Application Note

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US EPA Method 524.2: Successful Measurement of Purgeable Organic Compounds in Drinking Water by Agilent 8860/5977B GC/MSD

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Abstract

An Agilent 8860/5977B GC/MSD system coupled with a Teledyne Tekmar Lumin purge and trap (P&T) concentrator and an AQUATek liquid vial autosampler (LVA) was successfully used for the analysis of volatile organic compounds (VOCs) to the requirements of United States Environmental Protection Agency (US EPA) method 524.2. The analysis of VOCs in water following this or similar methods are widely used around the world as part of insuring the safety of potable water supplies.

Introduction

US EPA method 524.2 provides procedures and requirements for the quantitation of VOCs in water by GC/MS[1]. A challenge presented by this method is the potentially large amount of water vapor transferred to the GC/MS system due to the recommended four-minute desorb time from the analytical trap. The GC inlet, column, and MSD could be sensitive to water, which could result in a performance decline over the course of analysis.

This Application Note demonstrates a configuration capable of successfully meeting and exceeding the requirements of EPA method 524.2 in analyzing VOCs with a previously optimized P&T method:[2]

- Recommended GC and MS consumables including drawout lens, liner, and column, along with methods and other materials provided by Agilent Technologies to produce the required performance
- An automated tuning approach to reliably satisfy EPA tuning requirements for 4-bromofluorobenzene (BFB), and to improve sensitivity
- Teledyne Tekmar's Lumin P&T with innovative moisture control system (MCS) specifically designed to reduce the amount of water transferred to the Agilent GC/MSD during desorb
- P&T parameters for the Teledyne Tekmar Lumin P&T concentrator, and an AQUATek LVA that enable reliable and precise sample preparation and handling
- Software tools provided with Agilent MassHunter 10, including Unknowns Analysis for compound identification, Library Editor for creating custom libraries, and Quantitative Analysis for creating a quant method from acquired data with a library search.

VOC data included in this Application Note demonstrate the required performance for the initial calibration (ICAL) and method detection limit (MDL) studies, in terms of linearity and dynamic working range.

Experimental

The Agilent 5977B mass spectrometer (MS) was coupled to the Agilent 8860 GC equipped with a Split/Splitless (SSL) Inlet, the Teledyne Tekmar Lumin P&T concentrator, and an AQUATek LVA. The MSD was autotuned using an automated BFB tuning algorithm. The analytical method used an Agilent Ultra Inert straight-through 1.0 mm GC inlet liner (p/n 5190-4047) and a DB-624 UI, 20 m × 0.18 mm, 1 µm column (p/n 121-1324UI). The Teledyne Tekmar Lumin P&T was connected to the GC carrier gas inlet line between the GC control pneumatics and the GC injection port. The split ratio was set to 150:1. The stainless steel ion source (p/n G3870-67750) was equipped with a 6 mm diameter drawout lens (p/n G3163-20530).

Seven calibration levels ranging from 0.25 to 50 µg/L were prepared in water by spiking 10 µL of a corresponding stock solution into water in a 40 mL vial with septum. Removing 300 µL of water from a 40 mL vial filled to the very top (or the brim) prior to a spike with a stock solution allowed for the formation of an air bubble not exceeding 5 to 6 mm in diameter to ensure effective mixing. The spiking stock solutions were prepared using 60- and 24-compound standards mixes (AccuStandard M-502-10X and M-524R-B, respectively). A fortification solution containing fluorobenzene (internal standard), 1,2-dichlorobenzene-d_4 (surrogate), and BFB (surrogate) in methanol at 5 µg/mL each was prepared using a three-compound mix (AccuStandard M-524-FS). A 5 µL aliquot of the fortification solution was automatically added to a 5 mL water sample volume within the Lumin P&T Concentrator, giving a concentration of 5 µg/L of ISTD and the surrogates. MassHunter Workstation software was used for data acquisition and processing. Figure 1 shows the system configuration used. Table 1 lists the Agilent GC/MSD operating parameters. Table 2 shows the Lumin P&T concentrator and AQUATek LVA instrument operating conditions. Table 3 summarizes the target VOCs and their suggested quantitation ions.

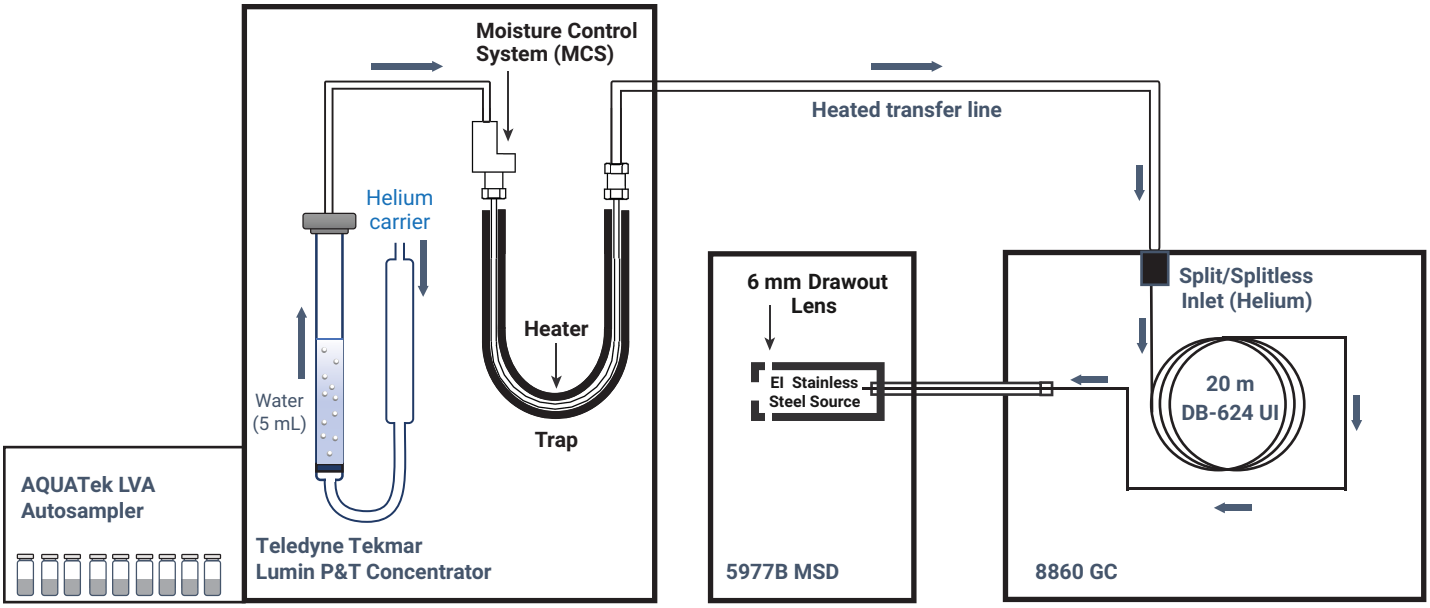


Figure 1. Instrument configuration.

Table 1. GC/MS conditions for VOCs analysis.

8860 GC	
Inlet	Split/Splitless (SSL)
Mode	Split
Split Ratio	150:1
Total Flow	100.07 mL/min
Carrier Gas	Helium
Inlet Liner	Straight-through 1.0 mm UI Liner
Inlet Liner Part Number	5190-4047
Injection Source	External device
Oven	
Initial Oven Temperature	35 °C
Initial Oven Hold	4 minutes
Ramp Rate 1	15 °C/min
Final Temp 1	240 °C
Final Hold 1	0.3333 minutes
Total Run Time	18 minutes
Equilibration Time	0 minutes
Column	
Type	DB-624 UI
Part Number	121-1324UI
Length	20 m
Diameter	0.18 mm
Film Thickness	1 µm

Control Mode	Constant Flow
Flow	0.663 mL/min
Inlet Connection	Split/Splitless Inlet (SSL)
Outlet Connection	MSD
MSD	
Model	5977B
Source	Stainless Steel Source
Vacuum Pump	Performance Turbo
Tune File	BFB_Atune.u
Mode	Scan
Solvent Delay	1.05 minutes
EM Voltage Gain Mode	5
Trace Ion Detection	On
Quad Temperature	200 °C
Source Temperature	250 °C
Transfer Line Temperature	250 °C
Scan Parameters	
Low Mass	35
High Mass	260
Threshold	0
Sample	2
	A/D samples: 4

Table 2. Teledyne Tekmar Lumin P&T/AQUATek LVA conditions for VOCs analysis.

Instrument type: Tekmar Lumin P&T/AQUATek LVA	
Standby	
Valve Oven Temperature	125 °C
Transfer Line Temperature	125 °C
Sample Mount Temperature	40 °C
Standby Flow	10 mL/min
Purge Ready Temperature	45 °C
MCS Purge Temperature	20 °C
Purge	
Purge Temperature	20 °C
Purge Time	11.00 minutes
Purge Flow	40 mL/min
Dry Purge Temperature	20 °C
Dry Purge Time	2.00 minutes
Dry Purge Flow	100 mL/min
Sample Temperature	40 °C
Prepurge Time	0.50 minutes
Prepurge Flow	40 mL/min
Preheat Time	1.00 minute
Sample Heater Enable	Off
Desorb	
Desorb Preheat Temperature	245 °C
Desorb Temperature	250 °C
Desorb Time	4.00 minutes
Drain Flow	400 mL/min
GC Start Signal	Begin Desorb
Bake	
Bake Time	6.00 minutes
Bake Temperature	260 °C
MCS Bake Temperature	200 °C
Bake Flow	200 mL/min
AQUATek LVA	
Sample Loop Time	0.50 minutes
Sample Transfer Time	0.50 minutes
Rinse Loop Time	0.25 minutes
Sweep Needle Time	0.30 minutes
Presweep Time	0.25 minutes
Water Temp	80 °C
Bake Rinse Cycles	3
Bake Rinse Drain Time	0.25 minutes

Table 3. VOC List with retention times and suggested quantitation ions.

Compound	Retention Time (min)	Target *m/z*	Qualifier 1 *m/z*	Qualifier 2 *m/z*	Qualifier 3 *m/z*
Fluorobenzene (ISTD)	6.613	96.0	77.0		
Dichlorodifluoromethane	1.207	85.0	87.0		
Chloromethane	1.362	50.0	52.0		
Chloroethene	1.462	62.0	64.0		
Bromomethane	1.752	94.0	96.0		
Ethyl Chloride	1.858	64.0	66.0	49.0	
Trichloromonofluoromethane	2.120	101.0	103.0		
Ethyl Ether	2.466	74.0	59.0	45.0	
1,1-Dichloroethene	2.683	61.0	96.0	98.0	63.0
Acetone	2.793	58.0	43.0		
Iodomethane	2.841	142.0	127.0		
Carbon Disulfide	2.907	76.0			
Allyl Chloride	3.145	76.0	41.0	39.0	
Methylene Chloride	3.310	84.0	49.0	86.0	47.0
Acrylonitrile	3.696	52.0	53.0	51.0	
trans-1,2-Dichloroethylene	3.709	61.0	96.0	98.0	63.0
Methyl *tert*-butyl Ether	3.787	73.0	57.0	43.0	
1,1-Dichloroethane	4.370	63.0	65.0		
2,2-Dichloropropane	5.192	77.0	79.0	97.0	
cis-1,2-Dichloroethylene	5.202	61.0	96.0	98.0	63.0
2-Butanone	5.296	72.0	43.0		
Propanenitrile	5.346	54.0	52.0		
Methyl Acrylate	5.424	55.0	85.0	42.0	
Bromochloromethane	5.503	130.0	128.0	49.0	132.0
Methylacrylonitrile	5.535	67.0	52.0	66.0	
Tetrahydrofuran	5.615	72.0	71.0		
Trichloromethane	5.640	83.0	85.0	47.0	
1,1,1-Trichloroethane	5.831	97.0	99.0	61.0	
1-Chlorobutane	5.985	56.0	49.0		
Carbon Tetrachloride	6.029	117.0	119.0	47.0	121.0
1,1-Dichloropropene	6.038	75.0	110.0	112.0	77.0
Benzene	6.274	78.0	77.0		
1,2-Dichloroethane	6.308	62.0	49.0	64.0	
Trichloroethylene	7.023	130.0	132.0	95.0	97.0
1,2-Dichloropropane	7.261	63.0	62.0	76.0	65.0
Dibromomethane	7.379	174.0	172.0	176.0	93.0
Methyl Methacrylate	7.453	100.0	69.0	99	
Bromodichloromethane	7.568	83.0	85.0		
2-Nitropropane	7.820	43.0	41.0		
cis-1,3-Dichloropropene	8.044	75.0	110.0	77.0	
2,2-Dimethoxybutane	8.105	89.0	87.0	55.0	
Methyl Isobutyl Ketone (MIBK)	8.237	58.0	43.0	41.0	

Table 3. VOC List with the retention times and suggested quantitation ions (continued).

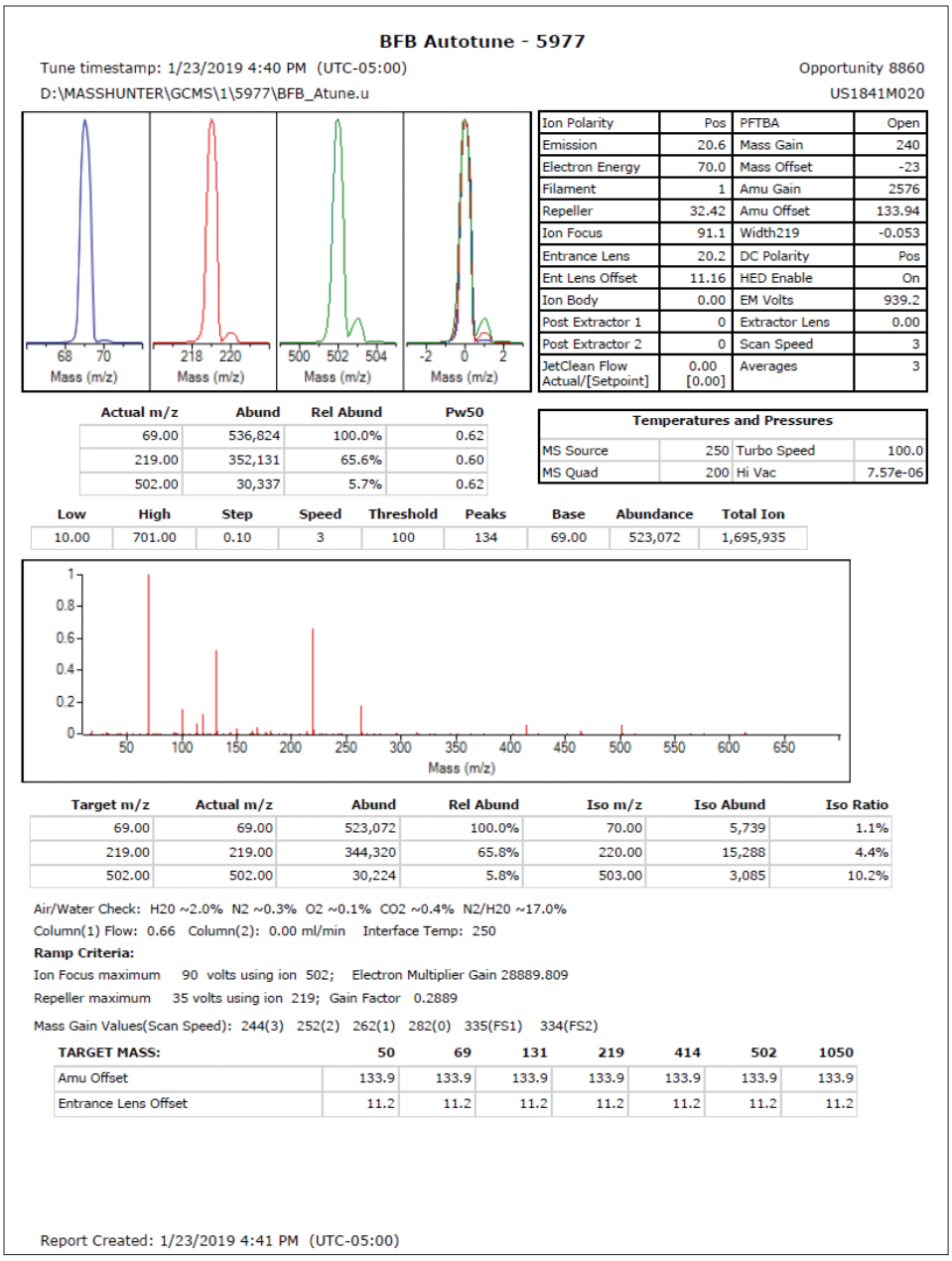
Compound	Retention Time (min)	Target *m/z*	Qualifier 1 *m/z*	Qualifier 2 *m/z*	Qualifier 3 *m/z*
Toluene	8.380	91.0	92.0		
trans-1,3-Dichloropropene	8.619	75.0	110.0	77.0	
Ethyl Methacrylate	8.748	69.0	41.0	39.0	
1,1,2-Trichloroethane	8.797	97.0	99.0	83.0	61.0
Tetrachloroethylene	8.933	164.0	166.0	129.0	168.0
1,3-Dichloropropane	8.961	76.0	78.0		
2-Hexanone	9.082	58.0	43.0	57.0	
Dibromochloromethane	9.181	129.0	127.0	131.0	
1,2-Dibromoethane	9.284	109.0	107.0		
Chlorobenzene	9.787	112.0	114.0	77.0	
1,1,1,2-Tetrachloroethane	9.875	133.0	131.0	117.0	119.0
Ethylbenzene	9.909	91.0	106.0		
m+p-Xylene	10.028	91.0	106.0	105.0	
o-Xylene	10.418	91.0	106.0	105.0	
Styrene	10.431	104.0	103.0	78.0	105.0
Tribromomethane	10.600	173.0	171.0	175.0	79.0
Isopropylbenzene	10.791	105.0	120.0		
p-Bromofluorobenzene (SURR)	10.933	174.0	176.0	95.0	75.0
Bromobenzene	11.074	158.0	156.0	77.0	50.0
1,1,2,2-Tetrachloroethane	11.083	83.0	85.0		
1,2,3-Trichloropropane	11.121	75.0	110.0	112.0	61.0
1,4-Dichlorobut-2-ene	11.142	89.0	88.0	53.0	124.0
Propylbenzene	11.200	91.0	120.0		
2-Chlorotoluene	11.274	91.0	126.0		
Mesitylene (1,3,5-Trimethylbenzene)	11.379	105.0	120.0		
tert-Butylbenzene	11.701	119.0	91.0	134.0	
1,2,4-Trimethylbenzene	11.748	105.0	120.0		
1-Methylpropyl Benzene	11.919	105.0	134.0		
1,3-Dichlorobenzene	12.014	146.0	148.0	111.0	75.0
p-Cymene (4-Isopropyltoluene)	12.067	119.0	134.0	91.0	
1,4-Dichlorobenzene	12.102	146.0	148.0	111.0	75.0
1,2-Dichlorobenzene-d_4 (SURR)	12.452	152.0	150.0	115.0	
1,2-Dichlorobenzene	12.470	146.0	148.0	111.0	75.0
n-Butylbenzene	12.473	91.0	92.0	134.0	
Hexachloroethane	12.727	166.0	164.0	201.0	203.0
1,2-Dibromo-3-chloropropane	13.241	155.0	75.0	157.0	159.0
Nitrobenzene	13.446	93.0	123.0	77.0	51.0
1,2,4-Trichlorobenzene	14.072	180.0	182.0	145.0	184.0
1,1,2,3,4,4-Hexachlorobuta-1,3-diene	14.256	225.0	227.0	262.0	260.0
Naphthalene	14.311	128.0	127.0	129.0	
1,2,3-Trichlorobenzene	14.554	180.0	182.0	145.0	184.0

Meeting BFB ion abundance criteria with BFB Autotune

Tuning MSD

The BFB Autotune process for Agilent MSD systems was designed to support the hardware configuration used for VOC analysis, and provides the best system performance while maintaining BFB spectral ion ratios established by the EPA.[2] The MSD hardware configuration included a stainless steel ion source (p/n G3870-67750) equipped with a 6 mm diameter drawout lens (p/n G3163-20530). For optimal operation, the ion source and quadrupole temperatures were set to 250 °C and 200 °C, respectively.

BFB Autotune (BFB_Atune.U) is provided in MassHunter Acquisition on the Tune dropdown menu, in the Tune and Vacuum Control view. Figure 2 shows an example of the BFB_ATUNE.U file report.



BFB Autotune - 5977

Tune timestamp: 1/23/2019 4:40 PM (UTC-05:00) Opportunity 8860

D:\MASSHUNTER\GCMS\1\5977\BFB_Atune.u US1841M020

Ion Polarity	Pos	PFTBA	Open
Emission	20.6	Mass Gain	240
Electron Energy	70.0	Mass Offset	-23
Filament	1	Amu Gain	2576
Repeller	32.42	Amu Offset	133.94
Ion Focus	91.1	Width219	-0.053
Entrance Lens	20.2	DC Polarity	Pos
Ent Lens Offset	11.16	HED Enable	On
Ion Body	0.00	EM Volts	939.2
Post Extractor 1	0	Extractor Lens	0.00
Post Extractor 2	0	Scan Speed	3
JetClean Flow Actual/[Setpoint]	0.00 [0.00]	Averages	3

Actual m/z	Abund	Rel Abund	Pw50
69.00	536,824	100.0%	0.62
219.00	352,131	65.6%	0.60
502.00	30,337	5.7%	0.62

Temperatures and Pressures			
MS Source	250	Turbo Speed	100.0
MS Quad	200	Hi Vac	7.57e-06

Low	High	Step	Speed	Threshold	Peaks	Base	Abundance	Total Ion
10.00	701.00	0.10	3	100	134	69.00	523,072	1,695,935

Target m/z	Actual m/z	Abund	Rel Abund	Iso m/z	Iso Abund	Iso Ratio
69.00	69.00	523,072	100.0%	70.00	5,739	1.1%
219.00	219.00	344,320	65.8%	220.00	15,288	4.4%
502.00	502.00	30,224	5.8%	503.00	3,085	10.2%

Air/Water Check: H20 ~2.0% N2 ~0.3% O2 ~0.1% CO2 ~0.4% N2/H20 ~17.0%

Column(1) Flow: 0.66 Column(2): 0.00 ml/min Interface Temp: 250

Ramp Criteria:

Ion Focus maximum 90 volts using ion 502; Electron Multiplier Gain 28889.809

Repeller maximum 35 volts using ion 219; Gain Factor 0.2889

Mass Gain Values(Scan Speed): 244(3) 252(2) 262(1) 282(0) 335(FS1) 334(FS2)

TARGET MASS:	50	69	131	219	414	502	1050
Amu Offset	133.9	133.9	133.9	133.9	133.9	133.9	133.9
Entrance Lens Offset	11.2	11.2	11.2	11.2	11.2	11.2	11.2

Report Created: 1/23/2019 4:41 PM (UTC-05:00)

Figure 2. BFB Autotune report.

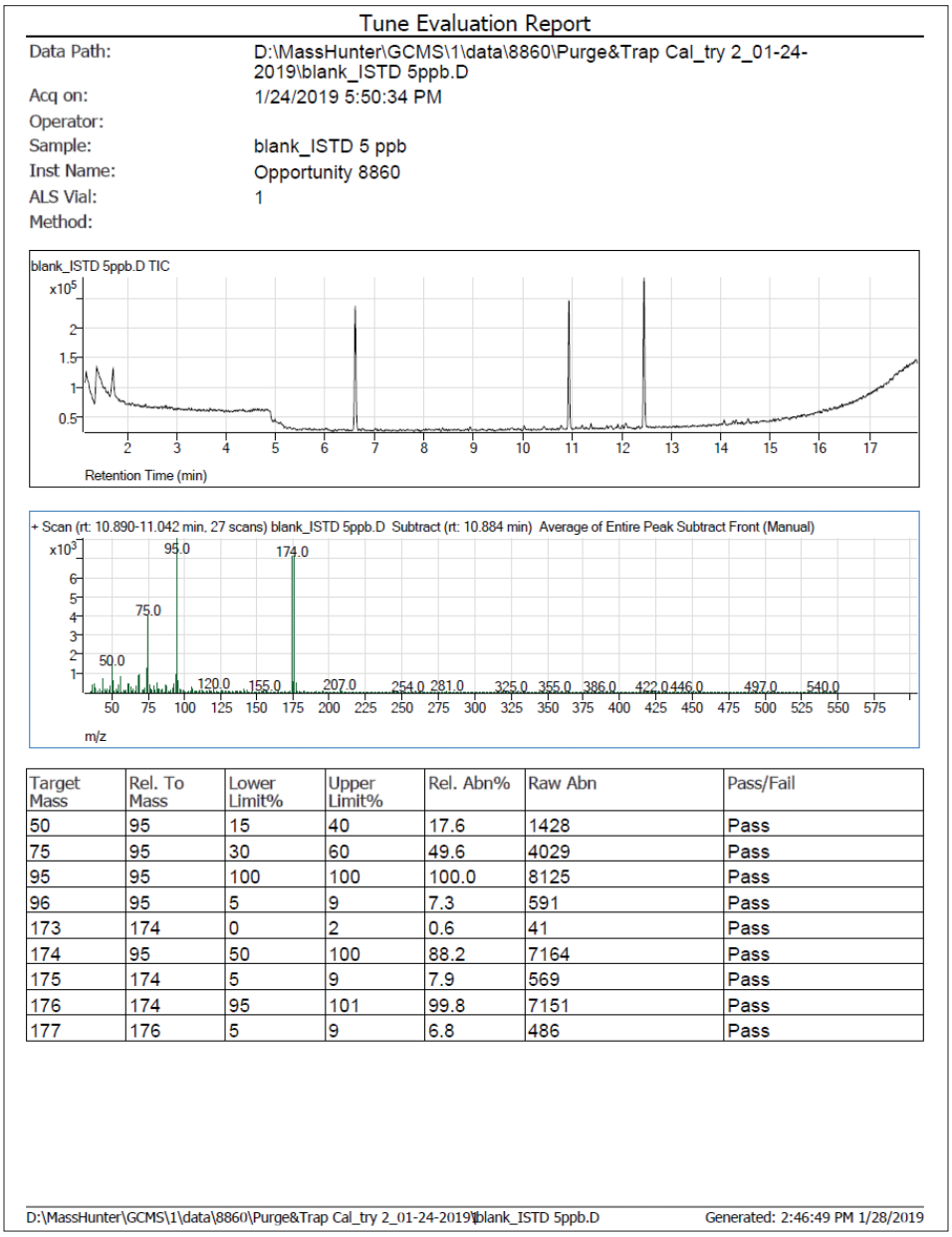
Evaluating BFB spectra

Method 524.2 requires 25 ng or less on-column of BFB to be introduced by either purging or direct injection (sections 6.3.1 and 10.2.2), and tested against the criteria provided by the EPA 524.2 method. The Tune Evaluation tool is provided in the Agilent MassHunter Quantitative Analysis Package, and by-default, includes the method for BFB spectra evaluation. Figure 3 shows the Tune Evaluation report. Tuning routines, including BFB Autotune and tune evaluation, can also be invoked in a sequence by keyword requests allowing for a system performance check.

Results and discussion

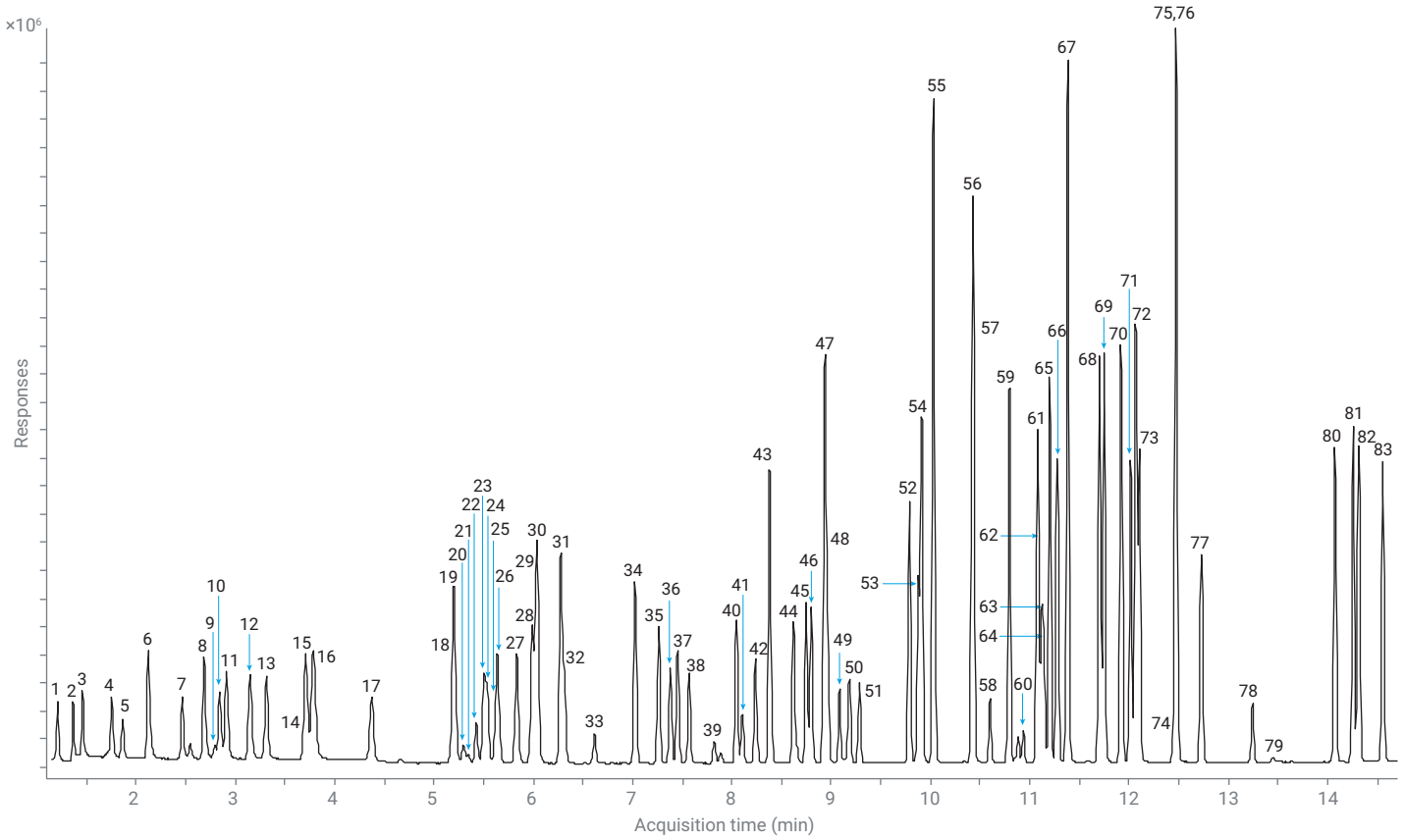
Initial calibration (ICAL)

After a successful BFB injection and spectral evaluation, an ICAL was performed over the specified working range, which is typically from 0.5 to 50 µg/L. Figure 4 shows a typical total ion chromatogram (TIC) acquired using the GC, MSD, and P&T parameters specified in this analysis.



Target Mass	Rel. To Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Pass/Fail
50	95	15	40	17.6	1428	Pass
75	95	30	60	49.6	4029	Pass
95	95	100	100	100.0	8125	Pass
96	95	5	9	7.3	591	Pass
173	174	0	2	0.6	41	Pass
174	95	50	100	88.2	7164	Pass
175	174	5	9	7.9	569	Pass
176	174	95	101	99.8	7151	Pass
177	176	5	9	6.8	486	Pass

Figure 3. BFB Tune check report.



1	Dichlorodifluoromethane	22	Methyl Acrylate	43	Toluene	64	1,4-Dichlorobut-2-ene
2	Chloromethane	23	Bromochloromethane	44	*trans*-1,3-Dichloropropene	65	Propylbenzene
3	Chloroethene	24	Methylacrylonitrile	45	Ethyl Methacrylate	66	2-Chlorotoluene
4	Bromomethane	25	Tetrahydrofuran	46	1,1,2-Trichloroethane	67	Mesitylene (1,3,5-Trimethylbenzene)
5	Ethyl Chloride	26	Trichloromethane	47	Tetrachloroethylene	68	*tert*-Butylbenzene
6	Trichloromonofluoromethane	27	1,1,1-Trichloroethane	48	1,3-Dichloropropane	69	1,2,4-Trimethylbenzene
7	Ethyl Ether	28	1-Chlorobutane	49	2-Hexanone	70	1-Methylpropyl Benzene
8	1,1-Dichloroethene	29	Carbon Tetrachloride	50	Dibromochloromethane	71	1,3-Dichlorobenzene
9	Acetone	30	1,1-Dichloropropene	51	1,2-Dibromoethane	72	*p*-Cymene (4-Isopropyltoluene)
10	Iodomethane	31	Benzene	52	Chlorobenzene	73	1,4-Dichlorobenzene
11	Carbon Disulfide	32	1,2-Dichloroethane	53	1,1,1,2-Tetrachloroethane	74	1,2-Dichlorobenzene-d_4 (SURR)
12	Allyl Chloride	33	Fluorobenzene (ISTD)	54	Ethylbenzene	75	1,2-Dichlorobenzene
13	Methylene Chloride	34	Trichloroethylene	55	*m+p*-Xylene	76	*n*-Butylbenzene
14	Acrylonitrile	35	1,2-Dichloropropane	56	*o*-Xylene	77	Hexachloroethane
15	*trans*-1,2-Dichloroethylene	36	Dibromomethane	57	Styrene	78	1,2-Dibromo-3-chloropropane
16	Methyl *tert*-butyl Ether	37	Methyl Methacrylate	58	Tribromomethane	79	Nitrobenzene
17	1,1-Dichloroethane	38	Bromodichloromethane	59	Isopropylbenzene	80	1,2,4-Trichlorobenzene
18	2,2-Dichloropropane	39	2-Nitropropane	60	*p*-Bromofluorobenzene (SURR)	81	1,1,2,3,4,4-Hexachlorobuta-1,3-diene
19	*cis*-1,2-Dichloroethylene	40	*cis*-1,3-Dichloropropene	61	Bromobenzene	82	Naphthalene
20	2-Butanone	41	2,2-Dimethoxybutane	62	1,1,2,2-Tetrachloroethane	83	1,2,3-Trichlorobenzene
21	Propanenitrile	42	MIBK	63	1,2,3-Trichloropropane		

Figure 4. TIC of EPA method 524.2: 50 µg/L standard, ISTD, and surrogates (5 µg/L).

Table 4 presents typical ICAL results over a concentration range of 0.25 to 50 µg/L. Method 524.2 specifies that all compounds must have %RSD values less than 20 % to apply quantitation by average relative response factor. Otherwise, compounds must use the calibration curve fit routines of linear or quadratic curves.

The results in Table 4 demonstrate that the calibration for 79 compounds met the criteria of less than 20 % RSD. Iodomethane, which often exhibits problems with linearity due to its known stability issues, had an RSD over 20 %, and thus, a quadratic fit was applied as recommended in the method. The %RSDs for the internal standard and surrogate compounds introduced by the AQUATek LVA are <5 % RSD.

These results demonstrate the high degree of precision achievable with the Agilent GC/MSD and the Teledyne Tekmar P&T instrumentation.

Typical reporting limits for most compounds here are 0.25 or 0.50 µg/L. However, there are some compounds that have higher reporting limits. For example, the ketones, such as acetone and 2-butanone, have reporting limits at 5.0 µg/L.

Table 4. ICAL for Method 524.2 From 0.25 to 50 µg/L.

Compound	RT (min)	0.25 µg/L RRF	0.5 µg/L RRF	1 µg/L RRF	5 µg/L RRF	10 µg/L RRF	25 µg/L RRF	50 µg/L RRF	Avg RRF	%RSD
Fluorobenzene (ISTD)	6.613	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	1.000	0.6
Dichlorodifluoromethane	1.207	0.087	0.150	0.178	0.136	0.157	0.153	0.153	0.145	19.6
Chloromethane	1.362	0.148	0.178	0.143	0.159	0.170	0.162	0.161	0.160	7.5
Chloroethene	1.462	0.244	0.182	0.212	0.180	0.191	0.183	0.184	0.196	12.0
Bromomethane	1.752				0.149	0.145	0.119	0.117	0.132	12.8
Ethyl Chloride	1.858	0.066	0.118	0.104	0.099	0.115	0.113	0.112	0.104	17.2
Trichloromonofluoromethane	2.120	0.299	0.353	0.342	0.337	0.334	0.337	0.336	0.334	5.0
Ethyl Ether	2.466	0.062	0.096	0.115	0.099	0.105	0.105	0.106	0.098	17.4
1,1-Dichloroethene	2.683	0.223	0.280	0.274	0.262	0.252	0.255	0.250	0.257	7.3
Acetone	2.793				0.021	0.022	0.020	0.020	0.021	5.1
Iodomethane	2.841	0.186	0.160	0.177	0.171	0.209	0.275	0.297	0.211	0.9963*
Carbon Disulfide	2.907	0.603	0.515	0.516	0.477	0.489	0.483	0.489	0.510	8.5
Allyl Chloride	3.145	0.074	0.089	0.071	0.091	0.099	0.089	0.089	0.086	11.8
Methylene Chloride	3.310	0.225	0.182	0.178	0.179	0.174	0.171	0.171	0.183	10.5
Acrylonitrile	3.696		0.071	0.094	0.060	0.061	0.067	0.064	0.070	18.2
trans-1,2-Dichloroethylene	3.709	0.245	0.252	0.251	0.239	0.228	0.231	0.228	0.239	4.3
Methyl *tert*-butyl Ether	3.787	0.658	0.564	0.657	0.575	0.564	0.567	0.566	0.593	7.5
1,1-Dichloroethane	4.370	0.253	0.269	0.296	0.303	0.307	0.306	0.294	0.290	7.1
2,2-Dichloropropane	5.192	0.236	0.282	0.258	0.257	0.278	0.244	0.239	0.256	7.2
cis-1,2-Dichloroethylene	5.202	0.371	0.298	0.314	0.287	0.287	0.284	0.279	0.303	10.6
2-Butanone	5.296				0.026	0.026	0.023	0.023	0.024	7.8
Propanenitrile	5.346		0.023	0.027	0.030	0.031	0.030	0.032	0.029	11.6
Methyl Acrylate	5.424	0.128	0.190	0.170	0.187	0.174	0.182	0.182	0.173	12.3
Bromochloromethane	5.503	0.126	0.070	0.125	0.123	0.130	0.128	0.128	0.118	18.2
Methylacrylonitrile	5.535		0.108	0.104	0.094	0.088	0.092	0.089	0.096	8.8
Tetrahydrofuran	5.615		0.035	0.032	0.036	0.029	0.031	0.032	0.033	7.6
Trichloromethane	5.640	0.232	0.293	0.297	0.321	0.322	0.327	0.320	0.302	11.0
1,1,1-Trichloroethane	5.831	0.364	0.271	0.277	0.287	0.280	0.292	0.292	0.295	10.7
1-Chlorobutane	5.985	0.294	0.360	0.284	0.340	0.354	0.345	0.348	0.332	9.1
Carbon Tetrachloride	6.029	0.209	0.200	0.208	0.226	0.231	0.217	0.232	0.218	5.7

* Compound was second order regressed.

Table 4. ICAL for Method 524.2 From 0.25 to 50 µg/L (continued).

Compound	RT (min)	0.25 µg/L RRF	0.5 µg/L RRF	1 µg/L RRF	5 µg/L RRF	10 µg/L RRF	25 µg/L RRF	50 µg/L RRF	Avg RRF	%RSD
1,1-Dichloropropene	6.038	0.293	0.255	0.265	0.260	0.252	0.259	0.255	0.263	5.3
Benzene	6.274	0.857	0.695	0.749	0.710	0.717	0.707	0.700	0.734	7.8
1,2-Dichloroethane	6.308	0.304	0.253	0.266	0.263	0.258	0.263	0.263	0.267	6.2
Trichloroethylene	7.023	0.299	0.156	0.221	0.233	0.224	0.227	0.230	0.227	18.4
1,2-Dichloropropane	7.261	0.245	0.189	0.196	0.185	0.179	0.183	0.183	0.194	11.8
Dibromomethane	7.379	0.118	0.098	0.117	0.135	0.141	0.142	0.144	0.128	13.6
Methyl Methacrylate	7.453		0.063	0.073	0.056	0.058	0.061	0.060	0.062	9.8
Bromodichloromethane	7.568	0.249	0.209	0.192	0.197	0.210	0.205	0.215	0.211	8.7
2-Nitropropane	7.820			0.021	0.026	0.029	0.025	0.027	0.026	11.9
cis-1,3-Dichloropropene	8.044	0.308	0.331	0.290	0.281	0.275	0.284	0.284	0.293	6.7
2,2-Dimethoxybutane	8.105			0.078	0.079	0.050	0.068	0.066	0.068	17.0
MIBK	8.237	0.111	0.097	0.083	0.083	0.081	0.086	0.086	0.090	12.1
Toluene	8.380	0.903	0.821	0.809	0.828	0.801	0.811	0.806	0.826	4.3
trans-1,3-Dichloropropene	8.619	0.303	0.237	0.259	0.239	0.257	0.265	0.262	0.260	8.5
Ethyl Methacrylate	8.748	0.286	0.288	0.293	0.282	0.273	0.284	0.287	0.285	2.2
1,1,2-Trichloroethane	8.797	0.159	0.153	0.174	0.169	0.172	0.177	0.175	0.168	5.4
Tetrachloroethylene	8.933	0.359	0.342	0.273	0.333	0.267	0.302	0.336	0.316	11.3
1,3-Dichloropropane	8.961	0.382	0.292	0.329	0.312	0.307	0.307	0.303	0.319	9.4
2-Hexanone	9.082	0.077	0.078	0.080	0.087	0.086	0.086	0.090	0.084	6.0
Dibromochloromethane	9.181	0.147	0.140	0.143	0.140	0.146	0.148	0.154	0.146	3.5
1,2-Dibromoethane	9.284	0.187	0.162	0.179	0.172	0.172	0.176	0.182	0.176	4.6
Chlorobenzene	9.787	0.558	0.520	0.556	0.526	0.519	0.529	0.531	0.534	3.0
1,1,1,2-Tetrachloroethane	9.875	0.160	0.163	0.143	0.147	0.157	0.158	0.166	0.156	5.3
Ethylbenzene	9.909	0.985	0.950	0.950	0.929	0.915	0.925	0.918	0.939	2.6
m+p-Xylene	10.028	0.851	0.748	0.730	0.710	0.702	0.712	0.715	0.738	7.1
o-Xylene	10.418	0.885	0.706	0.733	0.720	0.718	0.727	0.740	0.747	8.3
Styrene	10.431	0.640	0.631	0.542	0.610	0.581	0.597	0.610	0.601	5.5
Tribromomethane	10.600	0.076	0.078	0.097	0.093	0.099	0.101	0.110	0.094	13.3
Isopropylbenzene	10.791	0.961	0.989	0.954	0.910	0.914	0.919	0.944	0.942	3.1
p-Bromofluorobenzene (SURR)	10.933	0.326	0.342	0.343	0.324	0.351	0.330	0.359	0.339	3.9
Bromobenzene	11.074	0.304	0.260	0.232	0.243	0.241	0.246	0.248	0.254	9.4
1,1,2,2-Tetrachloroethane	11.083	0.178	0.207	0.241	0.217	0.234	0.236	0.232	0.221	10.1
1,2,3-Trichloropropane	11.121	0.230	0.254	0.252	0.258	0.285	0.278	0.306	0.266	9.5
1,4-Dichlorobut-2-ene	11.142		0.027	0.032	0.030	0.039	0.041	0.043	0.035	18.3
Propylbenzene	11.200	1.207	1.059	1.096	1.078	1.097	1.094	1.110	1.106	4.3
2-Chlorotoluene	11.274	0.695	0.596	0.594	0.609	0.606	0.618	0.637	0.622	5.7
Mesitylene (1,3,5-Trimethylbenzene)	11.379	0.825	0.774	0.789	0.777	0.772	0.790	0.827	0.793	2.9
tert-Butylbenzene	11.701	0.978	0.807	0.871	0.707	0.762	0.718	0.738	0.797	12.3
1,2,4-Trimethylbenzene	11.748	0.896	0.869	0.845	0.786	0.805	0.810	0.852	0.838	4.7
1-Methylpropyl Benzene	11.919	1.202	1.070	1.041	1.037	1.043	1.049	1.090	1.076	5.4
1,3-Dichlorobenzene	12.014	0.529	0.470	0.464	0.454	0.464	0.464	0.480	0.475	5.3
p-Cymene (4-Isopropyltoluene)	12.067	0.910	0.876	0.959	0.883	0.906	0.905	0.961	0.914	3.7

Table 4. ICAL for Method 524.2 From 0.25 to 50 µg/L (continued).

Compound	RT (min)	0.25 µg/L RRF	0.5 µg/L RRF	1 µg/L RRF	5 µg/L RRF	10 µg/L RRF	25 µg/L RRF	50 µg/L RRF	Avg RRF	%RSD
1,4-Dichlorobenzene	12.102	0.503	0.465	0.507	0.462	0.465	0.476	0.503	0.483	4.2
1,2-Dichlorobenzene-d_4 (SURR)	12.452	0.380	0.394	0.384	0.373	0.390	0.395	0.418	0.391	3.7
1,2-Dichlorobenzene	12.470	0.512	0.441	0.446	0.433	0.434	0.440	0.472	0.454	6.3
n-Butylbenzene	12.473	0.991	0.874	0.917	0.831	0.864	0.868	0.909	0.893	5.8
Hexachloroethane	12.727	0.133	0.107	0.100	0.111	0.111	0.119	0.121	0.115	9.4
1,2-Dibromo-3-chloropropane	13.241	0.030	0.050	0.049	0.045	0.051	0.054	0.061	0.048	20.0
Nitrobenzene	13.446				0.008	0.009	0.009	0.009	0.008	7.7
1,2,4-Trichlorobenzene	14.072	0.488	0.382	0.345	0.346	0.359	0.359	0.377	0.380	13.1
1,1,2,3,4,4-Hexachlorobuta-1,3-diene	14.256	0.318	0.261	0.234	0.236	0.233	0.240	0.251	0.253	11.9
Naphthalene	14.311	1.023	0.862	0.836	0.876	0.865	0.908	0.963	0.905	7.3
1,2,3-Trichlorobenzene	14.554	0.401	0.385	0.348	0.327	0.349	0.349	0.369	0.361	7.0

MDLs

An MDL study was performed after demonstrating acceptable linearity of greater than two orders of magnitude for the target analytes using an average relative response curve fit (all %RSD are <20 %). Eight trials were performed at the lowest level of calibration, 0.25 µg/L. The calculated MDLs were obtained by applying the formula shown in Equation 1. For the compounds with higher reporting limits, eight trials were performed at the concentration of 1 µg/L. Table 5 lists the calculated MDLs for 80 VOCs, which are typical using the conditions of this analysis.

Equation 1. Formula for MDL calculations.

$$MDL = s \times t_{(n-1,\ 1-alpha\ =\ 99)} = s \times 2.998$$

Where:

$t_{(n-1,\ 1-alpha)}$ = t value for the 99 % confidence level with n – 1 degrees of freedom

n = Number of trials (8)

s = Standard deviation of the eight trials

Table 5. Calculated MDLs for VOCs.

Compound	RT (min)	Calculated Concentration in the Sample (µg/L)									Average concentration (µg/L)	SD	MDL
		Spike (µg/L)	Samp. 1	Samp. 2	Samp. 3	Samp. 4	Samp. 5	Samp. 6	Samp. 7	Samp. 8			
Fluorobenzene (ISTD)	6.613	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	N/A	N/A
Dichlorodifluoromethane	1.207	0.25	0.23	0.24	0.24	0.24	0.26	0.22	0.24	0.23	0.24	0.010	0.031
Chloromethane	1.362	0.25	0.26	0.26	0.25	0.24	0.23	0.29	0.25	0.26	0.26	0.017	0.052
Chloroethene	1.462	0.25	0.17	0.17	0.14	0.14	0.18	0.15	0.16	0.19	0.16	0.017	0.051
Bromomethane	1.752	1.00	0.88	1.03	0.90	0.91	0.78	0.74	0.75	0.97	0.87	0.106	0.316
Ethyl Chloride	1.858	0.25	0.36	0.34	0.29	0.35	0.29	0.38	0.37	0.37	0.34	0.035	0.103
Trichloromonofluoromethane	2.120	0.25	0.25	0.24	0.18	0.23	0.18	0.26	0.20	0.21	0.22	0.030	0.091
Ethyl Ether	2.466	0.25	0.28	0.21	0.29	0.27	0.33	0.23	0.27	0.30	0.27	0.038	0.114
1,1-Dichloroethene	2.683	0.25	0.24	0.24	0.27	0.23	0.24	0.21	0.24	0.25	0.24	0.019	0.057
Acetone	2.793	1.00	1.04	1.21	1.51	1.33	1.37	1.25	1.03	1.08	1.23	0.173	0.518

Table 5. Calculated MDLs for VOCs (continued).

Compound	RT (min)	Calculated Concentration in the Sample (µg/L)									Average concentration (µg/L)	SD	MDL
		Spike (µg/L)	Samp. 1	Samp. 2	Samp. 3	Samp. 4	Samp. 5	Samp. 6	Samp. 7	Samp. 8			
Iodomethane	2.841	0.25	0.23	0.17	0.16	0.17	0.19	0.24	0.20	0.21	0.20	0.031	0.092
Carbon Disulfide	2.907	0.25	0.21	0.23	0.23	0.21	0.22	0.20	0.22	0.21	0.21	0.011	0.033
Allyl Chloride	3.145	0.25	0.26	0.30	0.24	0.31	0.40	0.36	0.31	0.27	0.31	0.052	0.156
Methylene Chloride	3.310	0.25	0.25	0.22	0.22	0.29	0.30	0.23	0.24	0.27	0.25	0.029	0.088
Acrylonitrile	3.696	1.00	0.94	0.86	0.90	0.87	0.91	1.19	0.92	0.91	0.94	0.105	0.315
trans-1,2-Dichloroethylene	3.709	0.25	0.19	0.19	0.22	0.24	0.20	0.21	0.20	0.22	0.21	0.018	0.054
Methyl *tert*-butyl Ether	3.787	0.25	0.29	0.28	0.24	0.27	0.28	0.26	0.27	0.29	0.27	0.015	0.046
1,1-Dichloroethane	4.370	0.25	0.26	0.32	0.32	0.28	0.27	0.29	0.30	0.29	0.29	0.021	0.063
2,2-Dichloropropane	5.192	0.25	0.27	0.27	0.25	0.23	0.26	0.28	0.27	0.27	0.26	0.017	0.050
cis-1,2-Dichloroethylene	5.202	0.25	0.20	0.22	0.24	0.24	0.29	0.22	0.21	0.22	0.23	0.027	0.080
2-Butanone	5.296	1.00	1.02	0.87	1.07	1.13	1.50	1.41	1.44	1.39	1.23	0.234	0.701
Propanenitrile	5.346	1.00	1.18	1.21	1.04	1.21	1.03	1.21	0.83	1.24	1.12	0.143	0.428
Methyl Acrylate	5.424	0.25	0.30	0.29	0.29	0.31	0.31	0.32	0.29	0.28	0.30	0.014	0.042
Bromochloromethane	5.503	0.25	0.33	0.21	0.28	0.34	0.17	0.24	0.32	0.21	0.26	0.064	0.192
Methylacrylonitrile	5.535	0.25	0.24	0.27	0.23	0.22	0.26	0.29	0.26	0.16	0.24	0.038	0.114
Tetrahydrofuran	5.615	1.00	1.01	0.99	1.18	0.77	1.09	1.09	1.23	1.22	1.07	0.152	0.455
Trichloromethane	5.640	0.25	0.30	0.28	0.25	0.27	0.25	0.30	0.31	0.26	0.28	0.024	0.072
1,1,1-Trichloroethane	5.831	0.25	0.22	0.21	0.20	0.27	0.24	0.23	0.26	0.22	0.23	0.023	0.068
1-Chlorobutane	5.985	0.25	0.31	0.31	0.34	0.34	0.28	0.32	0.28	0.29	0.31	0.025	0.074
Carbon Tetrachloride	6.029	0.25	0.26	0.27	0.26	0.25	0.27	0.29	0.29	0.22	0.26	0.023	0.070
1,1-Dichloropropene	6.038	0.25	0.18	0.19	0.22	0.22	0.22	0.23	0.23	0.23	0.21	0.021	0.062
Benzene	6.274	0.25	0.19	0.21	0.20	0.19	0.23	0.24	0.22	0.20	0.21	0.020	0.061
1,2-Dichloroethane	6.308	0.25	0.28	0.26	0.23	0.26	0.29	0.26	0.27	0.22	0.26	0.024	0.073
Trichloroethylene	7.023	0.25	0.22	0.25	0.25	0.31	0.27	0.21	0.19	0.19	0.24	0.040	0.120
1,2-Dichloropropane	7.261	0.25	0.20	0.20	0.22	0.22	0.23	0.24	0.24	0.19	0.22	0.021	0.062
Dibromomethane	7.379	0.25	0.35	0.25	0.22	0.21	0.24	0.27	0.31	0.32	0.27	0.051	0.152
Methyl Methacrylate	7.453	0.25	0.30	0.37	0.20	0.34	0.33	0.19	0.26	0.34	0.29	0.066	0.197
Bromodichloromethane	7.568	0.25	0.26	0.26	0.23	0.26	0.26	0.21	0.25	0.19	0.24	0.028	0.083
2-Nitropropane	7.820	1.00	1.14	1.53	1.23	1.27	1.38	1.35	1.64	1.68	1.40	0.196	0.587
cis-1,3-Dichloropropene	8.044	0.25	0.24	0.23	0.23	0.28	0.22	0.23	0.21	0.25	0.23	0.021	0.062
2,2-Dimethoxybutane	8.105	1.00	1.06	1.03	0.94	0.99	0.99	1.13	1.08	1.11	1.04	0.067	0.202
MIBK	8.237	0.25	0.23	0.27	0.25	0.29	0.27	0.26	0.25	0.24	0.26	0.017	0.051
Toluene	8.380	0.25	0.24	0.26	0.23	0.23	0.22	0.23	0.26	0.20	0.23	0.020	0.060
trans-1,3-Dichloropropene	8.619	0.25	0.28	0.24	0.23	0.22	0.28	0.28	0.25	0.22	0.25	0.027	0.081
Ethyl Methacrylate	8.748	0.25	0.29	0.31	0.24	0.24	0.25	0.23	0.24	0.21	0.25	0.031	0.093
1,1,2-Trichloroethane	8.797	0.25	0.28	0.28	0.24	0.25	0.27	0.30	0.31	0.29	0.28	0.023	0.069
Tetrachloroethylene	8.933	0.25	0.17	0.18	0.20	0.19	0.21	0.19	0.21	0.24	0.20	0.019	0.058
1,3-Dichloropropane	8.961	0.25	0.24	0.22	0.20	0.23	0.26	0.25	0.20	0.19	0.22	0.027	0.081
2-Hexanone	9.082	0.25	0.26	0.30	0.29	0.34	0.36	0.31	0.28	0.28	0.30	0.032	0.097
Dibromochloromethane	9.181	0.25	0.24	0.31	0.31	0.26	0.26	0.25	0.30	0.32	0.28	0.031	0.092
1,2-Dibromoethane	9.284	0.25	0.26	0.25	0.30	0.29	0.27	0.22	0.25	0.27	0.26	0.025	0.074
Chlorobenzene	9.787	0.25	0.25	0.23	0.26	0.26	0.26	0.26	0.24	0.21	0.25	0.018	0.055

Table 5. Calculated MDLs for VOCs (continued).

Compound	RT (min)	Calculated Concentration in the Sample (µg/L)									Average concentration (µg/L)	SD	MDL
		Spike (µg/L)	Samp. 1	Samp. 2	Samp. 3	Samp. 4	Samp. 5	Samp. 6	Samp. 7	Samp. 8			
1,1,1,2-Tetrachloroethane	9.875	0.25	0.38	0.30	0.41	0.39	0.37	0.28	0.31	0.32	0.35	0.046	0.138
Ethylbenzene	9.909	0.25	0.25	0.22	0.22	0.25	0.22	0.24	0.25	0.22	0.23	0.015	0.044
m+p-Xylene	10.028	0.25	0.45	0.50	0.48	0.46	0.49	0.49	0.40	0.45	0.46	0.033	0.100
o-Xylene	10.418	0.50	0.26	0.22	0.23	0.21	0.23	0.25	0.26	0.21	0.23	0.020	0.061
Styrene	10.431	0.25	0.22	0.24	0.24	0.24	0.26	0.29	0.25	0.25	0.25	0.019	0.056
Tribromomethane	10.600	0.25	0.38	0.41	0.29	0.35	0.40	0.28	0.35	0.38	0.36	0.049	0.146
Isopropylbenzene	10.791	0.25	0.24	0.28	0.24	0.25	0.27	0.28	0.26	0.23	0.25	0.020	0.059
p-Bromofluorobenzene (SURR)	10.933	5.00	4.95	5.07	4.98	5.03	4.94	4.69	4.91	4.85	4.93	0.117	N/A
Bromobenzene	11.074	0.25	0.22	0.24	0.19	0.18	0.19	0.27	0.22	0.21	0.21	0.032	0.096
1,1,2,2-Tetrachloroethane	11.083	0.25	0.30	0.31	0.27	0.27	0.34	0.30	0.25	0.30	0.29	0.029	0.086
1,2,3-Trichloropropane	11.121	0.25	0.29	0.31	0.33	0.36	0.36	0.33	0.30	0.27	0.32	0.033	0.099
1,4-Dichlorobut-2-ene	11.142	1.00	1.25	1.21	1.10	1.21	1.11	1.03	1.27	0.85	1.13	0.139	0.415
Propylbenzene	11.200	0.25	0.24	0.24	0.24	0.23	0.25	0.23	0.26	0.24	0.24	0.011	0.033
2-Chlorotoluene	11.274	0.25	0.25	0.28	0.26	0.23	0.23	0.28	0.25	0.24	0.25	0.020	0.061
Mesitylene (1,3,5-Trimethylbenzene)	11.379	0.25	0.27	0.26	0.25	0.27	0.23	0.27	0.24	0.24	0.25	0.015	0.044
tert-Butylbenzene	11.701	0.25	0.23	0.20	0.22	0.18	0.22	0.21	0.19	0.20	0.20	0.015	0.045
1,2,4-Trimethylbenzene	11.748	0.25	0.23	0.26	0.25	0.24	0.25	0.25	0.25	0.22	0.24	0.013	0.040
1-Methylpropyl Benzene	11.919	0.25	0.21	0.26	0.23	0.26	0.24	0.27	0.21	0.22	0.24	0.023	0.070
1,3-Dichlorobenzene	12.014	0.25	0.24	0.21	0.29	0.24	0.22	0.28	0.25	0.21	0.24	0.028	0.085
p-Cymene (4-Isopropyltoluene)	12.067	0.25	0.28	0.27	0.29	0.28	0.28	0.28	0.28	0.24	0.28	0.015	0.044
1,4-Dichlorobenzene	12.102	0.25	0.25	0.26	0.26	0.23	0.26	0.30	0.31	0.25	0.26	0.025	0.076
1,2-Dichlorobenzene-d_4 (SURR)	12.452	5.00	4.95	4.91	5.10	4.97	4.93	4.85	4.92	4.82	4.93	0.085	N/A
1,2-Dichlorobenzene	12.470	0.25	0.25	0.26	0.29	0.26	0.27	0.26	0.24	0.23	0.26	0.019	0.057
n-Butylbenzene	12.473	0.25	0.25	0.27	0.22	0.26	0.24	0.26	0.26	0.22	0.25	0.019	0.058
Hexachloroethane	12.727	0.25	0.28	0.27	0.33	0.24	0.29	0.29	0.24	0.26	0.27	0.033	0.098
1,2-Dibromo-3-chloropropane	13.241	0.25	0.33	0.43	0.47	0.39	0.37	0.40	0.39	0.39	0.40	0.042	0.126
Nitrobenzene	13.446	1.00	1.36	1.60	1.65	1.19	1.95	1.65	1.79	1.95	1.64	0.267	0.800
1,2,4-Trichlorobenzene	14.072	0.25	0.15	0.19	0.23	0.20	0.21	0.23	0.21	0.19	0.20	0.027	0.081
1,1,2,3,4,4-Hexachlorobuta-1,3-diene	14.256	0.25	0.19	0.23	0.20	0.25	0.22	0.13	0.21	0.21	0.21	0.035	0.104
Naphthalene	14.311	0.25	0.27	0.28	0.26	0.31	0.28	0.28	0.24	0.25	0.27	0.022	0.065
1,2,3-Trichlorobenzene	14.554	0.25	0.27	0.21	0.24	0.30	0.25	0.26	0.23	0.21	0.25	0.033	0.098

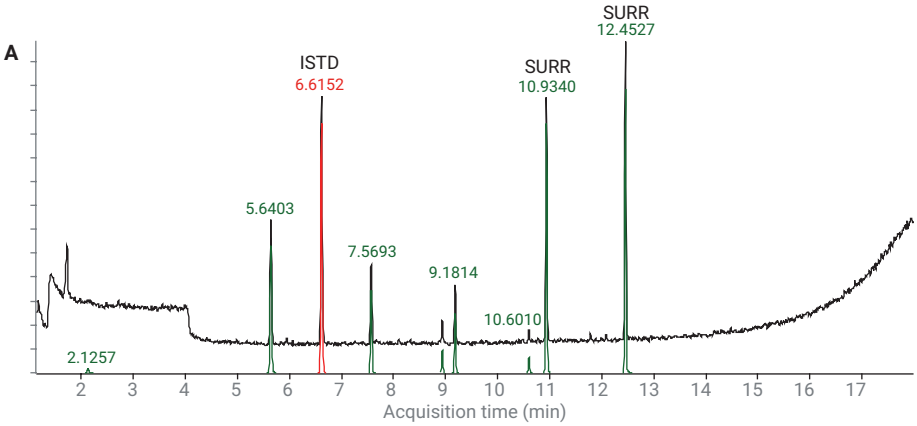
VOCs Found in drinking water

To test the method's applicability to the real-world samples, finished drinking (from tap) water from several sources in Pennsylvania was subjected to the analysis. Several VOCs were identified in tap water with MassHunter Unknowns Analysis using a retention time-locked VOC spectral library (Figure 5). The concentration of VOCs was determined using MassHunter Quantitative Analysis. Table 6 presents the results.

Figure 6 shows the information displayed in Unknowns Analysis when analyzing a tap water sample with a VOC library and inspecting the hits, in this case, trichloromonofluoromethane, tetrachloroethylene, and tribromomethane. The left figure overlays the component profile with the EICs of the ions the software has identified as being part of the spectrum. The overlay is inspected to see if the EICs have similar shape and RT, as they do here. The spectrum on the lower right of each Figure 6 section is the average of the raw spectra over the component profile of the peak. Its purpose is to show the degree of interfering ions from coeluting compounds. In the upper right is the deconvoluted spectrum of the found component compared to the library reference spectrum. The deconvolution process has removed the interfering ions, producing an LMS of 71.8 even when the compound is present only at three-times the MDL level.

Table 6. VOCs Determined in tap water.

Compound	RT (min)	Concentration (µg/L)			
		Southern Pennsylvania	Eastern Pennsylvania	Southeastern Pennsylavania	City of Philadelphia
Trichloromonofluoromethane	2.120		0.30		
Trichloromethane (Chloroform)	5.640	1.05	7.15	12.56	14.06
Bromodichloromethane	7.568		5.15	4.81	5.77
Toluene	8.380	0.29			
Tetrachloroethylene	8.933		0.36		
Dibromochloromethane	9.181		4.49	1.03	1.44
Tribromomethane	10.600		1.26		



B

Components

Component RT	Compound Name	Match Factor	Best Hit	Formula	Library RT	Delta RT
2.1257	Trichloromonofluoromethane	71.8	☑	CCl3F	2.1205	-0.0052
5.6403	Trichloromethane	95.2	☑	CHCl3	5.6396	-0.0007
6.6152	Benzene, fluoro-	97.5	☑	C6H5F	6.6141	-0.0011
7.5693	Methane, bromodichloro-	95.3	☑	CHBrCl2	7.5681	-0.0012
8.9355	Tetrachloroethylene	91.9	☑	C2Cl4	8.9325	-0.0029
9.1814	Methane, dibromochloro-	94.3	☑	CHBr2Cl	9.1810	-0.0004
10.6010	Methane, tribromo-	75.5	☑	CHBr3	10.5996	-0.0014
10.9340	p-Bromofluorobenzene	98.3	☑	C6H4BrF	10.9330	-0.0010
12.4527	1,2-Dichlorobenzene-D4	87.0	☑	C6D4Cl2	12.4523	-0.0004

Figure 5. VOCs found in a tap water sample from eastern Pennsylvania.

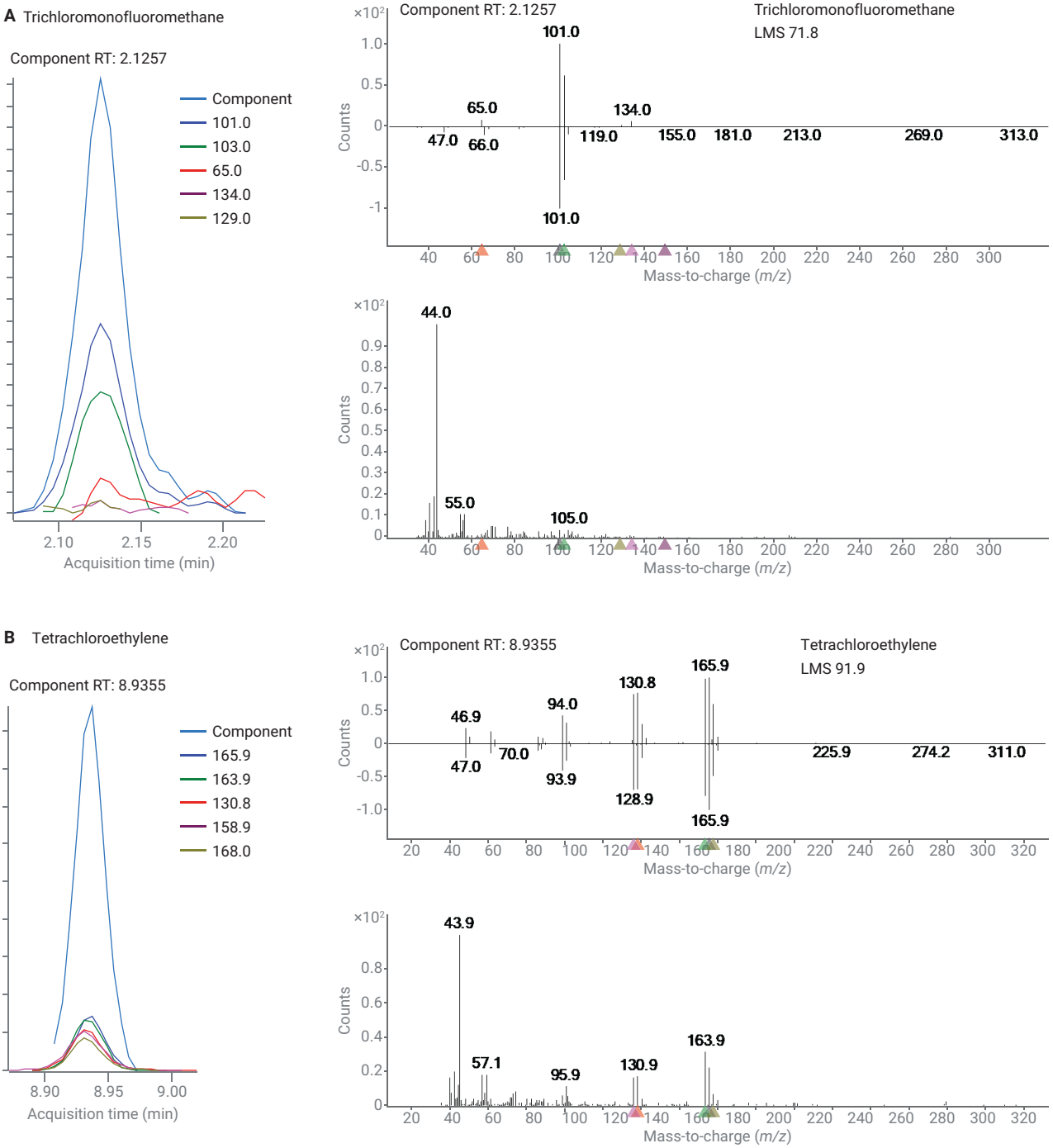


Figure 6. Identification of: A) Trichloromonofluoromethane at 3x MDL level; B) Tetrachloroethylene; C) Tribromomethane in a water sample from Eastern Pennsylvania with MassHunter Unknowns Analysis (continued next page).

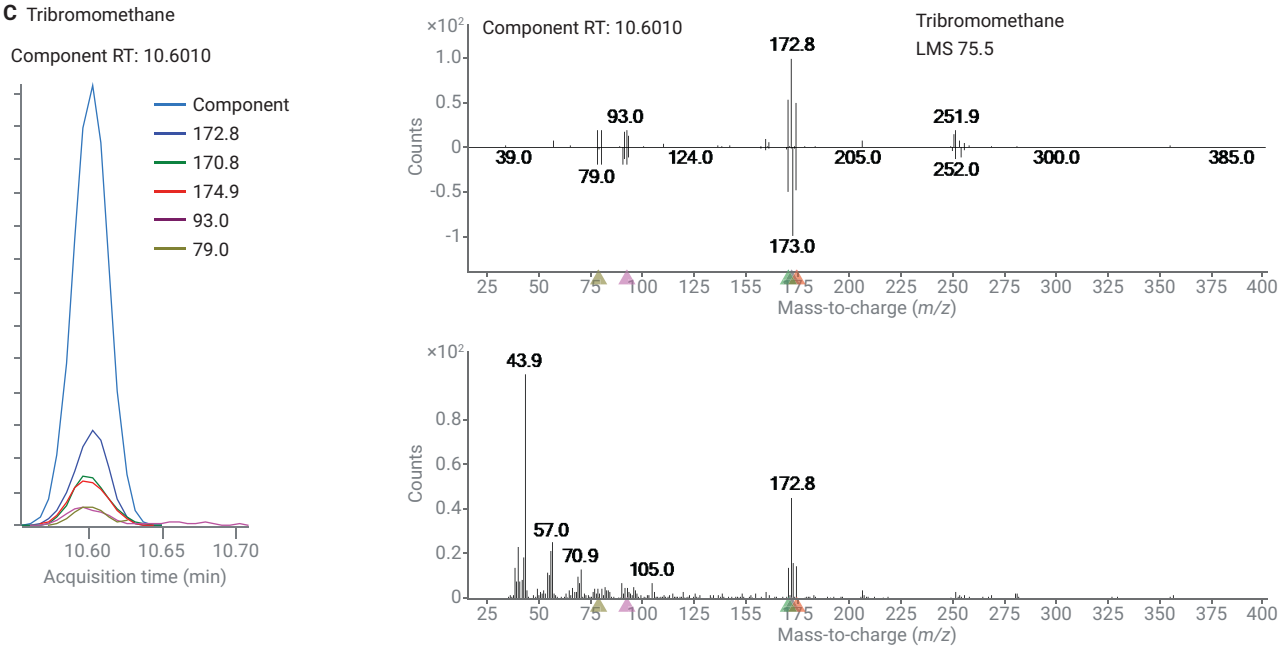


Figure 6. Identification of: A) Trichloromonofluoromethane at 3x MDL level; B) Tetrachloroethylene; C) Tribromomethane in a water sample from eastern Pennsylvania with MassHunter Unknowns Analysis (continued).

Conclusions

The 8860/5977B GC/MSD system coupled with a Teledyne Tekmar Lumin P&T concentrator, and an AQUATek LVA have been demonstrated to be suitable for analyzing VOCs with an ICAL range of 0.25 to 50 µg/L. BFB Autotune provides high sensitivity and stability, while maintaining the required ion abundance ratios in the BFB spectrum. Calculated MDLs were in ppt levels, which is typical using the conditions of this analysis. The applicability of the method was demonstrated using real-world drinking water samples, in which several VOCs were identified and quantified at concentrations varying over the range of 0.3 to 14.1 µg/L, in many cases much lower than current EPA 524.2 MDLs.

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2. Hollis, J. S.; Prest, H. Volatile Organic Compound Analysis Using Purge and Trap. Success with VOC analysis using the Agilent 5975C Mass Selective Detector. *Agilent Technologies Application Note*, publication number 5991-0029EN.

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