Chromatography
Application Note GCS-AN2402
July 26, 2024

US EPA Method 524.4 with the Tekmar Lumin P&T and the Thermo Scientific™ TRACE™ 1610 GC and ISQ™ 7610 MS System with an HeSaver-H_2Safer™ SSL Injector and an ExtractaBrite Source

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water testing, helium conservation, GC/MS, Thermo HeSaver-H_2Safer SSL Injector, Tekmar AQUATek LVA, Tekmar Lumin Purge and Trap, EPA Method 524.4

Abstract

As helium supplies become scarcer and more expensive, customers have been seeking alternative carrier gases or ways to conserve helium without sacrificing system performance. This analysis will evaluate the Teledyne LABS Tekmar Lumin Purge and Trap (P&T) concentrator along with the AQUATek LVA autosampler in conjunction with a Thermo Scientific TRACE 1610 Gas Chromatograph (GC) equipped with the HeSaver-H_2Safer™ SSL injector and the ISQ 7610 Mass Spectrometer (MS) with an ExtractaBrite source performing US EPA Method 524.4 to determine the concentration of Volatile Organic Compounds (VOCs) in drinking water matrices. Using nitrogen as the purge gas, along with the HeSaver-H_2Safer™ SSL injector, significantly reduces helium gas consumption during analysis. The method was validated by 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.

Figure 1 Lumin P&T Concentrator with AQUATek LVA Autosampler

Introduction

The Tekmar Lumin P&T has an innovative moisture control system (MCS) that improves water vapor removal by as much as 60%, thereby reducing peak interference and increasing GC column lifespan. The AQUATek LVA autosampler has an 84-position chiller enabled sample tray and 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. In addition to other refinements, the AQUATek LVA incorporates a precision-machined valve manifold block to reduce potential leak sources and ensure the system is both reliable and robust.

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	140 °C	Desorb Preheat Temp	245 °C
Transfer Line Temp	140 °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	3.00 min
Purge Temp	20 °C	Bake Temp	270 °C
Purge Time	8.00 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	0.30 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	0.35 min

Table II Thermo Scientific TRACE 1610 GC and ISQ 7610 MS System Conditions

Thermo Scientific TRACE 1610 GC Conditions	
Column	TG-VMS, 30 m x 0.25 mm, 1.4µm Film, Column Flow, Helium – 1.5 mL/min
Oven Profile	35 °C, 2 min, 15 °C/min to 100 °C, 30 °C/min to 225 °C, 1 min Hold, Run Time 12.5 min
Inlet	200 °C, 30:1 Split, Purge Flow 5.0 mL/min, 0.30 min Helium Delay
Thermo Scientific ISQ 7610 MS Conditions	
Temp	Transfer Line 230 °C; Ion Source 280 °C
Scan	Range 35 *m/z* to 260 *m/z*, Solvent Delay 1.40 min, Dwell/Scan Time 0.10 sec
Current	Emission Current 30 µA, Gain 3.00E+005

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 2 displays a 10 ppb calibration standard, 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**

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	Cal Type	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	HR^{PIR}	LPIR (≥50%)	UPIR (≤150%)
Dichlorodifluoromethane	1.48	85	Lin	0.997	0.445	0.06	4.1	0.08	85	117
Chlorodifluoromethane	1.52	51	Lin	0.997	0.877	0.14	9.0	0.18	65	136
Chloromethane	1.65	50	Lin	0.998	0.953	0.11	6.9	0.14	77	135
Vinyl Chloride	1.72	62	Lin	0.998	0.475	0.12	6.9	0.15	79	138
1,3-Butadiene	1.74	54	Lin	0.998	0.43	0.04	2.8	0.05	87	109
Bromomethane	2.02	94	Lin, W/Offset, 1/A	0.997	0.383	0.10	6.5	0.13	75	127
Trichlorofluoromethane	2.28	101	Lin	0.998	0.489	0.05	3.8	0.07	77	104
Diethyl Ether	2.58	59	Lin	0.999	0.250	0.05	2.7	0.06	103	127
1,1-Dichloroethene	2.75	96	Lin	0.999	0.168	0.11	6.9	0.14	75	131
Carbon Disulfide	2.76	76	Lin, W/Offset, 1/A	0.998	0.215	0.09	6.2	0.11	69	114
Iodomethane	2.87	142	Lin, W/Offset, 1/A	0.995	0.274	0.10	7.9	0.13	56	108
Allyl Chloride	3.18	41	Lin	0.998	0.138	0.12	6.8	0.15	84	145
Methylene Chloride	3.27	84	Lin	0.997	0.269	0.06	3.9	0.08	89	122
trans-1,2-Dichloroethene	3.40	96	Lin	0.998	0.189	0.10	5.6	0.13	89	139
Methyl Acetate	3.43	43	Lin	0.999	0.320	0.14	9.4	0.18	61	133
Methyl-tert-Butyl Ether-d3 (SURR)	3.49	76	AvgCalFact	2.09	1.08		1.5	0.57	94	105
Methyl-tert-Butyl Ether	3.50	73	Lin	0.999	0.980	0.05	2.6	0.06	102	126
tert-Butyl Alcohol	3.59	59	Lin	0.999	0.040	0.08	4.6	0.10	91	131
Diisopropyl Ether	3.83	45	Lin	1.00	1.55	0.06	3.6	0.07	85	113
1,1-Dichloroethane	3.91	63	Lin	0.998	0.549	0.07	3.9	0.09	94	129
tert-Butyl Ethyl Ether	4.12	59	Lin	1.00	1.04	0.05	3.4	0.07	88	115
cis-1,2-Dichloroethene	4.35	96	Lin	0.999	0.298	0.06	3.2	0.07	100	129
Bromochloromethane	4.50	128	Lin	0.999	0.137	0.07	4.0	0.09	93	129
Chloroform	4.57	83	Lin	0.998	0.574	0.06	3.4	0.08	99	130
Carbon Tetrachloride	4.67	117	Lin	0.999	0.261	0.10	6.5	0.13	73	123
Tetrahydrofuran	4.68	72	Lin	0.999	0.045	0.13	7.1	0.16	82	146

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	Cal Type	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	HR^{PIR}	LPIR (≥50%)	UPIR (≤150%)
1,1,1-Trichloroehane	4.72	97	Lin	0.998	0.380	0.09	5.6	0.11	79	125
1,1-Dichloropropene	4.82	75	Lin	0.998	0.098	0.10	6.1	0.12	76	124
1-Chlorobutane	4.86	56	Lin	0.999	0.436	0.06	4.0	0.08	86	118
Benzene	5.05	78	Lin	1.00	1.04	0.06	3.3	0.07	94	123
tert-Amyl Methyl Ether	5.11	73	Lin	1.00	0.905	0.06	3.8	0.07	82	111
1,2-Dichloroethane	5.17	62	Lin	0.998	0.401	0.06	3.3	0.07	97	126
Trichloroethylene	5.47	132	Lin	1.00	0.236	0.07	4.9	0.10	87	128
1,4-Difluorobenzene (IS)	5.50	114	AvgCalFact							
tert-Amyl Ethyl Ether	5.67	59	Lin	1.00	0.808	0.04	2.3	0.05	91	109
Dibromomethane	5.80	93	Lin	0.998	0.202	0.05	2.9	0.06	98	124
1,2-Dichloropropane	5.88	63	Lin	0.999	0.365	0.04	2.4	0.05	100	121
Bromodichloromethane	5.94	83	Lin	0.999	0.448	0.08	4.6	0.10	85	124
cis-1,3-Dichloropropene	6.44	75	Lin	1.00	0.567	0.09	5.5	0.12	82	128
Toluene	6.63	92	Lin, W/Offset, 1/A	1.00	0.948	0.06	4.4	0.08	72	102
Tetrachloroethylene	6.91	166	Lin	0.999	0.455	0.10	6.0	0.13	82	133
trans-1,3-Dichloropropene	6.93	75	Lin	1.00	0.560	0.04	2.5	0.05	94	115
1,1,2-Trichloroethane	7.05	83	Lin	1.00	0.325	0.07	4.4	0.09	85	122
Ethyl Methacrylate	7.05	69	Lin	1.00	0.468	0.07	4.2	0.08	84	118
Dibromochloromethane	7.17	129	Lin	1.00	0.337	0.04	2.4	0.05	90	109
1,3-Dichloropropane	7.24	76	Lin	1.00	0.608	0.07	3.9	0.08	91	125
1,2-Dibromoethane	7.34	107	Lin	0.999	0.329	0.06	3.4	0.07	91	119
Chlorobenzene-d5 (IS)	7.68	117	AvgCalFact							
Chlorobenzene	7.69	112	Lin	1.00	0.876	0.05	2.9	0.06	92	116
Ethylbenzene	7.71	91	Lin	0.998	1.44	0.06	3.7	0.08	93	125
1,1,1,2-Tetrachloroethane	7.73	131	Lin	1.00	0.305	0.04	2.5	0.05	87	106
m,p-Xylene	7.81	106	Lin	0.999	0.567	0.10	3.3	0.13	88	115
o-Xylene	8.09	106	Lin	0.999	0.607	0.05	3.3	0.07	89	116
Styrene	8.12	104	Lin	0.999	0.994	0.05	3.3	0.07	90	117
Bromoform	8.14	173	Lin	0.999	0.283	0.06	4.6	0.08	71	103
Isopropylbenzene	8.29	105	Lin	1.00	1.52	0.07	4.5	0.08	78	112
4-Bromofluorobenzene (SURR)	8.47	95	AvgCalFact	3.20	0.799		2.1	0.83	93	110

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	Cal Type	Linearity (r^2 ≥0.995)	Avg. RRF	MDL (ppb)	Precision (≤20%)	HR^{PIR}	LPIR (≥50%)	UPIR (≤150%)
Bromobenzene	8.53	156	Lin	1.00	0.694	0.05	3.0	0.07	97	124
n-Propylbenzene	8.55	91	Lin	0.999	2.78	0.08	4.7	0.10	86	125
1,1,2,2-Tetrachloroethane	8.59	83	Lin	0.999	0.747	0.04	2.2	0.05	96	115
2-Chlorotoluene	8.64	91	Lin	1.00	0.551	0.05	2.9	0.06	97	122
1,3,5-Trimethylbenzene	8.66	105	Lin	0.998	1.99	0.07	4.5	0.09	86	124
1,2,3-Trichloropropane	8.68	110	Lin	0.998	0.187	0.07	4.0	0.09	91	125
4-Chlorotoluene	8.75	91	Lin	0.999	1.83	0.07	3.9	0.08	93	127
tert-Butylbenzene	8.86	119	Lin	1.00	0.570	0.07	4.6	0.09	83	119
Pentachloroethane	8.88	167	Lin	0.999	0.243	0.13	10.0	0.17	52	120
1,2,4-Trimethylbenzene	8.90	105	Lin	0.999	2.08	0.05	3.0	0.06	94	119
sec-Butylbenzene	8.97	105	Lin	0.999	2.66	0.05	3.5	0.07	84	111
4-Isopropyltoluene	9.05	119	Lin	1.00	2.18	0.04	2.7	0.05	87	108
1,3-Dichlorobenzene	9.10	146	Lin	0.999	1.50	0.04	2.2	0.05	100	119
1,4-Dichlorobenzene-d4 (IS)	9.15	152	AvgCalFact							
1,4-Dichlorobenzene	9.16	146	Lin	0.999	1.56	0.06	3.2	0.07	96	124
n-Butylbenzene	9.30	91	Lin	0.999	0.749	0.07	4.6	0.08	75	108
Hexachloroethane	9.39	201	Lin	0.997	0.296	0.12	6.7	0.16	87	150
1,2-Dichlorobenzene-d4 (SURR)	9.40	152	AvgCalFact	1.93	1.01		0.8	0.32	96	102
1,2-Dichlorobenzene	9.41	146	Lin	0.999	1.59	0.05	2.9	0.06	96	121
1,2-Dibromo-3-Chloropropane	9.88	157	Lin	0.999	0.220	0.06	3.8	0.08	90	122
Hexachlorobutadiene	10.25	225	Lin	0.999	0.073	0.06	3.7	0.07	88	118
1,2,4-Trichlorobenzene	10.27	180	Lin	1.00	1.28	0.05	3.5	0.07	86	113
Naphthalene	10.46	128	Lin	0.999	2.89	0.04	2.7	0.05	90	111
1,2,3-Trichlorobenzene	10.57	180	Lin	0.999	1.22	0.04	2.8	0.06	91	114

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

Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
Dichlorodifluoromethane	10.3	8.6	103
Chlorodifluoromethane	10.3	4.9	103
Chloromethane	10.3	5.1	103
Vinyl Chloride	10.8	4.9	108
1,3-Butadiene	10.6	4.0	106
Bromomethane	10.3	4.0	103
Trichlorofluoromethane	10.4	4.0	104
Diethyl Ether	10.6	2.3	106
1,1-Dichloroethene	10.5	4.1	105
Carbon Disulfide	10.4	3.8	104
Iodomethane	9.7	2.5	97
Allyl Chloride	10.4	2.9	104
Methylene Chloride	10.6	1.9	106
trans-1,2-Dichloroethene	10.6	3.6	106
Methyl Acetate	10.2	2.7	102
Methyl-tert-Butyl Ether-d3 (SURR)	9.9	1.6	99
Methyl-tert-Butyl Ether	10.3	2.4	103
tert-Butyl Alcohol	10.6	4.2	106
Diisopropyl Ether	10.0	2.4	100
1,1-Dichloroethane	10.5	3.5	105
tert-Butyl Ethyl Ether	10.1	2.4	101
cis-1,2-Dichloroethene	10.2	2.7	102
Bromochloromethane	10.4	2.6	104
Chloroform	10.4	2.3	104
Carbon Tetrachloride	10.2	4.5	102
Tetrahydrofuran	10.3	2.5	103
1,1,1-Trichloroehane	10.3	3.4	103
1,1-Dichloropropene	10.4	4.1	104
1-Chlorobutane	10.4	4.3	104
Benzene	10.2	2.8	102
tert-Amyl Methyl Ether	9.8	1.9	98
1,2-Dichloroethane	10.4	2.2	104
Trichloroethylene	10.3	3.6	103
1,4-Difluorobenzene (IS)			

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

Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
tert-Amyl Ethyl Ether	9.9	2.0	99
Dibromomethane	10.4	2.0	104
1,2-Dichloropropane	10.4	2.3	104
Bromodichloromethane	10.0	1.8	100
cis-1,3-Dichloropropene	10.0	2.6	100
Toluene	9.8	3.7	98
Tetrachloroethylene	11.7	6.3	117
trans-1,3-Dichloropropene	10.0	2.9	100
1,1,2-Trichloroethane	9.7	3.0	97
Ethyl Methacrylate	9.8	2.2	98
Dibromochloromethane	9.9	2.7	99
1,3-Dichloropropane	10.1	2.8	101
1,2-Dibromoethane	10.1	2.6	101
Chlorobenzene-d5 (IS)			
Chlorobenzene	9.9	3.3	99
Ethylbenzene	10.3	4.4	103
1,1,1,2-Tetrachloroethane	9.8	3.0	98
m,p-Xylene	20.0	4.0	100
o-Xylene	9.9	3.9	99
Styrene	9.9	4.9	99
Bromoform	9.2	2.5	92
Isopropylbenzene	9.8	4.0	98
4-Bromofluorobenzene (SURR)	9.8	1.5	98
Bromobenzene	10.4	2.0	104
n-Propylbenzene	10.4	3.1	104
1,1,2,2-Tetrachloroethane	10.2	2.3	102
2-Chlorotoluene	10.2	2.9	102
1,3,5-Trimethylbenzene	10.5	2.6	105
1,2,3-Trichloropropane	10.4	3.1	104
4-Chlorotoluene	10.4	2.3	104
tert-Butylbenzene	9.7	3.6	97
Pentachloroethane	10.2	8.7	102
1,2,4-Trimethylbenzene	10.5	2.9	105
sec-Butylbenzene	10.3	3.5	103

Table IV US EPA 524.4 Mid-Point Calibration Check Data (cont)			
Compounds	Avg. Conc.	Precision (≤20%)	Accuracy (±20%)
4-Isopropyltoluene	10.0	3.3	100
1,3-Dichlorobenzene	10.2	2.1	104
1,4-Dichlorobenzene-d4 (IS)			
1,4-Dichlorobenzene	10.2	1.0	102
n-Butylbenzene	9.5	3.3	95
Hexachloroethane	10.9	3.4	109
1,2-Dichlorobenzene-d4 (SURR)	9.9	2.1	99
1,2-Dichlorobenzene	10.2	2.2	102
1,2-Dibromo-3-Chloropropane	10.2	2.2	102
Hexachlorobutadiene	10.4	4.6	104
1,2,4-Trichlorobenzene	9.7	2.8	97
Naphthalene	9.9	2.8	99
1,2,3-Trichlorobenzene	9.9	2.4	99

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	43.3	3.3	16.3	1.7	5.9	9.5	1.8
Chlorodifluoromethane	3.9	0.3	16.7	0.6	5.2	9.7	1.8
Chloromethane	18.8	11.6	11.3	13.2	3.5	8.1	1.6
Vinyl Chloride	11.8	12.3	13.5	1.9	8.2	7.5	1.6
1,3-Butadiene	31.6	3.4	17.7	4.7	7.9	8.0	1.7
Bromomethane	15.9	10.2	9.0	11.5	4.4	5.5	4.4
Trichlorofluoromethane	6.1	10.1	9.9	2.8	4.5	8.5	1.5
Diethyl Ether	19.3	12.9	7.7	5.7	5.7	4.8	1.1
1,1-Dichloroethene	4.2	5.6	5.1	4.0	2.9	7.2	1.3
Carbon Disulfide	7.7	3.7	3.5	2.8	2.6	5.7	3.1
Iodomethane	33.0	19.0	29.6	20.2	14.1	3.4	0.3
Allyl Chloride	4.5	4.7	7.0	6.8	4.4	7.2	1.4
Methylene Chloride	14.8	15.2	7.2	13.8	5.4	8.9	1.8
trans-1,2-Dichloroethene	31.3	2.1	8.1	8.0	4.6	7.8	1.5
Methyl Acetate	8.3	4.5	3.2	4.9	5.8	5.3	1.1
Methyl-tert-butyl Ether-d3 (SURR)	3.4	1.0	0.9	1.8	2.0	0.8	2.3

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%)
Methyl-tert-Butyl Ether	9.7	13.7	6.4	6.0	2.5	4.2	0.8
tert-Butyl Alcohol	1.8	6.1	6.6	2.1	8.5	2.5	0.7
Diisopropyl Ether	9.5	1.8	1.1	3.1	1.8	3.6	0.7
1,1-Dichloroethane	6.1	14.9	7.4	11.9	5.2	8.5	1.7
tert-Butyl Ethyl Ether	10.7	2.2	0.1	2.4	2.9	3.2	0.7
cis-1,2-Dichloroethene	4.6	17.7	4.4	10.2	4.2	5.2	1.1
Bromochloromethane	41.6	11.2	6.3	10.6	4.2	5.6	1.2
Chloroform	25.5	21.0	2.1	11.9	5.3	7.0	1.5
Carbon Tetrachloride	11.7	5.2	4.1	2.9	2.3	7.1	1.2
Tetrahydrofuran	3.3	3.3	3.2	2.0	5.2	6.8	1.3
1,1,1-Trichloroehane	10.6	5.5	4.6	4.1	4.3	7.6	1.4
1,1-Dichloropropene	9.5	2.2	1.6	1.8	4.4	9.0	1.6
1-Chlorobutane	11.1	7.4	7.5	2.1	3.2	7.0	1.3
Benzene	15.7	9.7	0.8	4.5	0.4	4.1	0.7
tert-Amyl Methyl Ether	5.9	7.6	4.3	0.7	0.2	3.5	0.6
1,2-Dichloroethane	17.6	15.7	9.7	10.9	7.0	6.5	1.5
Trichloroethylene	20.4	2.5	4.1	4.0	1.5	3.6	0.7
tert-Amyl Ethyl Ether	5.9	1.1	4.1	0.8	1.0	1.6	0.3
Dibromomethane	20.4	12.8	4.3	12.9	4.8	8.6	1.7
1,2-Dichloropropane	15.2	8.9	6.2	7.4	4.6	5.2	1.1
Bromodichloromethane	20.3	5.6	0.2	4.6	0.5	5.0	0.8
cis-1,3-Dichloropropene	18.4	10.8	1.6	4.4	1.7	3.8	0.7
Toluene	0.8	3.0	1.1	0.3	1.0	3.6	1.6
Tetrachloroethylene	8.8	12.4	9.5	5.7	2.2	5.5	0.7
trans-1,3-Dichloropropene	19.8	6.9	0.7	3.6	0.8	4.1	0.7
Ethyl Methacrylate	7.0	0.2	2.7	1.2	1.4	3.0	0.5
1,1,2-Trichloroethane	7.5	2.3	2.7	3.9	1.0	2.4	0.5
Dibromochloromethane	8.1	1.8	6.6	0.1	1.5	3.1	0.4
1,3-Dichloropropane	17.6	9.0	2.4	6.7	2.7	3.8	0.8
1,2-Dibromoethane	18.6	5.7	2.0	3.6	3.4	5.5	1.1
Chlorobenzene	13.1	2.5	0.4	3.8	0.7	3.1	0.6
Ethylbenzene	16.7	9.7	2.9	5.4	4.1	8.6	1.6
1,1,1,2-Tetrachloroethane	1.5	0.7	4.7	0.6	1.2	2.1	0.4

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	6.0	1.3	2.4	2.5	0.3	6.1	1.0
o-Xylene	4.8	11.8	2.8	1.1	0.7	5.0	0.8
Styrene	13.6	1.0	3.7	1.0	0.5	4.9	0.8
Bromoform	12.8	19.4	16.2	10.8	7.7	0.5	0.4
Isopropylbenzene	2.0	3.2	3.7	4.0	3.6	3.5	0.4
4-Bromofluorobenzene (SURR)	2.5	2.3	1.9	3.3	2.1	3.8	4.1
Bromobenzene	23.8	12.8	1.7	9.9	0.6	3.6	0.7
n-Propylbenzene	16.4	5.8	3.7	5.6	3.8	6.2	1.2
1,1,2,2-Tetrachloroethane	14.1	9.3	4.2	7.3	4.0	6.0	1.2
2-Chlorotoluene	17.7	8.8	0.1	5.1	0.9	1.1	0.3
1,3,5-Trimethylbenzene	10.9	4.8	1.6	7.6	3.8	7.6	1.4
1,2,3-Trichloropropane	12.8	7.9	0.9	6.6	7.4	6.5	1.4
4-Chlorotoluene	19.1	8.3	3.9	8.6	3.3	6.2	1.2
tert-Butylbenzene	8.3	1.7	1.9	0.0	1.5	0.7	0.1
Pentachloroethane	8.9	15.2	5.3	4.1	3.9	5.5	1.0
1,2,4-Trimethylbenzene	9.8	6.7	2.5	8.5	3.1	5.6	1.1
sec-Butylbenzene	9.6	3.3	1.4	0.4	2.0	7.1	1.2
4-Isopropyltoluene	5.3	3.5	3.1	1.5	2.5	3.8	0.5
1,3-Dichlorobenzene	25.1	8.8	4.6	6.9	0.4	5.6	1.0
1,4-Dichlorobenzene	28.3	8.1	3.7	8.7	3.3	5.9	1.2
n-Butylbenzene	0.9	9.8	7.9	5.7	6.1	4.5	0.4
Hexachloroethane	14.0	13.8	17.6	12.8	7.3	10.3	2.1
1,2-Dichlorobenzene (SURR)	0.3	0.7	2.0	0.3	2.6	2.1	2.4
1,2-Dichlorobenzene	19.3	8.4	3.6	8.1	1.0	5.4	1.0
1,2-Dibromo-3-Chloropropane	13.7	10.6	1.3	0.5	0.2	4.7	0.8
Hexachlorobutadiene	31.7	5.1	11.9	3.1	2.3	6.1	1.1
1,2,4-Trichlorobenzene	21.2	3.3	3.7	1.6	5.1	2.3	0.1
Naphthalene	25.1	5.5	1.0	1.1	1.8	6.5	1.0
1,2,3-Trichlorobenzene	23.8	5.6	0.4	1.4	1.1	7.2	1.1

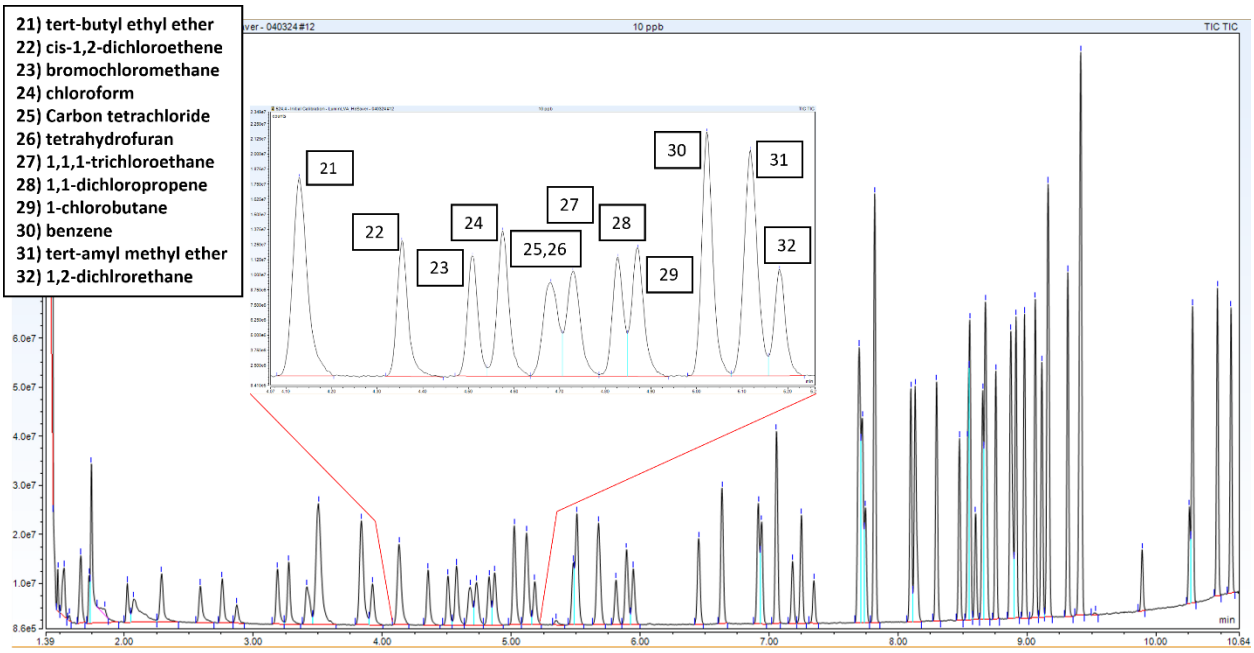

Figure 2 Total ion chromatogram (TIC) of a US EPA method 524.4 10 ppb calibration standard, with an inset demonstrating excellent peak shape and separation across all concentrations with minimal water interference for all target compounds.

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 Thermo Scientific TRACE 1610 GC and the ISQ 7610 MS. Utilizing the Lumin P&T's ability to purge with nitrogen, along with using Thermo's HeSaver-H_2Safer™ SSL injector, helium was conserved during this analysis 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 0.07 ppb with a 4.2% RSD. Seven 10 ppb mid-point calibration check standards averaged a 102% recovery with a 3.2% 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