	0.690	101
1,2-Dichlorobenzene	9.55	2.59	95
n-Butylbenzene	9.37	4.02	94
Hexachloroethane	9.71	3.81	97
1,2-Dibromo-3-Chloropropane	8.92	2.65	89
1,2,4-Trichlorobenzene	9.80	2.68	98
Hexachlorobutadiene	9.95	5.32	100
Naphthalene	9.90	2.07	99
1,2,3-Trichlorobenzene	9.69	2.21	97

Table V US EPA Method 524.4 Validation of the Initial Calibration Curve (0.2-50 ppb)							
Compounds	Cal Std 1 (±50%)	Cal Std 2 (±30%)	Cal Std 3 (±30%)	Cal Std 4 (±30%)	Cal Std 5 (±30%)	Cal Std 6 (±30%)	Cal Std 7 (±30%)
Dichlorodifluoromethane	1.28	0.980	1.92	1.49	0.350	0.210	0.370
Chlorodifluoromethane	4.05	5.56	1.28	0.590	0.330	2.84	6.64
Chloromethane	5.56	1.55	0.500	0.760	2.19	2.36	2.66
Vinyl Chloride	0.00	0.510	1.46	0.150	0.400	0.090	0.130
1,3-Butadiene	3.49	3.19	0.740	0.560	0.030	0.020	0.070
Bromomethane	7.14	1.55	0.250	0.050	0.660	2.25	7.03
Trichlorofluoromethane	1.28	2.63	0.250	0.150	0.670	0.920	1.26
Diethyl Ether	2.38	0.980	1.22	0.050	1.20	2.16	2.18
1,1-Dichloroethene	2.38	2.83	0.250	2.41	2.55	1.73	1.90
Iodomethane	1.22	1.55	2.63	2.52	0.760	2.71	4.91
Carbon Disulfide	0.00	0.510	0.980	0.100	0.050	0.200	0.480
Allyl Chloride	1.28	2.08	1.55	0.970	2.27	2.33	2.62
Methyl Acetate	5.56	3.27	1.69	0.510	1.47	0.980	1.70
Methylene Chloride	14.5	5.56	0.740	0.050	0.250	0.220	0.04
tert-Butyl Alcohol	1.22	1.46	1.92	0.050	0.320	1.65	2.57

Table V US EPA Method 524.4 Validation of the Initial Calibration Curve (0.2-50 ppb) (continued)							
Compounds	Cal Std 1 (±50%)	Cal Std 2 (±30%)	Cal Std 3 (±30%)	Cal Std 4 (±30%)	Cal Std 5 (±30%)	Cal Std 6 (±30%)	Cal Std 7 (±30%)
trans-1,2-Dichloroethene	2.38	0.980	0.250	1.55	1.49	1.01	1.53
Methyl-tert-Butyl Ether	5.56	0.00	1.22	1.07	1.33	2.12	3.09
1,1-Dichloroethane	2.38	1.46	2.83	0.860	1.79	1.93	2.33
Diisopropyl Ether	5.56	2.08	2.15	0.920	1.52	1.76	2.60
tert-Butyl Ethyl Ether	3.49	0.510	1.92	0.710	1.28	1.59	2.50
cis-1,2-Dichloroethene	3.49	0.980	1.46	1.44	2.49	2.45	3.03
Bromochloromethane	2.38	3.70	0.250	1.28	2.93	2.06	2.79
Tetrahydrofuran	0.00	1.46	3.05	0.300	0.450	2.65	2.75
Chloroform	3.49	1.55	0.00	1.33	0.510	0.340	0.350
1,1,1-Trichloroehane	3.49	1.02	0.980	1.02	0.840	0.590	1.06
1-Chlorobutane	4.55	0.500	0.980	1.33	1.65	1.59	2.09
1,1-Dichloropropene	2.63	0.980	0.980	0.660	0.200	0.110	0.760
Carbon Tetrachloride	3.49	1.55	0.00	1.33	0.510	0.340	0.350
Benzene	4.55	1.92	3.49	1.18	2.00	2.58	3.21
1,2-Dichloroethane	2.38	1.46	2.83	0.660	1.36	2.23	3.35
tert-Amyl Methyl Ether	3.49	0.510	1.69	0.350	0.920	1.36	2.32
Trichloroethylene	1.28	2.63	0.250	1.55	2.06	0.890	3.65
1,2-Dichloropropane	3.49	1.02	3.49	1.23	2.22	2.08	2.79
tert-Amyl Ethyl Ether	3.49	0.500	2.15	1.02	1.52	1.96	3.02
Dibromomethane	4.05	1.02	3.27	0.920	1.84	1.32	3.15
Bromodichloromethane	5.56	0.510	1.92	1.33	1.84	1.96	2.13
cis-1,3-Dichloropropene	3.49	1.02	1.46	0.510	1.47	1.60	1.81
Toluene	5.56	0.510	1.92	1.12	1.47	1.88	2.93
trans-1,3-Dichloropropene	4.55	1.02	1.22	1.18	1.10	1.43	2.07
Ethyl Methacrylate	4.55	0.510	1.02	1.23	0.330	1.14	1.75
1,1,2-Trichloroethane	5.56	1.46	1.22	2.03	1.36	2.55	3.19
1,3-Dichloropropane	1.28	0.00	1.92	0.300	0.350	1.55	2.81
Tetrachloroethylene	4.55	0.980	1.69	0.810	0.810	2.23	4.19
Dibromochloromethane	2.38	0.500	0.250	1.12	0.530	1.01	1.29
1,2-Dibromoethane	4.55	0.00	0.250	1.18	0.630	1.36	2.02
Chlorobenzene	3.49	0.980	1.92	0.810	1.26	1.88	3.28
1,1,1,2-Tetrachloroethane	2.38	1.02	1.22	0.760	0.660	1.02	1.23
Ethylbenzene	3.49	0.510	1.69	1.12	1.41	1.73	4.21

Table V **US EPA Method 524.4 Validation of the Initial Calibration Curve (0.2-50 ppb) (continued)**

Compounds	Cal Std 1 (±50%)	Cal Std 2 (±30%)	Cal Std 3 (±30%)	Cal Std 4 (±30%)	Cal Std 5 (±30%)	Cal Std 6 (±30%)	Cal Std 7 (±30%)
m,p-Xylene	4.55	0.510	1.69	0.860	0.880	1.69	2.99
o-Xylene	2.38	1.55	2.61	1.55	1.44	1.53	2.93
Styrene	4.55	0.980	0.980	2.14	2.14	2.11	3.86
Bromoform	1.22	2.08	0.00	0.250	1.03	0.270	0.150
Isopropylbenzene	3.49	0.00	1.22	0.810	0.330	0.860	2.85
Bromobenzene	3.49	0.510	2.15	1.18	0.610	1.91	2.78
1,1,2,2-Tetrachloroethane	1.22	1.02	1.81	1.23	1.22	0.030	1.09
1,2,3-Trichloropropane	2.38	3.27	0.980	1.76	0.070	2.47	3.16
n-Propylbenzene	3.49	0.980	2.15	1.23	0.050	1.76	4.21
2-Chlorotoluene	2.38	0.510	1.46	1.33	0.630	1.67	3.94
1,3,5-Trimethylbenzene	4.55	0.500	1.92	1.39	0.710	1.99	4.21
4-Chlorotoluene	3.49	0.00	1.69	1.55	0.660	1.79	4.49
tert-Butylbenzene	1.22	1.92	2.15	0.810	0.270	0.770	2.15
Pentachloroethane	3.49	0.00	3.27	2.30	3.73	0.370	4.44
1,2,4-Trimethylbenzene	3.49	0.500	1.69	1.44	0.660	2.06	4.41
sec-Butylbenzene	4.55	1.02	0.980	1.39	0.350	1.32	3.34
1,3-Dichlorobenzene	4.55	0.510	1.46	1.39	0.200	1.84	2.94
4-Isopropyltoluene	3.49	0.00	1.92	0.97	0.270	1.39	3.20
1,4-Dichlorobenzene	3.49	0.510	1.92	1.12	0.150	1.61	2.75
1,2-Dichlorobenzene	3.49	0.00	1.46	1.02	0.150	1.22	2.93
n-Butylbenzene	4.55	0.510	1.69	1.55	0.120	1.73	3.63
Hexachloroethane	1.28	0.500	0.760	2.36	0.740	1.30	2.85
1,2-Dibromo-3-Chloropropane	1.22	0.500	0.00	2.41	1.36	2.70	2.38
1,2,4-Trichlorobenzene	3.49	2.08	0.250	1.44	1.15	0.200	0.850
Hexachlorobutadiene	1.28	1.02	0.00	1.92	1.67	0.800	0.350
Naphthalene	3.49	1.55	0.250	0.810	0.810	0.810	2.71
1,2,3-Trichlorobenzene	4.55	2.08	0.510	1.98	1.98	0.250	0.670

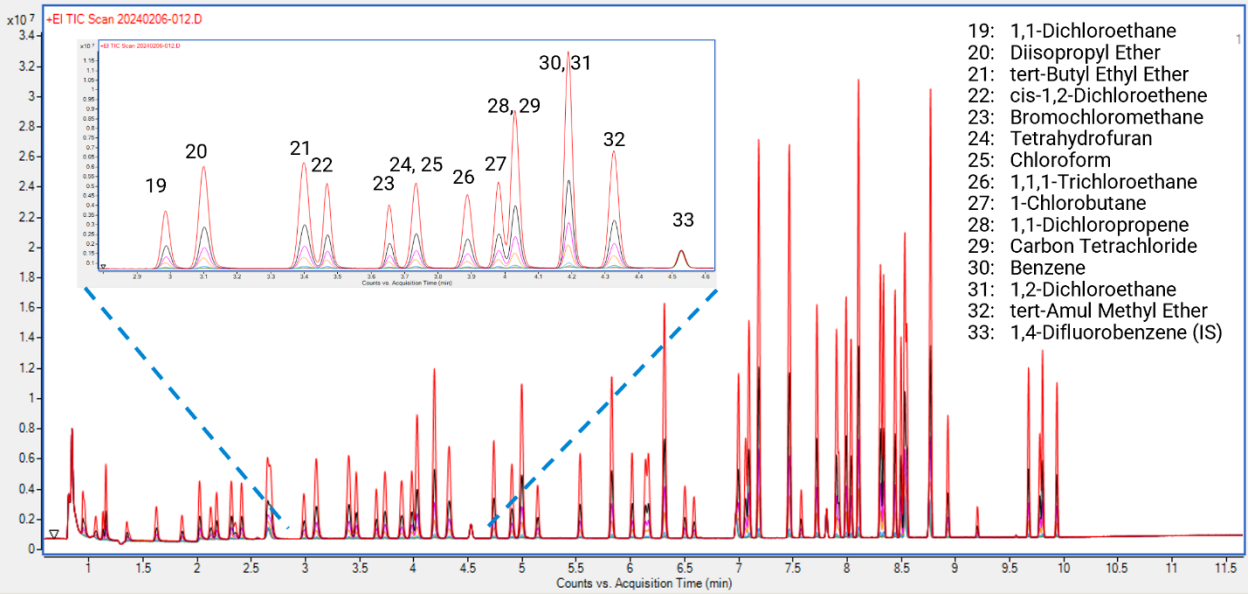

Figure 1 Total ion chromatogram (TIC) overlay of US EPA method 524.4 calibration standards, with an inset demonstrating excellent peak shape and separation across all concentrations with minimal water interference for all target compounds.

Conclusion

This study demonstrates the capability of the Tekmar Lumin P&T and AQUATek LVA system to process VOCs in drinking water samples following the US EPA Method 524.4 with detection by an Agilent 7890B GC/5977B MS with the HydroInert source. Utilizing the Lumin P&T's ability to purge with nitrogen, along with using Hydrogen as the GC/MS carrier gas with the HydroInert source, helps to conserve resources without sacrificing system performance.

The linearity of the calibration curve from 0.2 ppb to 50 ppb passed method requirements. Furthermore, the average MDL for all compounds was 0.09 ppb with a 5.9% RSD. Seven 10 ppb mid-point calibration check standards averaged a 96% recovery with a 3.3% RSD. The requirements for the MRL confirmation were met and the calibration curve was validated by the standards meeting the required true value percentages.

References

1. Munch, D.J..; Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry; US EPA Method 524.3 – Revision 1.0, June 2009.
2. Munch, D.J. and Wendelken, S.C.; Measurement of Purgeable Organic Compounds in Water by Gas Chromatography/Mass Spectrometry Using Nitrogen Purge Gas; US EPA Method 524.4 – May 2013