US EPA Method 524.2: Software Tools for Successful Method Development and Analysis of Purgeable Organic Compounds in Drinking Water

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Agilent
Trusted Answers

Volatile Organic Compound (VOC) Testing

EPA method 8260	Surface and ground water as well as solid sample matrices
EPA method 624	Wastewater samples The procedures are very similar to 8260, however, the reported compound lists are somewhat different
EPA method 524.2	Drinking water The conditions used in this method enables a lower detection limit to be reached. This provides results that meet drinking water regulatory limits or MCLs

Purge and Trap vs. Headspace Analysis for VOCs

EPA 524.2 requires the use of purge and trap

METHOD 524.2

MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY

1. SCOPE AND APPLICATION

1.1 This is a general purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in surface water, ground water, and drinking water in any stage of treatment (1,2). The method is applicable to a wide range of organic compounds, including the four trihalomethane disinfection by-products, that have sufficiently high volatility and low water solubility to be removed from water samples with purge and trap procedures. The following compounds can be determined by this method.

6.2 PURGE AND TRAP SYSTEM - The purge and trap system consists of three separate pieces of equipment: purging device, trap, and desorber. Systems are commercially available from several sources that meet all of the following specifications.

US EPA Method 524.2 VOCs in Water

EPA method 524.2 provides procedures and requirements for the quantitation of VOCs in surface water, ground water, and drinking water by GC/MS

Four specific groups of VOCs

- Trihalomethanes (THMs)
- Gases
- Aromatics
- Products of dehydrohalogenation

Dichlorodifluoromethane	Methyl acrylate	Toluene	1,4-Dichlorobut-2-ene
Chloromethane	Bromochloromethane	trans-1,3-Dichloropropene	Propylbenzene
Chloroethene	Methylacrylonitrile	Ethyl methacrylate	2-Chlorotoluene
Bromomethane	Tetrahydrofuran	1,1,2-Trichloroethane	Mesitylene (1,3,5-Trimethylbenzene)
Ethyl chloride	Trichloromethane	Tetrachloroethylene	tert-Butylbenzene
Trichloromonofluoromethane	1,1,1-Trichloroethane	1,3-Dichloropropane	1,2,4-Trimethylbenzene
Ethyl ether	1-Chlorobutane	2-Hexanone	1-Methylpropyl benzene
1,1-Dichloroethene	Carbon Tetrachloride	Dibromochloromethane	1,3-Dichlorobenzene
Acetone	1,1-Dichloropropene	1,2-Dibromoethane	p-Cymene (4-Isopropyltoluene)
Iodomethane	Benzene	Chlorobenzene	1,4-Dichlorobenzene
Carbon disulfide	1,2-Dichloroethane	1,1,1,2-Tetrachloroethane	***1,2-Dichlorobenzene-d4 (SURR)***
Allyl chloride	***Fluorobenzene (ISTD)***	Ethylbenzene	1,2-Dichlorobenzene
Methylene chloride	Trichloroethylene	m+p-Xylene	n-Butylbenzene
Acrylonitrile	1,2-Dichloropropane	o-Xylene	Hexachloroethane
trans-1,2-Dichloroethylene	Dibromomethane	Styrene	1,2-Dibromo-3-chloropropane
Methyl tert-butyl ether	Methyl methacrylate	Tribromomethane	Nitrobenzene
1,1-Dichloroethane	Bromodichloromethane	Isopropylbenzene	1,2,4-Trichlorobenzene
2,2-Dichloropropane	2-Nitropropane	***p-Bromofluorobenzene (SURR)***	1,1,2,3,4,4-Hexachlorobuta-1,3-diene
cis-1,2-Dichloroethylene	cis-1,3-Dichloropropene	Bromobenzene	Naphthalene
2-Butanone	2,2-Dimethoxybutane	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
Propanenitrile	Methyl Isobutyl Ketone (MIBK)	1,2,3-Trichloropropane	

80 target compounds

Parameters for GC, MSD, and P&T Necessary for Success

Hardware:

- GC/MS system coupled with a purge and trap (P&T) concentrator, and a liquid vial autosampler

MS Tuning:

- BFB Autotune

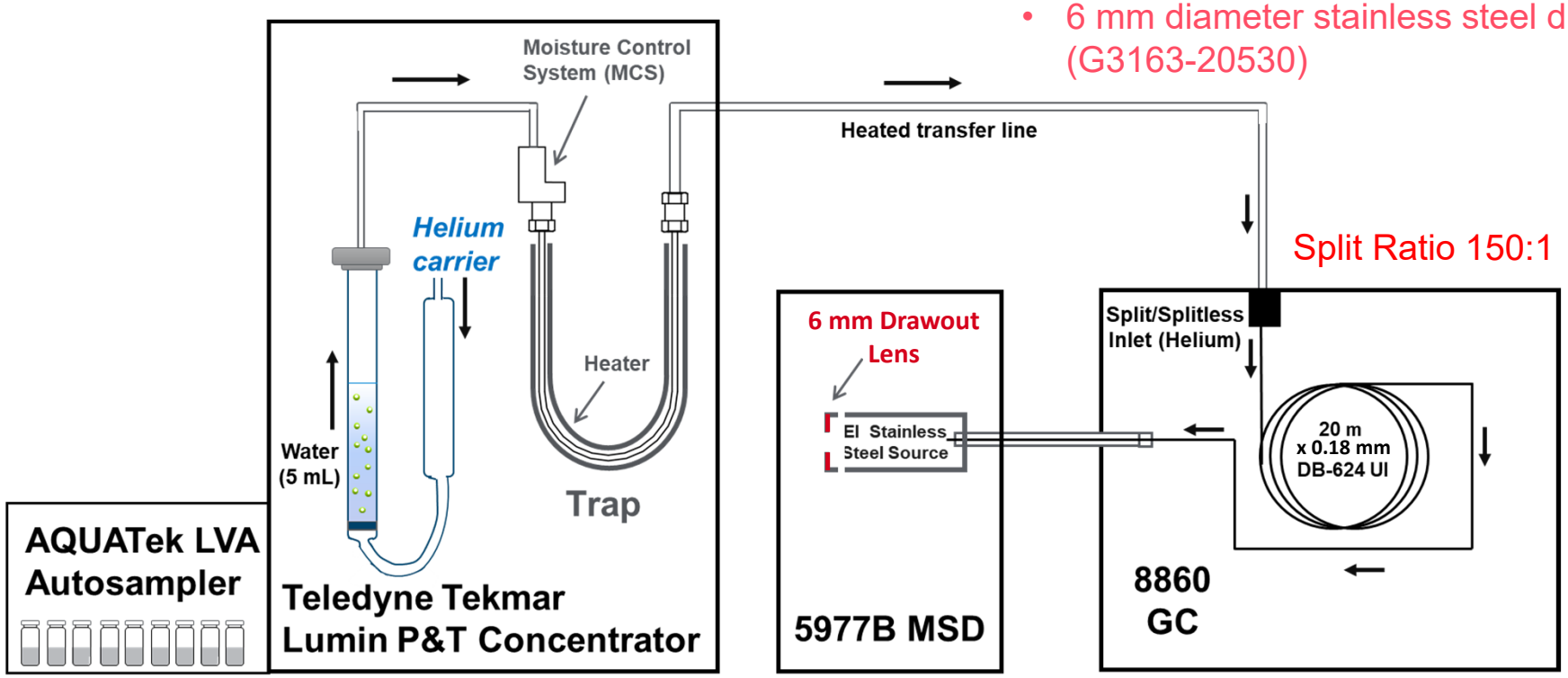
Software tools to streamline:

- Quantitation method development
- Data processing
- Reporting

US EPA Method 524.2 Instrument Configuration

Consumables:

- 1.0 mm inlet liner (5190-4047)
- DB-624UI, 20m x 0.18 mm x 1µm column (121-1324UI)
- 6 mm diameter stainless steel drawout lens (G3163-20530)



Tune Evaluation: Meeting BFB Tune Check Criteria

EPA 524.2 requires that the spectrometer must produce a mass spectrum that meets all criteria in Table 3 when 25 ng or less of 4-bromofluorobenzene (BFB) is introduced into the GC.

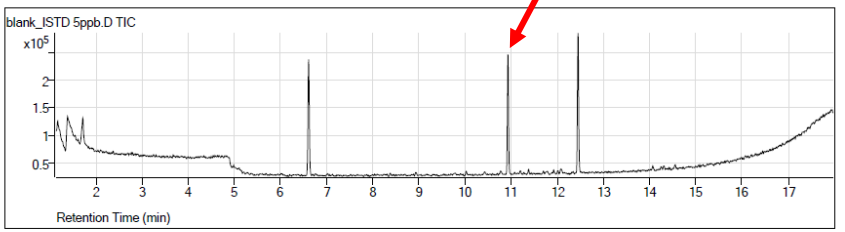
TABLE 3. ION ABUNDANCE CRITERIA FOR 4-BROMOFLUOROBENZENE (BFB)

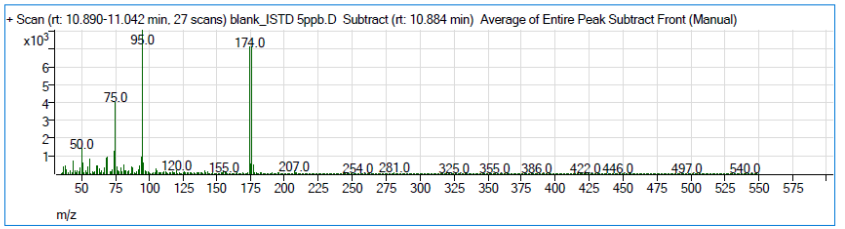
Mass (M/z)	Relative Abundance Criteria
50	15 to 40% of mass 95
75	30 to 80% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

Tune Evaluation Report

Data Path: D:\MassHunter\GCMS\1\data\8860\Purge&Trap Cal_try 2_01-24-2019\blank_ISTD 5ppb.D
Acq on: 1/24/2019 5:50:34 PM
Operator:
Sample: blank_ISTD 5 ppb
Inst Name: Opportunity 8860
ALS Vial: 1
Method:

BFB

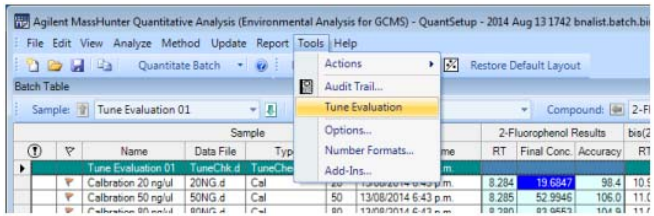




Target Mass	Rel. To Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Pass/Fail
50	95	15	40	17.6	1428	Pass
75	95	30	60	49.6	4029	Pass
95	95	100	100	100.0	8125	Pass
96	95	5	9	7.3	591	Pass
173	174	0	2	0.6	41	Pass
174	95	50	100	88.2	7164	Pass
175	174	5	9	7.9	569	Pass
176	174	95	101	99.8	7151	Pass
177	176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

Tune Evaluation: Meeting BFB Tune Check Criteria

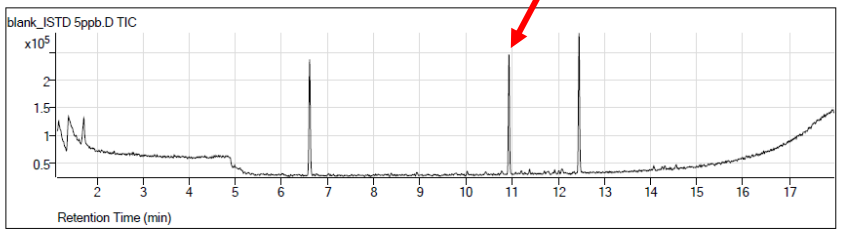
MassHunter Quantitative Analysis in Environmental Quant Mode (EnviroQuant)

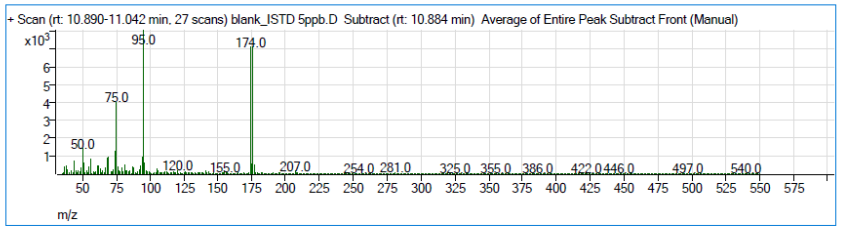


Tune Evaluation Report

Data Path:	D:\MassHunter\GCMS\1\data\8860\Purge&Trap Cal_try 2_01-24-2019\blank_ISTD 5ppb.D
Acq on:	1/24/2019 5:50:34 PM
Operator:	
Sample:	blank_ISTD 5 ppb
Inst Name:	Opportunity 8860
ALS Vial:	1
Method:	

BFB





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For more information: Agilent application note 5991-0029EN

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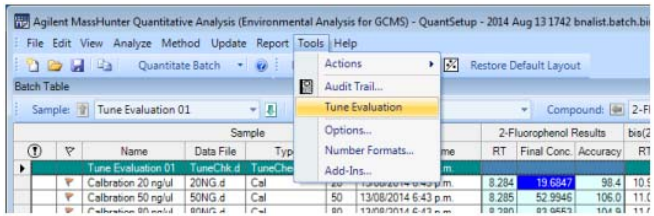

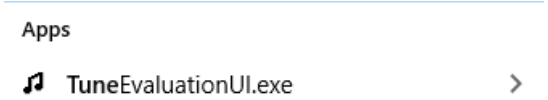

A stand-alone Tune Evaluation app (installed automatically with MassHunter Quantitative Analysis)

For more information: Agilent application note 5991-0029EN

Tune Evaluation Report

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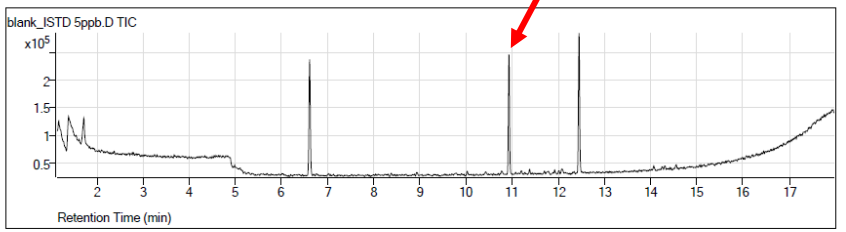

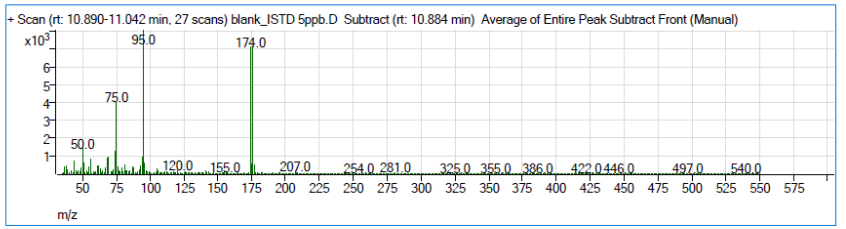

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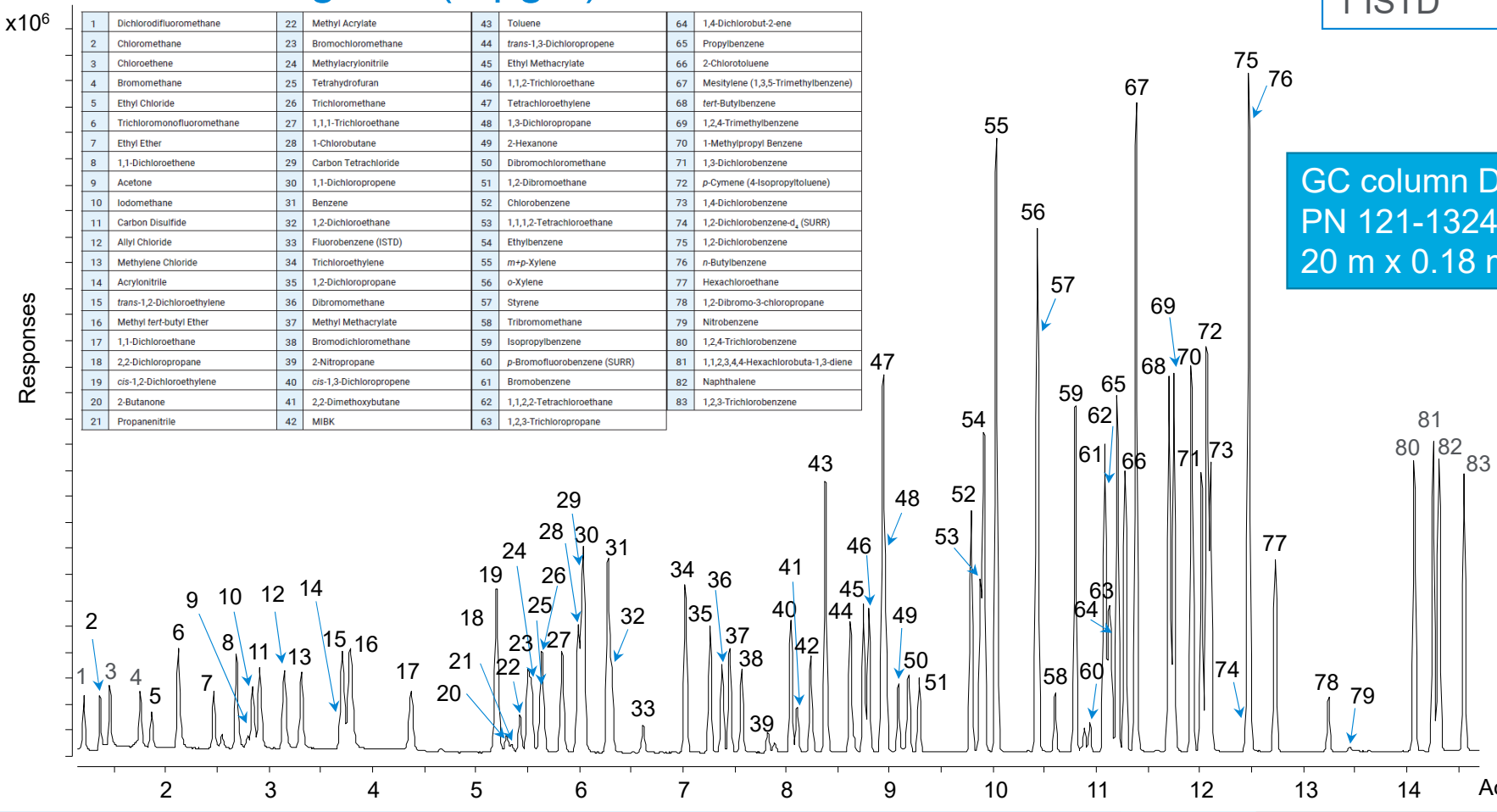
Total Ion Chromatogram of Method 524.2 50 µg/L Standard, ISTD and Surrogates (5 µg/L)

80 target compounds
2 surrogates
1 ISTD

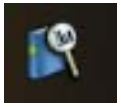
GC column DB-624 UI
PN 121-1324UI
20 m x 0.18 mm x 1 µm



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		

Setting up EPA 524.2 Analysis with MassHunter

1. A VOC standard was analyzed and the deconvoluted spectra were searched against the NIST spectral library for compound identification using MassHunter Unknowns Analysis



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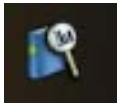

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2. A user library was created using MassHunter Library Editor. It included the identified compounds with their retention times and deconvoluted spectra

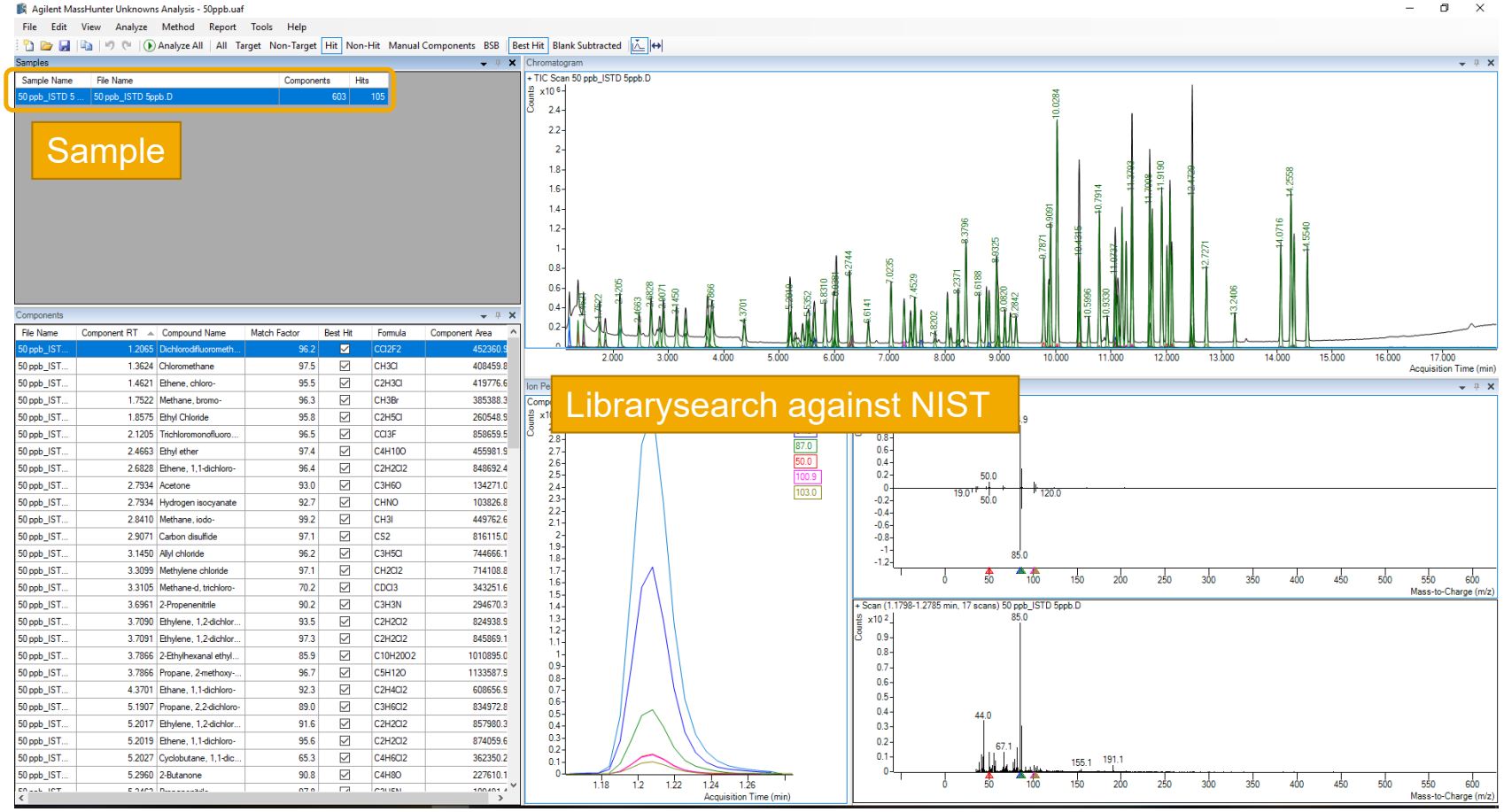
3. A quantitation method was created from the user spectral library using the compound information and retention times included in the library using MassHunter Quantitative Analysis

4. Real-world samples were analyzed with the quantitation method. Spectral deconvolution allowed for confirmation of compound identifications.

   The concentration of the VOCs in the real-world sample was determined against the initial calibration

Step 1: Analyzing a Standard

Analyze a calibration standard with Unknowns Analysis against the NIST library



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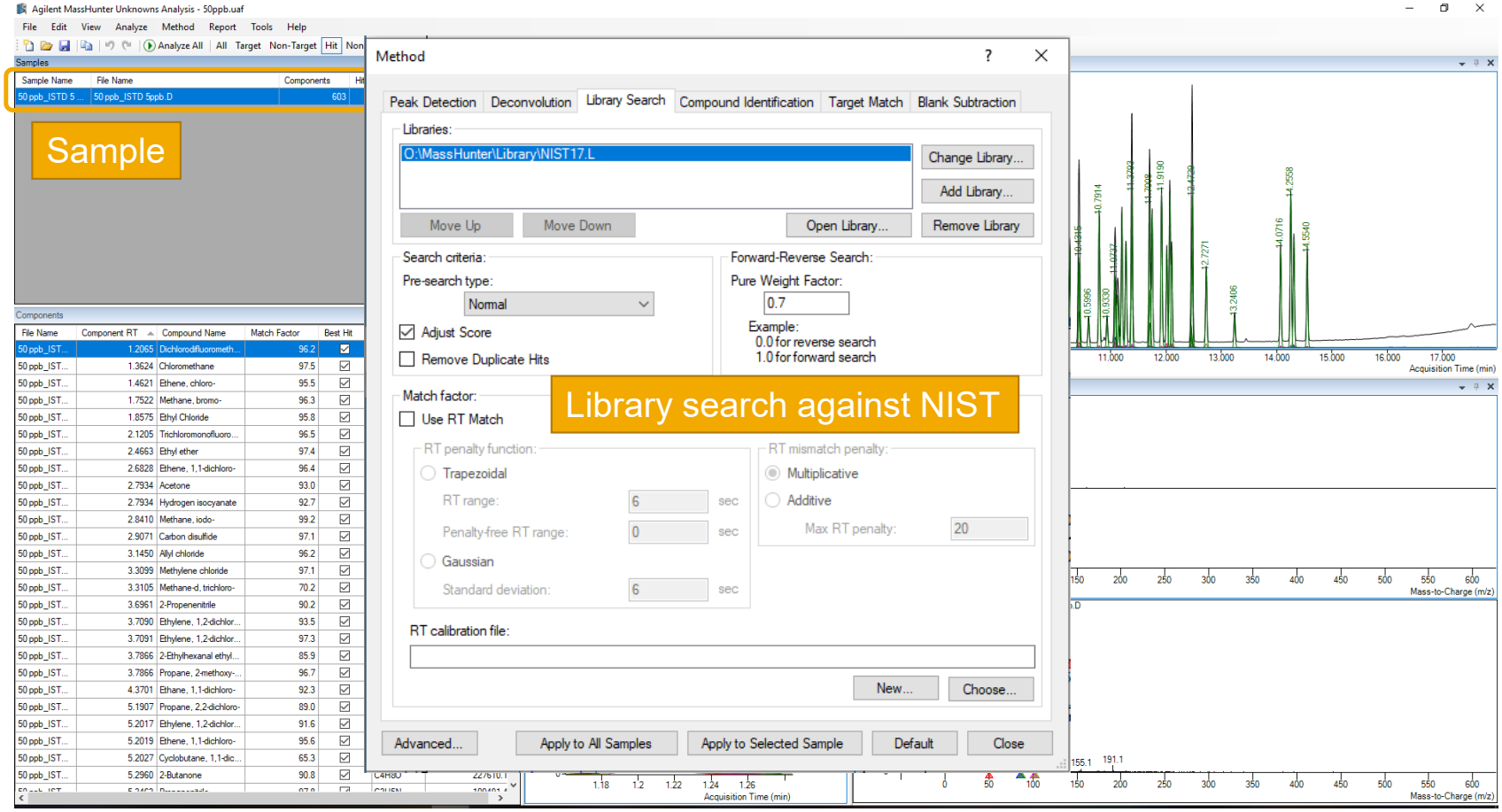
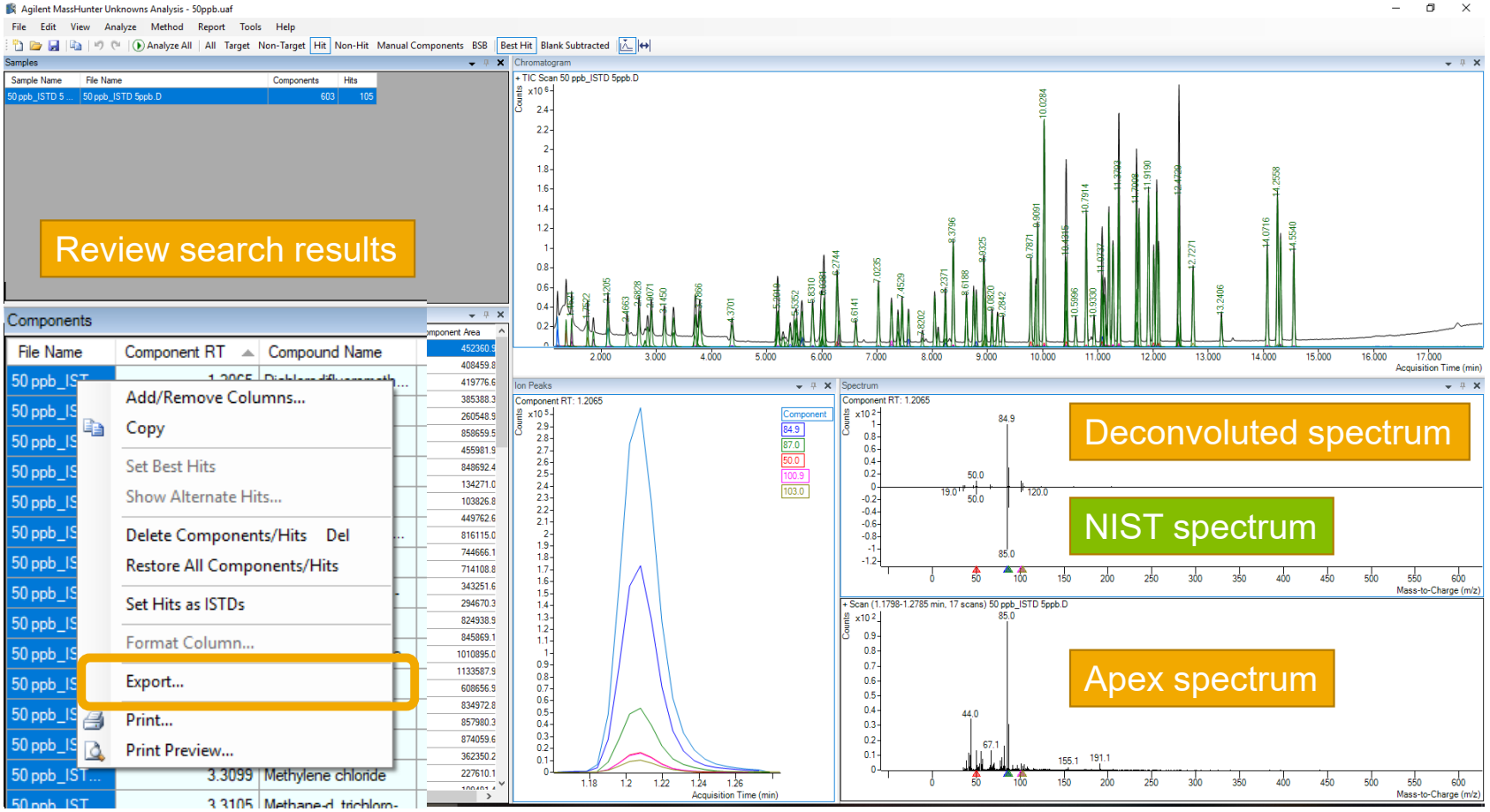


Step 2: Creating a Spectral Library

Analyze a calibration standard with Unknowns Analysis against the NIST library



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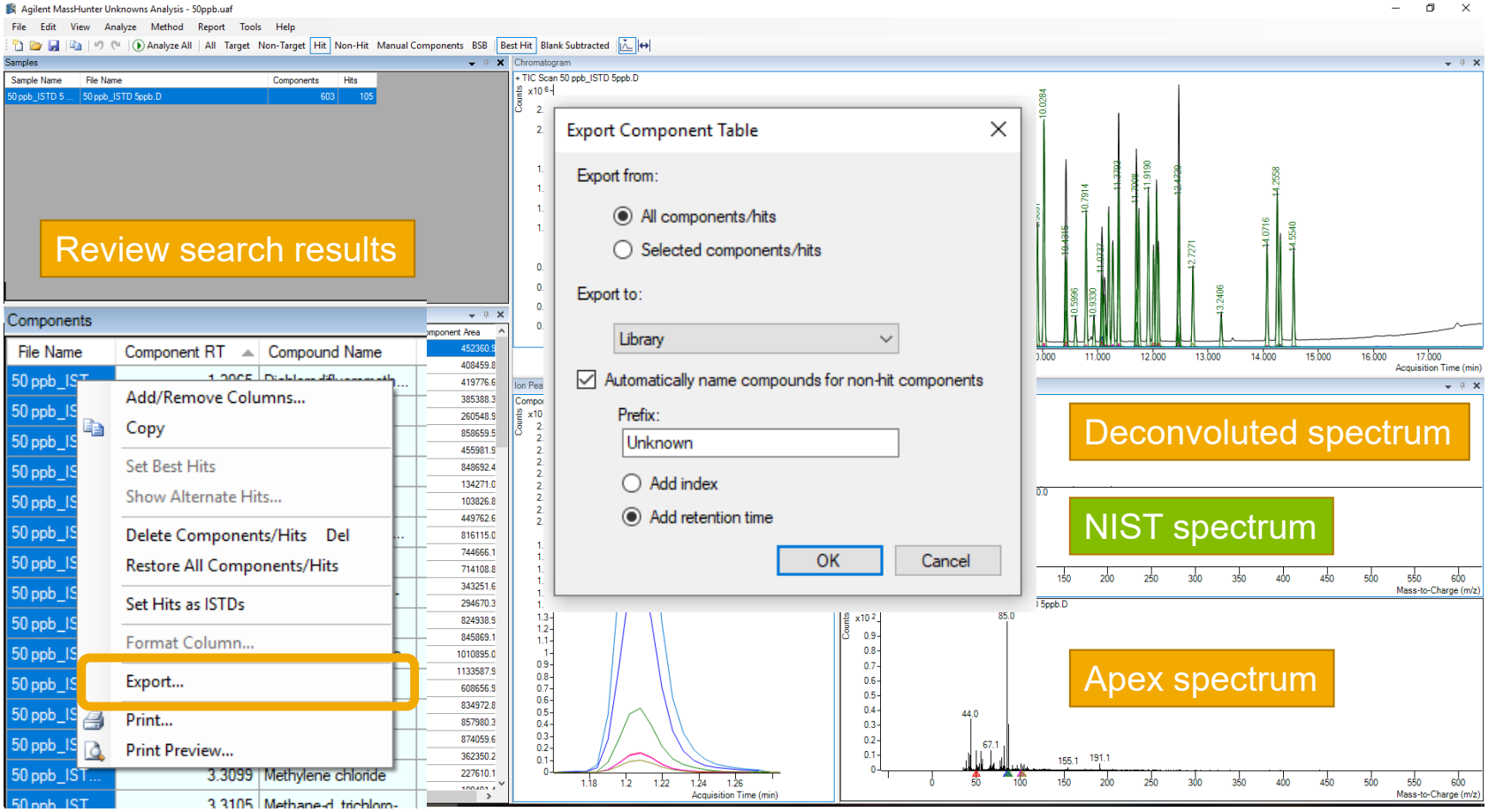


Step 2: Creating a Spectral Library

Export deconvoluted spectra to the Library Editor and review

Library Editor - ref_VOCs libr.reflibrary.xml

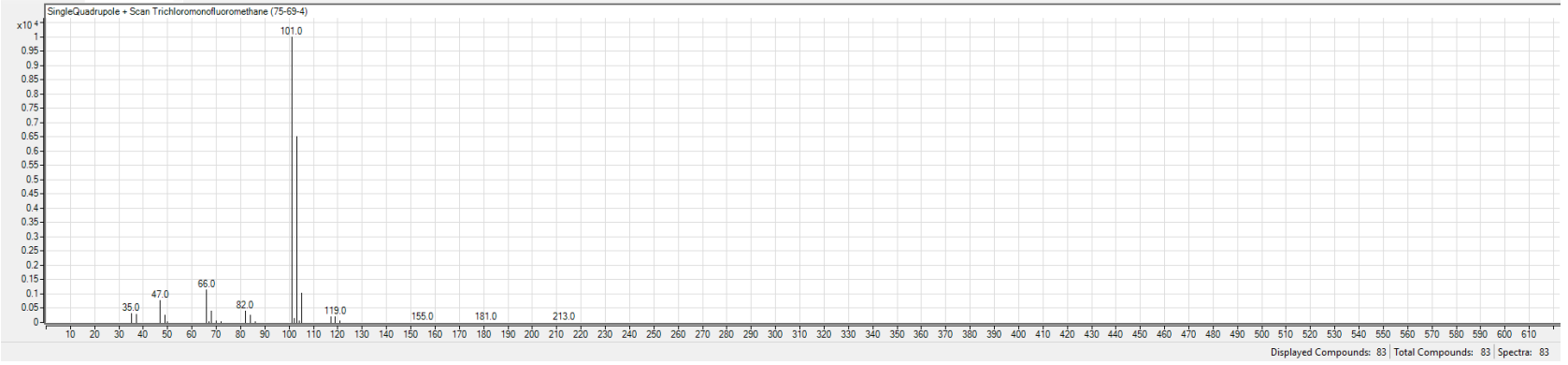
File Edit Tools Help

Compound Table

Compound ID	Compound Name	CAS#	Formula	Molecular Weight	Retention Time	Retention Index
3	Methane, bromo-	74-83-9	CH3Br	93.942	1.7522	
4	Ethyl Chloride	75-00-3	C2H5Cl	64.008	1.8575	445.0000
5	Trichloromonofluoromethane	75-69-4	CCl3F	135.905	2.1205	
6	Ethyl ether	60-29-7	C4H10O	74.073	2.4663	495.0000
7	Ethene, 1,1-dichloro-	75-35-4	C2H2Cl2	95.953	2.6828	540.0000
8	Acetone	67-64-1	C3H6O	58.042	2.7934	455.0000
9	Methane, iodo-	74-88-4	CH3I	141.928	2.8410	
10	Carbon disulfide	75-15-0	CS2	75.944	2.9071	
11	Allyl chloride	107-05-1	C3H5Cl	76.008	3.1450	535.0000
12	Methylene chloride	75-09-2	CH2Cl2	83.953	3.3099	
13	Acrylonitrile	107-13-1	C3H3N	53.027	3.6961	555.0000
14	Ethylene, 1,2-dichloro-, trans-	156-60-5	C2H2Cl2	95.953	3.7091	580.0000
15	Methyl tert-butyl ether	1634-04-4	C5H12O	88.089	3.7866	510.0000
16	Ethane, 1,1-dichloro-	75-34-3	C2H4Cl2	97.969	4.3699	566.0000
17	Propane, 2,2-dichloro-	594-20-7	C3H6Cl2	111.985	5.1918	550.0000
18	Ethylene, 1,2-dichloro-, cis-	156-59-2	C2H2Cl2	95.953	5.2017	580.0000
19	2-Butanone	78-93-3	C4H8O	72.058	5.2960	555.0000
20	Propanenitrile	107-12-0	C3H5N	55.042	5.3463	565.0000
21	2-Propenoic acid, methyl ester	96-33-3	C4H6O2	86.037	5.4245	576.0000
22	Methane, bromochloro-	74-97-5	CH2BrCl	127.903	5.5029	

Spectrum View

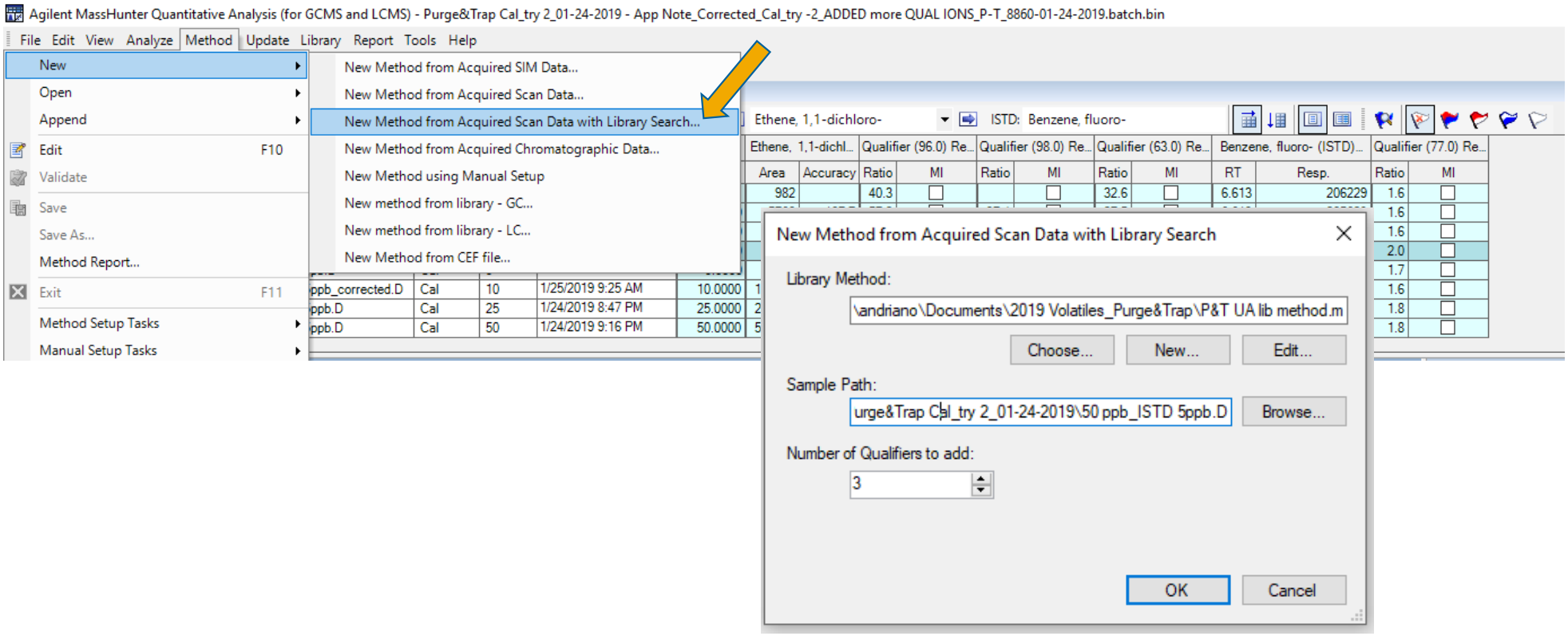
Max. # of panes: 2



Displayed Compounds: 83 | Total Compounds: 83 | Spectra: 83

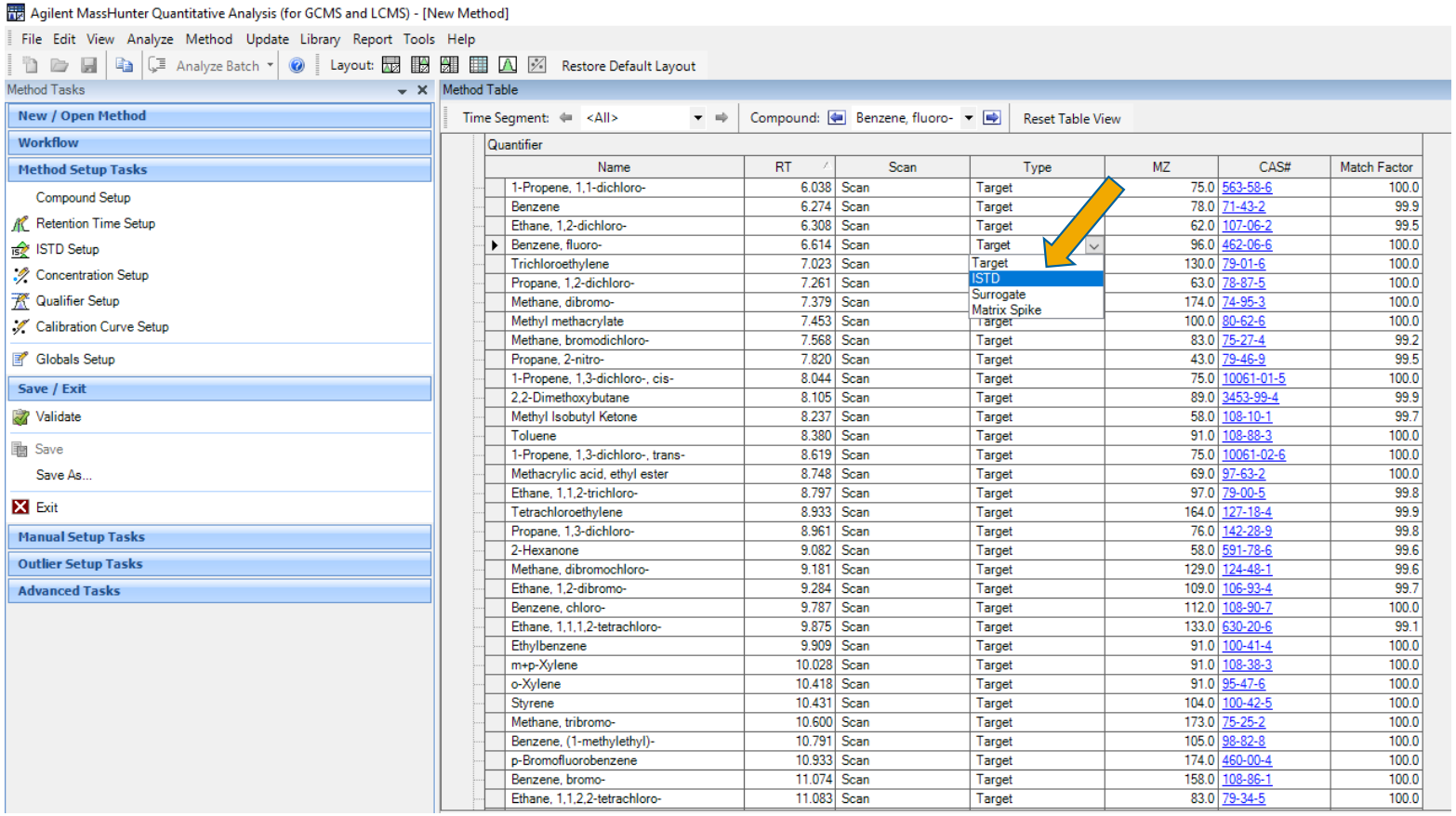
Step 3: Creating Quantitative Analysis Method

New method from acquired scan data with library search



Step 3: Creating Quantitative Analysis Method

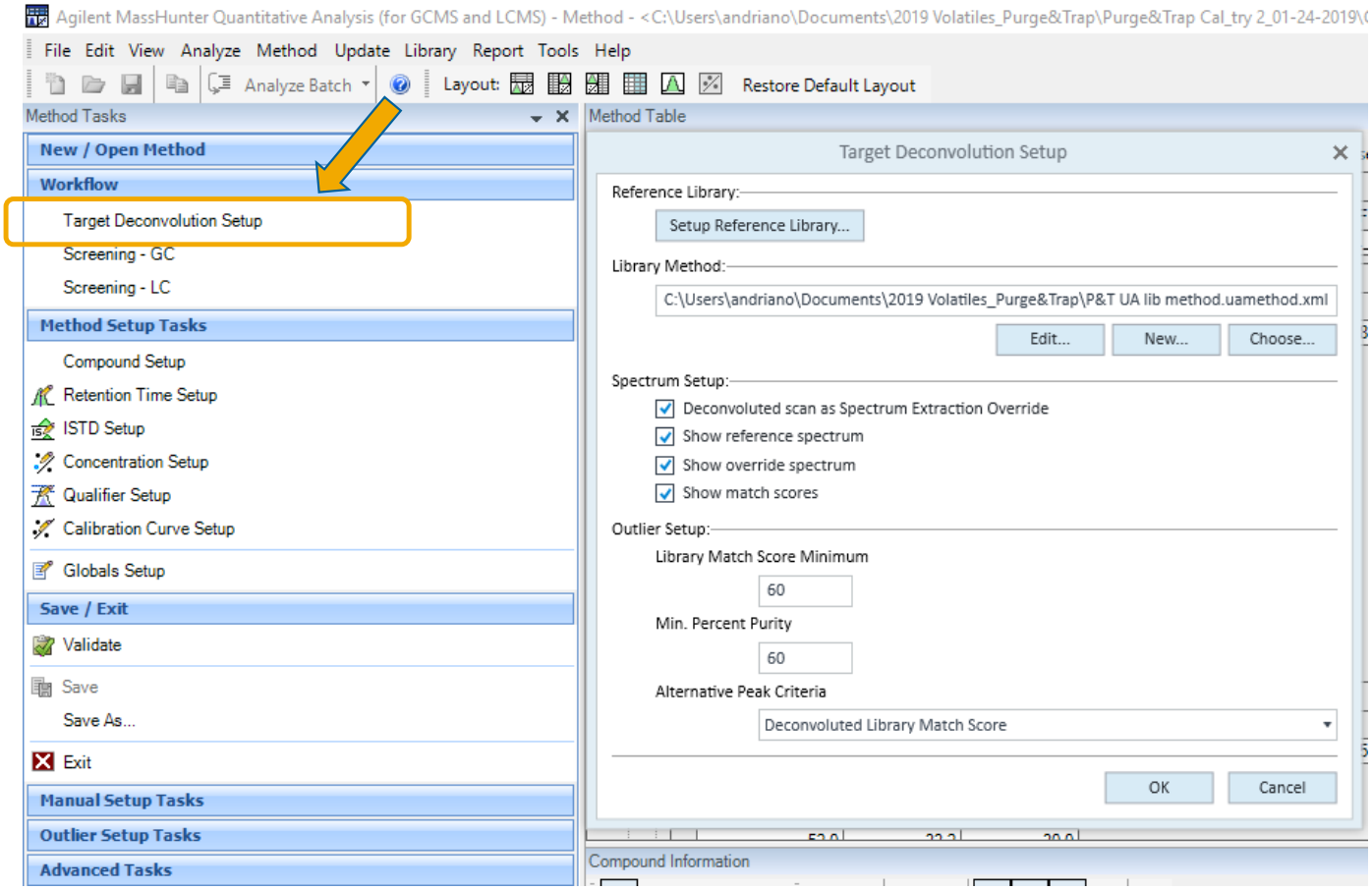
Define ISTD and surrogates



Name	RT	Scan	Type	MZ	CAS#	Match Factor
1-Propene, 1,1-dichloro-	6.038	Scan	Target	75.0	563-58-6	100.0
Benzene	6.274	Scan	Target	78.0	71-43-2	99.9
Ethane, 1,2-dichloro-	6.308	Scan	Target	62.0	107-06-2	99.5
Benzene, fluoro-	6.614	Scan	Target	96.0	462-06-6	100.0
Trichloroethylene	7.023	Scan	Target	130.0	79-01-6	100.0
Propane, 1,2-dichloro-	7.261	Scan	Target	63.0	78-87-5	100.0
Methane, dibromo-	7.379	Scan	Target	174.0	74-95-3	100.0
Methyl methacrylate	7.453	Scan	Target	100.0	80-62-6	100.0
Methane, bromodichloro-	7.568	Scan	Target	83.0	75-27-4	99.2
Propane, 2-nitro-	7.820	Scan	Target	43.0	79-46-9	99.5
1-Propene, 1,3-dichloro-, cis-	8.044	Scan	Target	75.0	10061-01-5	100.0
2,2-Dimethoxybutane	8.105	Scan	Target	89.0	3453-99-4	99.9
Methyl Isobutyl Ketone	8.237	Scan	Target	58.0	108-10-1	99.7
Toluene	8.380	Scan	Target	91.0	108-88-3	100.0
1-Propene, 1,3-dichloro-, trans-	8.619	Scan	Target	75.0	10061-02-6	100.0
Methacrylic acid, ethyl ester	8.748	Scan	Target	69.0	97-63-2	100.0
Ethane, 1,1,2-trichloro-	8.797	Scan	Target	97.0	79-00-5	99.8
Tetrachloroethylene	8.933	Scan	Target	164.0	127-18-4	99.9
Propane, 1,3-dichloro-	8.961	Scan	Target	76.0	142-28-9	99.8
2-Hexanone	9.082	Scan	Target	58.0	591-78-6	99.6
Methane, dibromochloro-	9.181	Scan	Target	129.0	124-48-1	99.6
Ethane, 1,2-dibromo-	9.284	Scan	Target	109.0	106-93-4	99.7
Benzene, chloro-	9.787	Scan	Target	112.0	108-90-7	100.0
Ethane, 1,1,1,2-tetrachloro-	9.875	Scan	Target	133.0	630-20-6	99.1
Ethylbenzene	9.909	Scan	Target	91.0	100-41-4	100.0
m+p-Xylene	10.028	Scan	Target	91.0	108-38-3	100.0
o-Xylene	10.418	Scan	Target	91.0	95-47-6	100.0
Styrene	10.431	Scan	Target	104.0	100-42-5	100.0
Methane, tribromo-	10.600	Scan	Target	173.0	75-25-2	100.0
Benzene, (1-methylethyl)-	10.791	Scan	Target	105.0	98-82-8	100.0
p-Bromofluorobenzene	10.933	Scan	Target	174.0	460-00-4	100.0
Benzene, bromo-	11.074	Scan	Target	158.0	108-86-1	100.0
Ethane, 1,1,2,2-tetrachloro-	11.083	Scan	Target	83.0	79-34-5	100.0

Step 3: Creating Quantitative Analysis Method

Add target deconvolution (optionally)



Step 3: Creating Quantitative Analysis Method

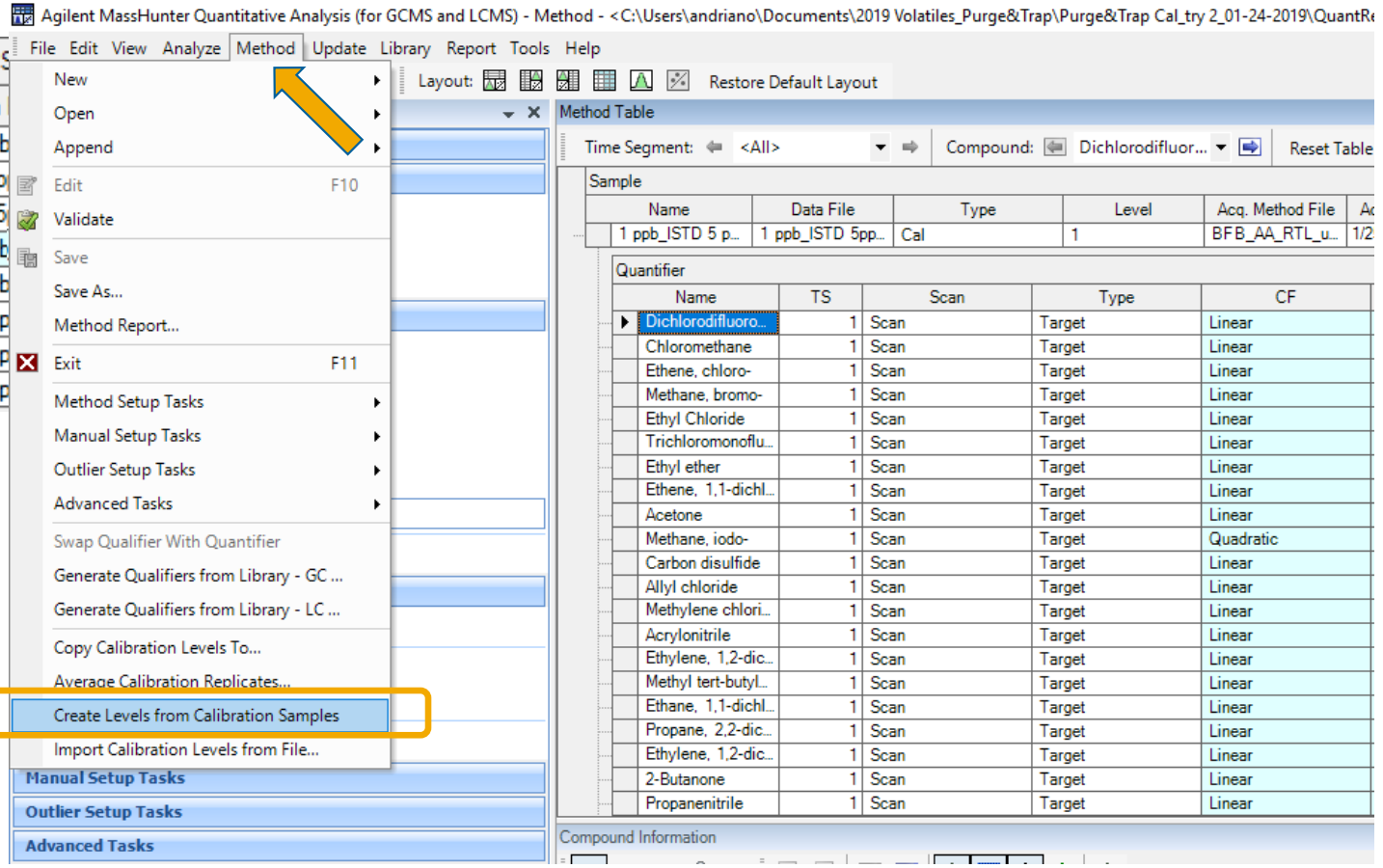
Create calibration levels from the analyzed calibration standards

Sample			
Name	Data File	Type	Level
blank_ISTD 5 ppb	blank_ISTD 5ppb.D	Blank	
0-5 ppb_ISTD 5 ppb	0-5 ppb_ISTD 5ppb.D	Cal	0.5
0-25 ppb_ISTD 5 ppb	0-25 ppb_ISTD 5ppb.D	Cal	0.25
1 ppb_ISTD 5 ppb	1 ppb_ISTD 5ppb.D	Cal	1
5 ppb_ISTD 5 ppb	5 ppb_ISTD 5ppb.D	Cal	5
10 ppb_ISTD 5 ppb	10 ppb_ISTD 5ppb.D	Cal	10
25 ppb_ISTD 5 ppb	25 ppb_ISTD 5ppb.D	Cal	25
50 ppb_ISTD 5 ppb	50 ppb_ISTD 5ppb.D	Cal	50

Step 3: Creating Quantitative Analysis Method

Create calibration levels from the analyzed calibration standards

Name	Data
blank_ISTD 5 ppb	blank_ISTD 5ppb
0-5 ppb_ISTD 5 ppb	0-5 ppb_ISTD 5p
0-25 ppb_ISTD 5 ppb	0-25 ppb_ISTD 5
1 ppb_ISTD 5 ppb	1 ppb_ISTD 5ppb
5 ppb_ISTD 5 ppb	5 ppb_ISTD 5ppb
10 ppb_ISTD 5 ppb	10 ppb_ISTD 5pp
25 ppb_ISTD 5 ppb	25 ppb_ISTD 5pp
50 ppb_ISTD 5 ppb	50 ppb_ISTD 5pp



Step 3: Creating Quantitative Analysis Method

Create calibration levels from the analyzed calibration standards

Quantifier

Name	TS	Scan	Type	MZ	RT
Ethene, 1,1-dichl...	1	Scan	Target	61.0	2.683

Qualifier

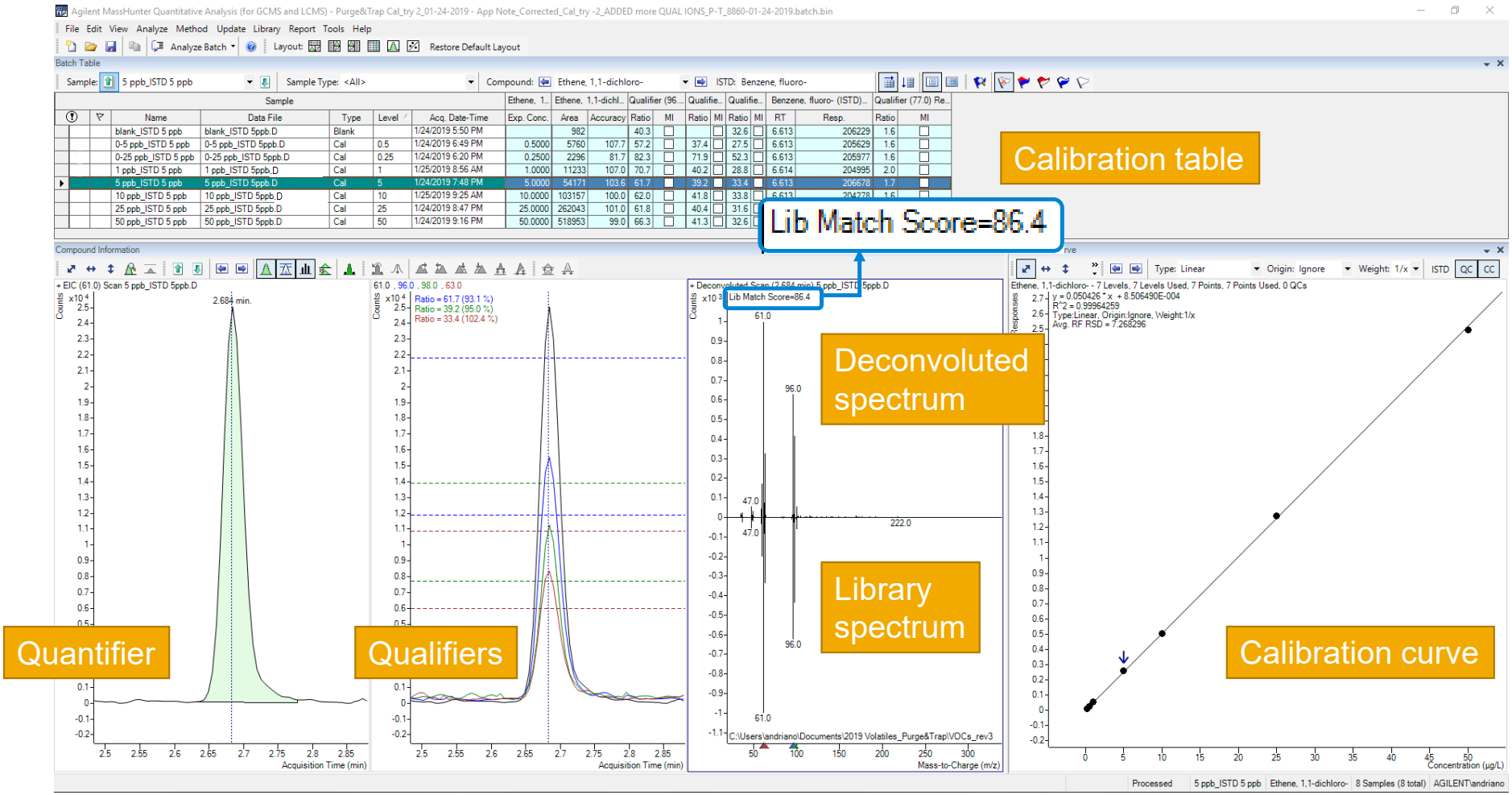
MZ	Rel. Resp.	Uncertainty
96.0	66.3	100.0
98.0	41.3	100.0
63.0	32.6	100.0

Calibration

Level	Conc.	Response
0.25	0.2500	2296
0.5	0.5000	5760
1	1.0000	11233
5	5.0000	54171
10	10.0000	103157
25	25.0000	262043
50	50.0000	518953

The method is ready to analyze calibration standards and samples

ICAL Review in MassHunter Quantitative Analysis 10.1



Initial Calibration (ICAL)

0.25–50 µg/L for 68 compounds (85%)

0.50–50 µg/L for 74 compounds (93%)

79 compounds out of 80 met the EPA criteria of less than 20% RSD with linear calibration

A quadratic fit with R^2 of 0.9963 was used for iodomethane

The %RSDs for the internal standard and surrogate compounds introduced by the AQUATek LVA were <5% RSD

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

Method Detection Limits (MDLs)

1.2 Method detection limits (MDLs) (3) are compound, instrument and especially matrix dependent and vary from approximately 0.02 to 1.6 µg/L.

<0.10 µg/L for 58 compounds (73%)

<0.15 µg/L for 66 compounds (83%)

8 trials at 0.25 µg/L for 66 compounds

For the compounds with higher reporting limits – 8 trials at 0.5 and 1 µg/L

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

MDLs in pg level

Table 5. Calculated MDLs for VOCs.

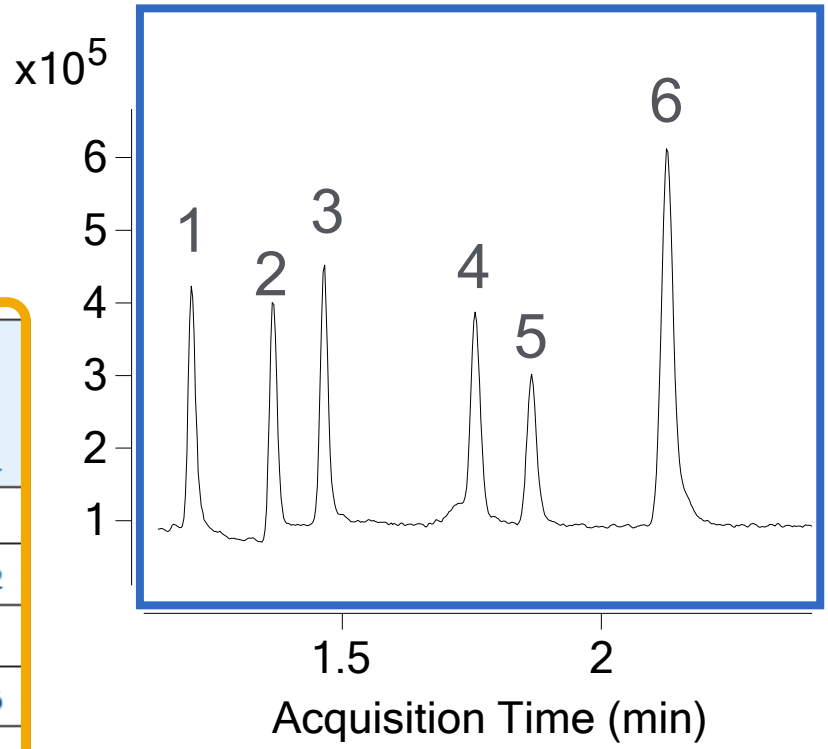
Compound	RT (min)	Calculated Concentration in the Sample (µg/L)									Average concentration (µg/L)	SD	MDL
		Spike (µg/L)	Samp. 1	Samp. 2	Samp. 3	Samp. 4	Samp. 5	Samp. 6	Samp. 7	Samp. 8			
Fluorobenzene (ISTD)	6.613	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	N/A	N/A
Dichlorodifluoromethane	1.207	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

MDLs for Gases

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

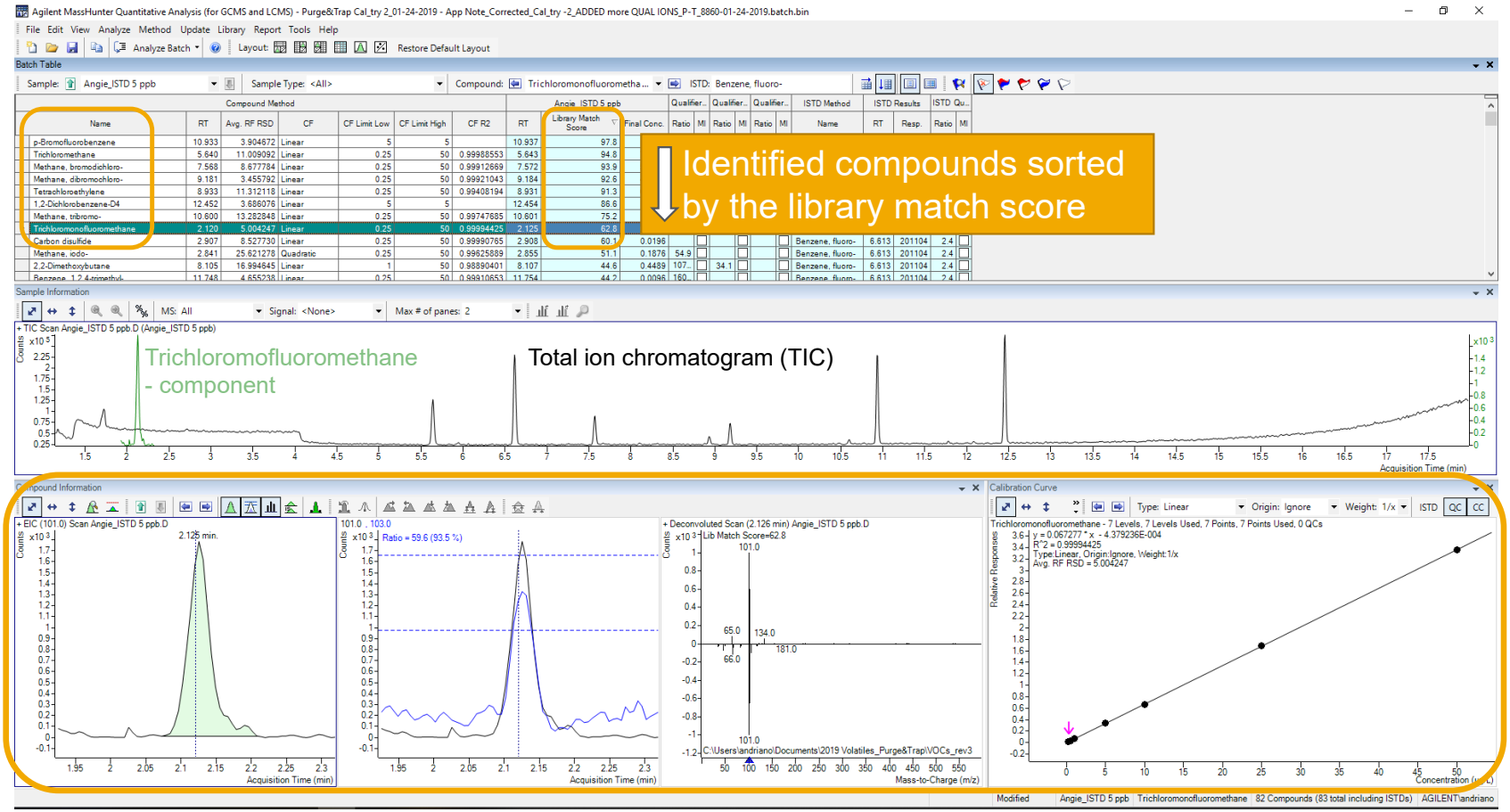
Table 5. Calculated MDLs for VOCs.

	Compound	RT (min)	Average concentration (µg/L)	SD	MDL
1	Dichlorodifluoromethane	1.207	0.24	0.010	0.031
2	Chloromethane	1.362	0.26	0.017	0.052
3	Chloroethene	1.462	0.16	0.017	0.051
4	Bromomethane	1.752	0.87	0.106	0.316
5	Ethyl Chloride	1.858	0.34	0.035	0.103
6	Trichloromonofluoromethane	2.120	0.22	0.030	0.091



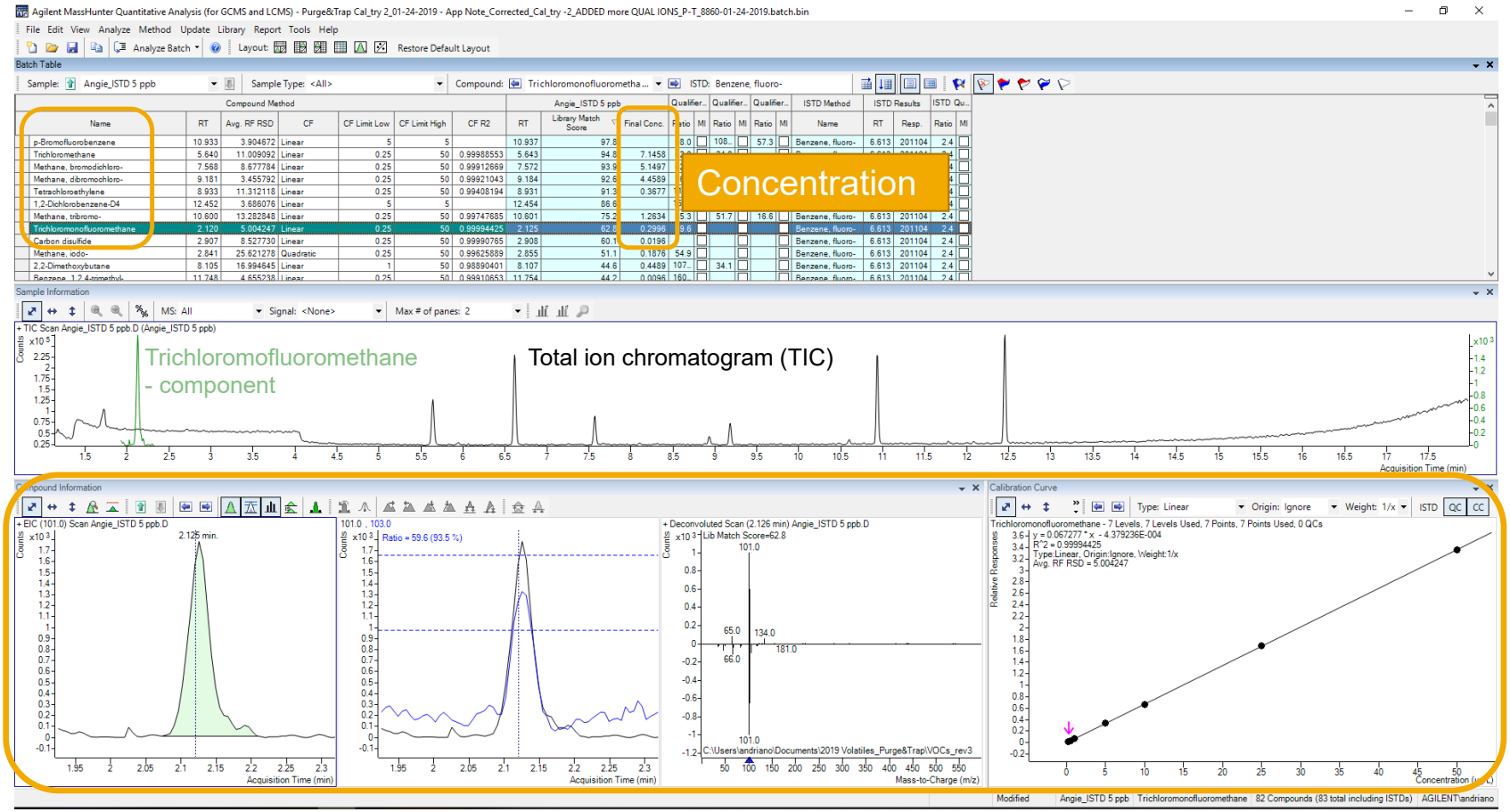
US EPA Method 524.2: Real World Samples

Tested tap water sample from Eastern Pennsylvania: MassHunter Quantitative Analysis



US EPA Method 524.2: Real World Samples

Tested tap water sample from Eastern Pennsylvania: MassHunter Quantitative Analysis



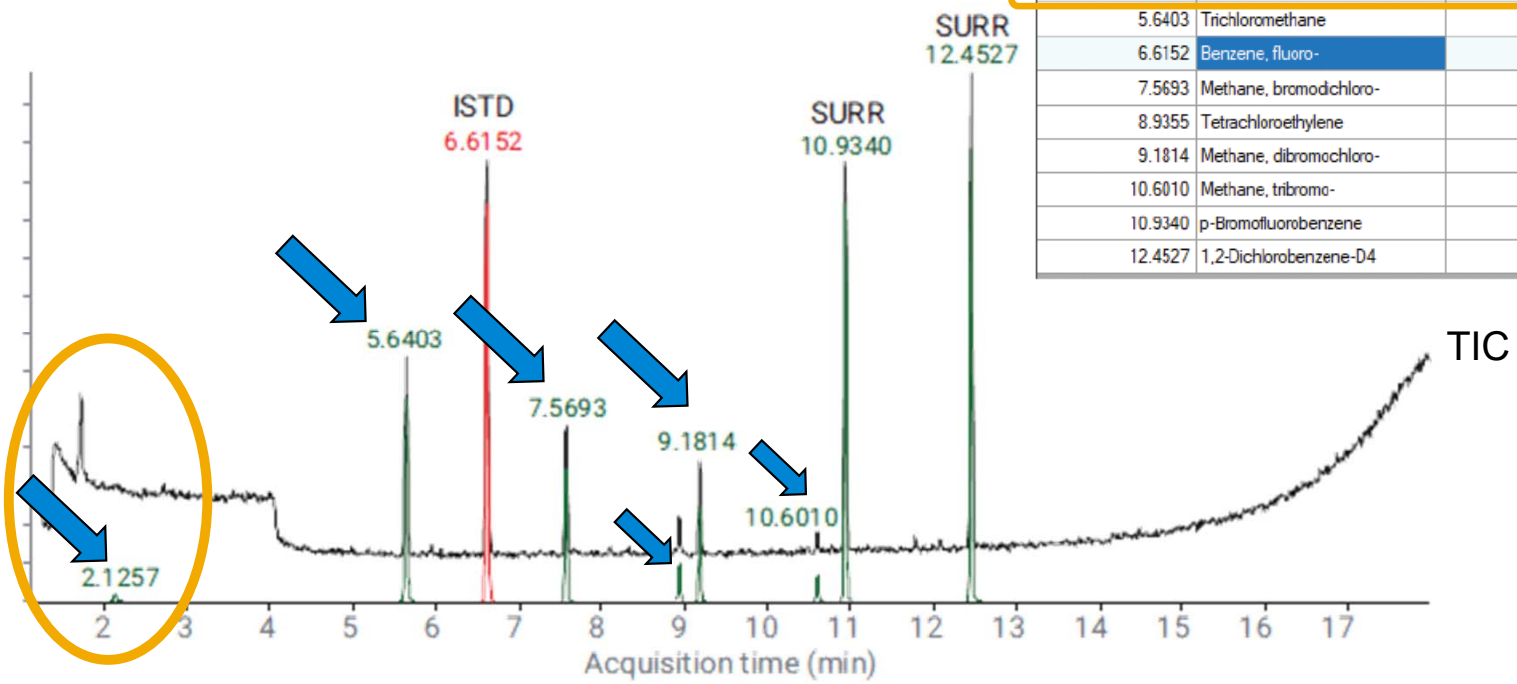
US EPA Method 524.2: Real World Samples

Tested tap water sample from Eastern Pennsylvania: MassHunter Unknowns Analysis

VOCs found:

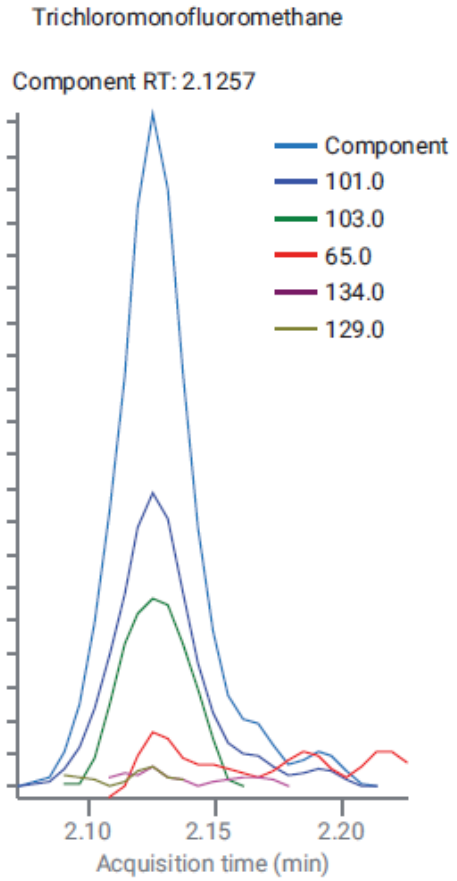
Components

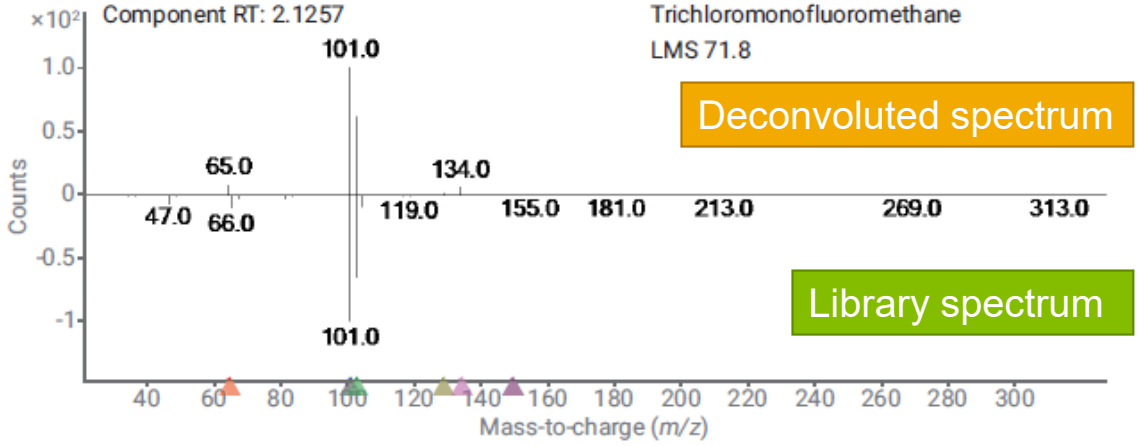
Component RT	Compound Name	Match Factor	Best Hit	Formula	Library RT	Delta RT
2.1257	Trichloromonofluoromethane	71.8	☑	CCl3F	2.1205	-0.0052
5.6403	Trichloromethane	95.2	☑	CHCl3	5.6396	-0.0007
6.6152	Benzene, fluoro-	97.5	☑	C6H5F	6.6141	-0.0011
7.5693	Methane, bromodichloro-	95.3	☑	CHBrCl2	7.5681	-0.0012
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

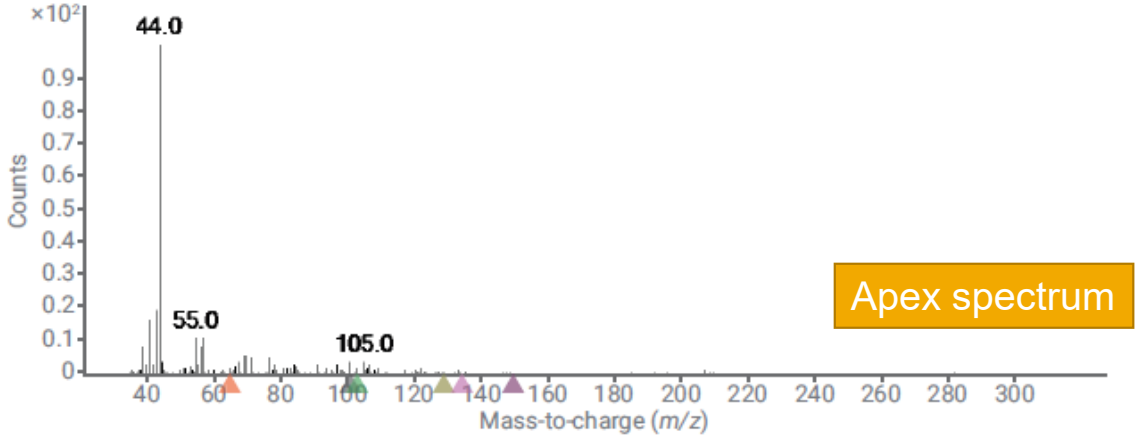


TIC

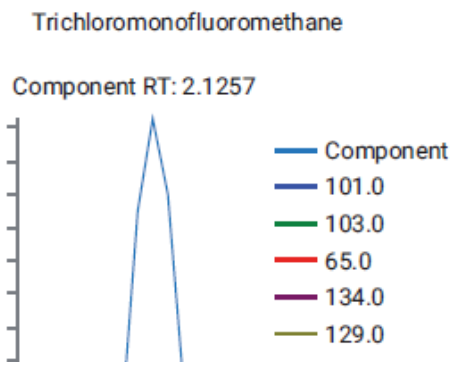
Trichloromonofluoromethane Found in a Tap Water Sample from Eastern Pennsylvania: Review in Unknowns Analysis

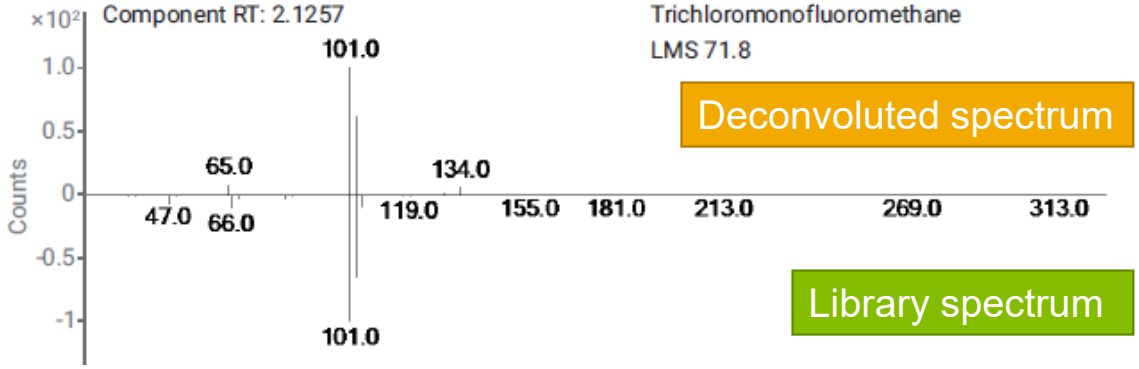






Trichloromonofluoromethane Found in a Tap Water Sample from Eastern Pennsylvania: Review in Unknowns Analysis



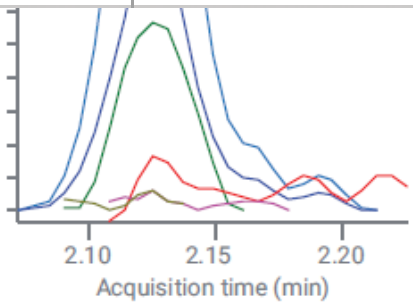


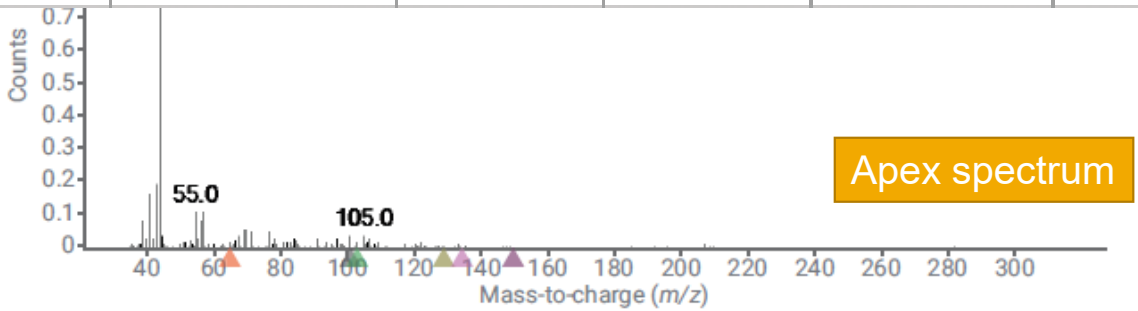
Deconvoluted spectrum

Library spectrum

Components

Component RT	Compound Name	Match Factor	Best Hit	Formula	Library RT	Delta RT
2.1257	Trichloromonofluoromethane	71.8	☑	CCl3F	2.1205	-0.0052





Apex spectrum

US EPA Method 524.2: Real World Samples

VOCs found in a tap water sample from four sources in Pennsylvania

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		

Summary

The software tools offer simple and rapid means to streamline quantitation method development, data processing, and reporting for the EPA 524.2 Method via:

- Built-in software tools for BFB tune check evaluation
- Creating user library of VOCs with deconvoluted spectra and retention times
- Creating a quantitation method from the user spectral library
- Analysis of the real-world samples using spectral deconvolution

ICAL was performed over the range of 0.25 to 50 µg/L

Several VOCs were identified and quantified in **real world tap water samples** at concentrations varying over the range of 0.3 to 14.1 µg/ L, in many cases much lower than current EPA 524.2 MCLs

EPA Method 524.2 with Agilent 8860/5977B

Application note 5994-0833EN

- Demonstrated applicability of an Agilent 8860/5977B GC/MSD system coupled with a Teledyne Tekmar Lumin purge and trap (P&T) concentrator, and an AQUATek LVA (liquid vial autosampler)
- ICAL range 0.25–50 µg/L
- MDLs in ppt levels
- Deconvoluted spectra for compound identification
- Several VOCs were identified and quantified in real-world drinking water samples

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

Authors
Bruce D. Quimby and
Anastasia A. Andrianova
Agilent Technologies, Inc.

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.

Thank you!