Volatile Organic Compound Analysis Using Purge and Trap

Success with VOC analysis using the Agilent 5975C Mass Selective Detector

Application Note

Environmental

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Abstract

Despite the status and widespread application of Purge and Trap (P&T) with GC/MS for volatile organics, various issues appear in consistent operation over the desired concentration ranges and at the required detection limits. This application note introduces an approach implemented and tested at several beta sites that consistently produces outstanding data. Detailed in this note are the hardware requirements, the P&T and GC/MS parameters, volatile organic compound (VOC) standard preparation, and a new automated tuning approach to meet USEPA tuning requirements that provides enhanced sensitivity and robustness. Initial calibration studies over the specified (USEPA 524.2) concentration range of 0.25 µg/L to 50 µg/L routinely produce VOC average relative response factors less than the 20% relative standard deviation (RSD) specified for average relative response factor quantitation. Data is also presented for the method detection limits determined at 0.10 µg/L as well as average relative response factors over the concentration range of 0.10 µg/L to 100 µg/L. Adhering to the details of this approach will help insure similar results are obtained for VOC analysis with P&T and GC/MS.

Agilent Technologies

Introduction

P&T methods for VOCs are some of the principle analyses performed by environmental laboratories over the last 35 years. Consequently there is a substantial history to P&T development (See Preface). The challenges presented in these methods are many as there are a multitude of parameters and analytes. This application note provides several key instructions for success in analyzing VOCs using P&T:

- Components of a GC/MS VOC accessory kit containing column, liner, methods, and other materials provided by Agilent Technologies to produce reliable and robust performance.
- P&T parameters for the Tekmar Atomix and StratUm/AQUATek 70 that have been implemented at several beta site laboratories as well as at Agilent Technologies.
- Details of a new automated tuning approach to reliably satisfy USEPA tuning requirements for 4-bromofluorobenzene (BFB) and improve sensitivity.

VOC data included in this application demonstrates excellent performance for the initial calibration (ICAL) and method detection limit (MDL) studies, in terms of linearity and dynamic working range. The application has been further investigated at a series of beta sites that also produced excellent datasets. To promote this approach, sections of the note address:

- Standard preparation and the performance of the semi-automated dilution capability of the Atomx and mirrored by manual dilution for the StratUm/AQUATek 70.
- Details of the ICAL and its requirements and performance under these conditions.
- Data demonstrating MDLs for the VOCs of interest in both 524.2 and 8260B.
- Troubleshooting the new tuning process and GC/MS system and indicators of issues in the P&T process for various analytes.

Deviations from the parameters presented in this recommended approach will produce a variety of problems in VOC analysis.

Preface

Recent commercialization of VOC analyses in soil/solid/sludge samples has made it important to review the milestones in the history and evolution of this technology because their fundamentals still apply today.

The first milestone in this analysis was set by Tom Bellar, a chemist at USEPA–Cincinnati, in 1974 when he invented a sample extraction technique known as Purge and Trap (P&T). This technique for extracting trace levels of volatile organic pollutants from water and soil improved current analytical instrumentation levels of detection and sample preparation techniques by several orders of magnitude, lowering these levels of detection into the part-per-billion (ppb) range. Using Tom Bellar's discovery as a basis, Lothar Witt and Jim Grote, the two founders of the Tekmar Company in Cincinnati, OH, worked with USEPA–Cincinnati in developing the first commercially available P&T device introduced in 1976. Trace levels of detection were achieved by coupling the P&T to a gas chromatograph (GC) and using one or more of the many available two-dimensional (2-d) detectors, such as an electrolytic conductivity detector (ELCD), or a photoionization detector (PID). Concurrently, the mass spectrometer began to be the detector of choice for VOC analysis. Because the levels of detection were superior with 2-d detectors and the mass flow of carrier gas required to achieve good chromatographic separation with larger id columns tended to be limiting, the common use of coupling a gas chromatograph with a mass spectrometer (GC/MS) for 3-dimensional (3-d) detection was not widely seen until the early to mid-1980s.

The next significant milestone in VOC analysis, and GC analysis in general, came in the mid to late 1970's when Hewlett-Packard Company scientists R.D. Dandeneau and E.H. Zerenner introduced polyimide reinforced fused-silica capillary columns at the Third Hindelang Symposium (29 April 3 May 1979) [1]. In their presentation, they described the production and use of thin-walled, flexible, fused-silica columns. Their tubing was an adaptation of the production of fiber optics manufactured by the Hewlett-Packard Company. Dandeneau and Zerenner realized that cracks could develop in the thin walls of the tubing, leading to breakage, so they coated the outside of the tubing immediately after drawing. Initially, they coated the tubing with silicone rubber but later changed to polyimide [2]. The introduction of fused-silica columns revolutionized the field of separation science. Soon column suppliers began to manufacture fused-silica capillary columns making other column types obsolete for many applications [3].

In the early 1990s, the Hewlett-Packard Company introduced the 5890A Series II Gas Chromatograph equipped with Electronic Pressure/Pneumatic Control (EPC), which significantly changed chromatographic performance by controlling column carrier gas through constant pressure or mass flow. This advancement provided superior carrier gas control on the analytical column, as well as appropriate mass flow control of the carrier gas into the high vacuum environment of a mass spectrometer. Specific to VOC analysis, at the 1993 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy held in Atlanta, GA, a novel approach to managing gas mass flows across the analytical column and the trap in the P&T device was introduced. This split-technique demonstrated an alternative way of transferring the sample from the P&T device into the GC. A microbore capillary column (20 m × 0.18 mm, 1.0 µm) was used in conjunction with EPC split/splitless injection port. The instrument configuration optimized the effects of column internal diameter, desorb flow rate, and injection port configuration on the resolution of chromatography, reproducibility, and the sensitivity of VOC analysis [4]. This configuration is widely used today for P&T, and headspace instrumentation, and remains the foundation of the successful approach cited in this application note.

Through the 1990's and into the 21st century, many significant advances occurred to analytical instrumentation used for VOC analysis by GC/MS. A partial chronological list of their introductions is:

- 1993 Tekmar LSC 3000 PTC
- 1996 Hewlett-Packard 5973A MSD
- 1998 Tekmar LSC 3100 PTC
- 1998 Tekmar AQUATek 70
- 2003 Teledyne-Tekmar Velocity PTC
- 2004 Agilent 7890A GC
- 2005 Agilent 5975A MSD
- 2007 Teledyne-Tekmar StratUm PTC
- 2009 Agilent 5975C MSD (with TAD)
- 2009 Teledyne-Tekmar Atomx ASPS

These advances provided distinct improvements in detectability. The ppb concentration range of 38 years ago was eclipsed when Tom Bellar at USEPA–Cincinnati invented the P&T extraction technique and when Lothar Witt and Jim Grote from the Tekmar Company introduced the LSC-1 and commercialized the analysis. Now, limits of detection are routinely in the low ppt range, and for some compounds approaching part-per-quadrillion (ppq) levels of detection is feasible. It is important to follow sound laboratory practices to achieve, optimize, and maintain method accuracy, precision, and sensitivity. In addition, best practices developed from decades of VOC analyses are even more critical. This application note applies these sound fundamentals and introduces a standard practice for tuning the MSD. This practice meets the method requirements cited by the USEPA for tuning for 4-bromofluorobenzene (BFB).

Methodology

This work is based on the two most widely used methods for VOC analysis by GC/MS:

1. *Method 524.2 Revision 4.1 – Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry.* 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. It is applicable to a wide range of organic compounds, including the four trihalomethane disinfection by-products (THMs), which have sufficiently high volatility and low water solubility to be removed from water samples with P&T procedures [5].
2. *Method 8260B – Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*. This method is used to determine volatile organic compounds in a variety of solid waste matrices. It is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments [6].

Fundamentally, the approach and the instrument methodology for these methods are the same. However, they address different matrices, therefore they require differences in QA/QC. The focus of this application note is specifically Method 524.2 Revision 4.1. The conditions under which results for Method 8260B were acquired are also presented.

Experimental

Numerous permutations of hardware internal to Agilent Technologies (R&D Santa Clara Site, CA) were used at selected test sites for this study. The instrumentation includes seven different configurations listed in Table 1.

The Agilent VOC Kit (p/n G7022A) was used for all tests at all sites. This kit has proven to be critical for successful VOC analysis. The contents of this kit are listed in Table 2.

The authors recommend an alternative sample pathway treatment process by Advanced Materials Components Express, L.L.C., Lemont, PA USA (ACMX) called Inertium. Inertium has all of the benefits expected from fused silica and glass lining surfaces such as Silcosteel and Silinert, or Silonite. Further research is needed to conclusively determine which components of the sample pathway in the P&T concentrator will benefit using tubing with the Inertium process. However, data collected by the authors and selected beta site participants suggests superior performance with the use of Inertium processed tubing, especially in the transfer line between the P&T concentrator and the inlet of the GC.

It is recommended that the GC/MS system demonstrates an acceptable performance level before implementing this analysis. There are many performance checks and tests available for the GC, MSD, and P&T hardware to ensure a level of performance within acceptable and expected limits. An evaluation of the autotune for MSD by executing the Checkout Tune is an example of such a test. Instructions for performing this evaluation is found in the Verifying EI Performance section of the Agilent 5975 Series Mass Selective Detectors Hardware Installation Manual [7]. Regardless of the steps taken, the GC/MS must be qualified in order to successfully implement this analysis.

Table 1. Instrument Configurations

System	Agilent GC	Agilent MSD	Purge and trap system	Method	Sample size (mL)
1	7890A	5975C w/TAD	Atomx	524.2	25
2	7890A	5975C w/TAD	Atomx	8260B	10
3	7890A	5975C w/TAD	StratUm + AQUATek 70	524.2	25
4	7890A	5975C w/TAD	StratUm + AQUATek 70	8260B	10
5	7890A	5975C w/TAD	Velocity + AQUATek 70	524.2	25
6	7890A	5975C w/TAD	Eclipse + Centurion WS	8260B	5
7	6890N	5973N	StratUm + AQUATek 70	8260B	5

Systems 1 and 3 were used at Agilent Technologies (R&D Santa Clara Site, CA).

Table 2. Agilent VOC Kit Contents (p/n G7022A)

Description	Agilent part number
6 mm Drawout plate (Inert) for Agilent 5973 and Agilent 5975 MSD Inert EI Ion Source	G2589-20045
DB-624UI Column (20 m × 0.18 mm, 1.0 µm film)	121-1324UI
Straight-through 1.0 mm UI Liner	5190-4047
Tekmar VOCARB 3000 (#K) Trap	5188-8820
Agilent GC/MS VOC Application Kit Disk with application note, kit instructions, instrument methods, and applicable technical notes	G7022-60001

The process of initially setting up the GC/MS for the analysis of VOCs and verifying method performance can be divided into three activities, referred to as the initial demonstration of capability (IDOC):

1. Acquiring and meeting the spectral fidelity requirements for 4-bromofluorobenzene (BFB) as the Tune Check Standard.
2. Initially calibrating for target analytes and meeting specific method requirements (ICAL).
3. Conducting a Method Detection Limit study (MDL) across the list of target analytes.

These initial three steps differ slightly based on the analytical method implemented. However, these three fundamental steps are common across virtually all USEPA GC/MS methods. The first step, acquiring and meeting the spectral fidelity requirements for BFB, is critical to the success of VOC analysis. Prior to acquiring any calibration or sample data, the MSD must be tuned so that the specified spectral fidelity criteria are met for BFB. In addition to meeting the spectral fidelity requirements, tune stability over time is required. Both Method 524.2 and 8260B have a 12-hour window in which the

MSD must remain stable. From a BFB spectral fidelity perspective, the analysis of BFB must be performed every 12 hours and the spectral fidelity of BFB evaluated. One of the objectives of this work is to create a stable environment for the MSD so that tune stability is improved over time. Detailed parameters for acquiring BFB as the Tune Check Standard are given in Appendixes A and C.

The analysis of BFB as the Tune Check Standard is very similar for both Method 524.2 and 8260B [5,6]. A specific amount of BFB is injected into the GC, providing a specific mass amount on-column. Method 524.2 specifies 25 ng or less and Method 8260B specifies 5 to 50 ng. Neither method specifies that BFB be introduced as a direct injection or by P&T.

If P&T is the selected method of introducing BFB, it should be noted that the sample is split at the inlet. Therefore, the mass amount on-column will be greatly reduced, reducing the response of BFB. As a result of this study, the authors recommend that a 1-µL solution of an appropriate mass amount of BFB be directly injected into the GC instead of purging. This ensures that a more representative amount of BFB is analyzed and evaluated against the method specific criteria.

Laboratories typically use a variety of sample sizes, but 5 mL and 25 mL are the most common volumes. The parameters for Tekmar P&T systems (Atomx and Stratum/AquaTEK 70) are listed in Appendixes C and D for both 5 mL and 25 mL sample sizes.

The software used on all PC's for acquisition and data reduction was the Agilent MSD Productivity ChemStation Revision E.02.02 SP1. The MS tuning algorithm BFB Autotune is part of this revision of the MSD Productivity ChemStation.

Tuning the MSD

Background

Early in the application of GC/MS to environmental chemistry, the USEPA developed criteria for spectral correspondence in reference compounds as an attempt to validate instrument performance, create greater quality assurance for data, improve spectral library matching and compound identification, and enhance the consistency of data. The result was ion ratio abundance criteria for significant ions in the 4-bromofluorobenzene spectrum. These criteria have become limiting because, as GC/MS instruments evolved, spectral performance changed. The addition of high-energy conversion dynodes (HEDs) to GC/MS detectors has enhanced signals for higher mass fragments that are more unique and have improved compound identification and detection.

Increased emphasis on high mass samples has continued as the use of pesticides and other higher molecular weight compounds PCBs, PBDEs expands. However, enhancement of higher mass ion abundance is in opposition to the criteria for BFB. To meet the spectral criteria developed in the late 1970's, present-day ion sources must be detuned from optimal conditions to achieve the spectral fidelity required by the method. The BFB spectral target ion abundance ratio criteria from USEPA Method 524.2 are given in Tables 3 and 4.

Table 3. USEPA Method 524.2 Ion Abundance Criteria for 4-Bromofluorobenzene (BFB) and Expected Range of Relative Abundances for BFB under BFB Autotune [5]

Mass (*m/z*)	Relative abundance criteria	Expected range of relative abundances for critical ions for BFB Autotune
50	15 to 40% of mass 95	15 to 20% 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	> ~80% of mass 95
175	5 to 9% of mass 174	
176	> 95% but < 101% of mass 174	
177	5 to 9% of mass 176	

Table 4. USEPA Method 8260B Ion Abundance Criteria for 4-Bromofluorobenzene (BFB) and Expected Range of Relative Abundances for BFB under BFB Autotune [6]

Mass (*m/z*)	Relative abundance criteria	Expected range of relative abundances for critical ions for BFB Autotune
50	15 to 40% of mass 95	15 to 20% of mass 95
75	30 to 60% 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	> ~80% of mass 95
175	5 to 9% of mass 174	
176	> 95% but < 101% of mass 174	
177	5 to 9% of mass 176	

The 5973 and 5975 MSD platforms use a process of ramping the voltages of the ion source lens as a function of mass to meet selected spectral targets for the PFTBA tuning compound, which allows injected BFB spectra to pass the spectral criteria. This approach is known as "Target Tuning" with dynamic lens ramping. This is user interactive and flexible at the expense of some sensitivity. A new, completely automated BFB tuning algorithm has been developed for simplification and ease-of-use. The BFB Autotune provides a completely automated tune for better consistency, robustness, sensitivity, and better dynamic working range than the Target Tuning approach. This new tune is a cornerstone of the approach to the VOC analysis described in this application note.

The new BFB Autotune

Requirements

The BFB Autotune was designed to support the particular hardware configuration and operating conditions used in this study. This requires that the large 6 mm drawout plate be installed in the EI ion source (p/n Standard EI ion source G3163-20530, Inert EI ion source G2589-20045). The standard 3 mm drawout plate may support this tune but is not the recommended approach for this VOC analysis. For optimal operation, set the ion source and quadrupole temperatures to 250 °C and 200 °C, respectively.

Operating the BFB Autotune

If the system is new, run an autotune and examine the ATUNE.U report to ensure that the tuning parameters are correct and the ion source appears clean. A clean ion source will have low background noise, abundant calibrant, and EM voltage within limits. To access the BFB autotune, click **BFB Autotune** (BFB_Atune.U) on the **Tune** dropdown menu, in the **Tune and Vacuum Control** view (Figure 1). BFB Autotune takes about twice the time of standard autotuning because more parameters are optimized. An example of the BFB_ATUNE.U file report is shown in Figure 2. The emission settings may deviate from 35 µA because this parameter is also optimized with the other lens elements. A noteworthy feature is the tilt against higher mass. Users should expect the ratio of the 219 *m/z* : 69 *m/z* PFTBA fragments to be in the range of approximately 45–75%. The repeller settings are expected to range from 20 V to 32 V.

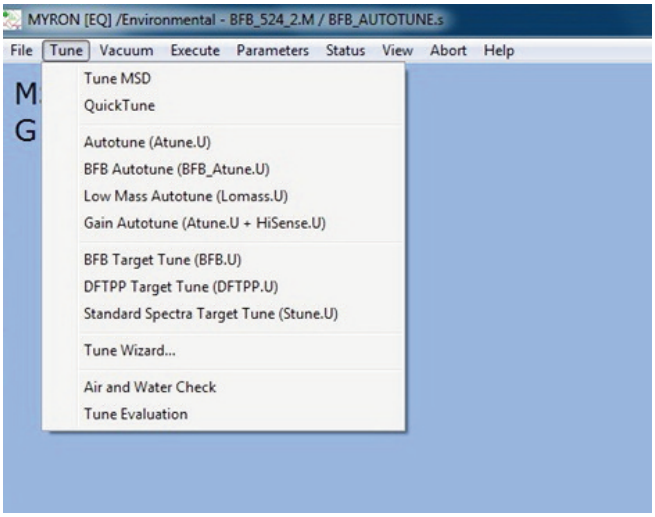


Figure 1. Tune and Vacuum Control view of the Agilent MSD Productivity ChemStation.

As with other MSD Productivity ChemStation tuning routines, the BFB Autotune can also be invoked in a sequence by keyword request.

1. In the **Sample Log Table**, select **Keyword** from the **Type** field dropdown menu.
2. Select **Tune** from the **Method/Keyword** field dropdown menu.
3. Enter **BFB_AUTUNE** in the **Comment/Keyword/String** field.
4. Click **OK** to save the changes to the sequence.

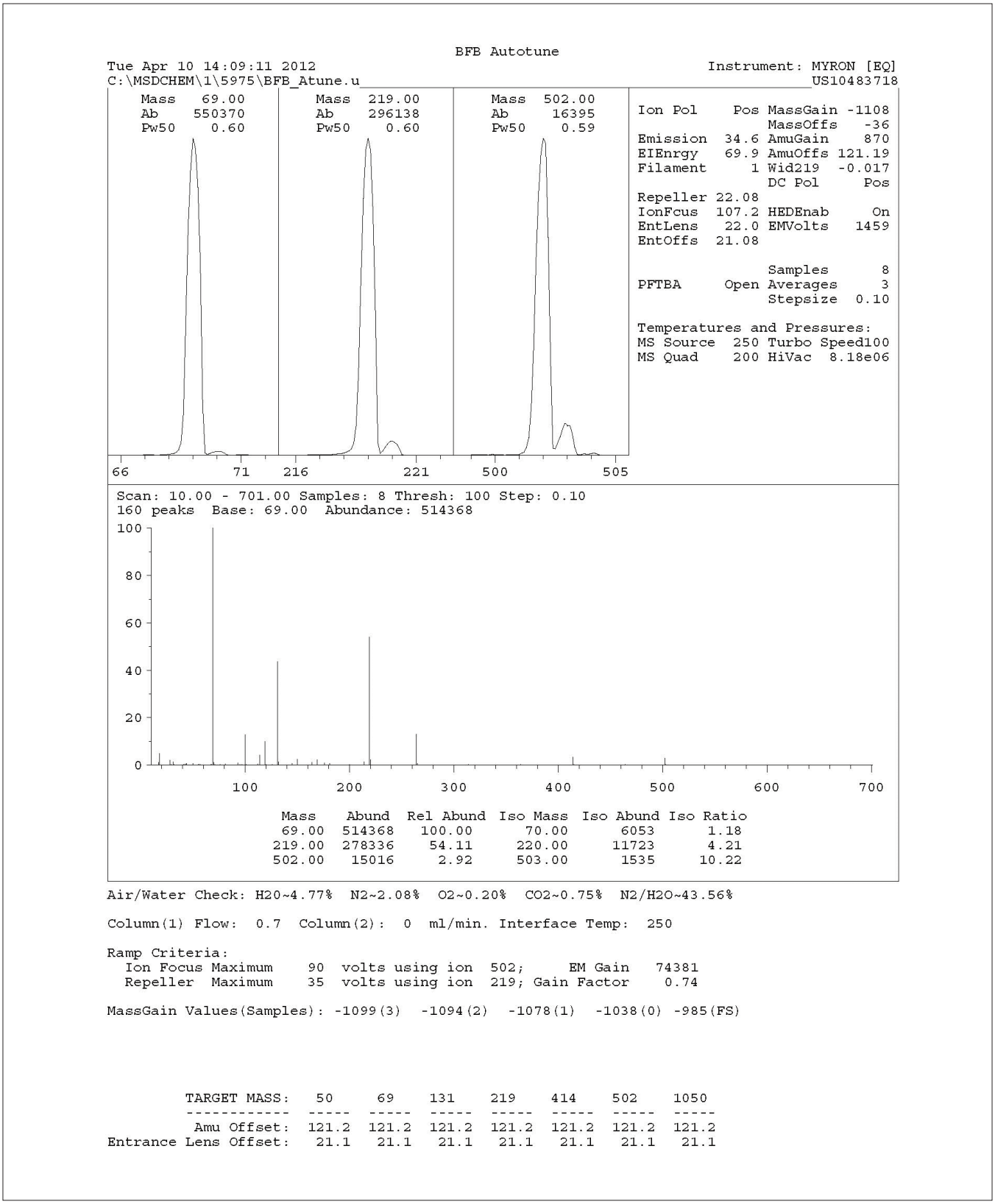


Figure 2. BFB Autotune report.

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 listed in Table 1. The MSD Productivity ChemStation EnviroQuant typically evaluates BFB as the average of the top three spectra across the chromatographic peak minus a background spectrum from the prior edge of the peak. A typical result of the BFB Autotune using an average of three scans for BFB is shown in Figure 4. Notice the BFB spectrum is comparatively strong in the 174 and 176 *m/z* ions and relatively low for mass 50 *m/z* relative to the 95 *m/z* fragment. This is characteristic of the BFB Autotune and the expected range in relative abundance for these critical ions is given in Table 1.

According to section 6.3.4 of Method 524.2, "An average spectrum across the BFB GC peak may be used to test instrument performance". The MSD Productivity ChemStation EnviroQuant (E.02.02 SP1) provides an option for evaluating an average across the peak (see Figure 5). This is more consistent with the fact that ion current peak areas are used for analyte quantitation.

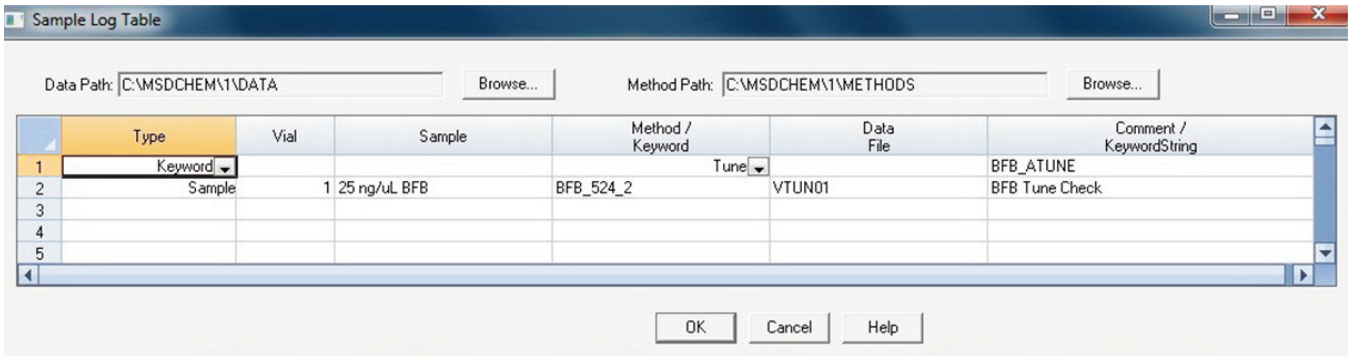


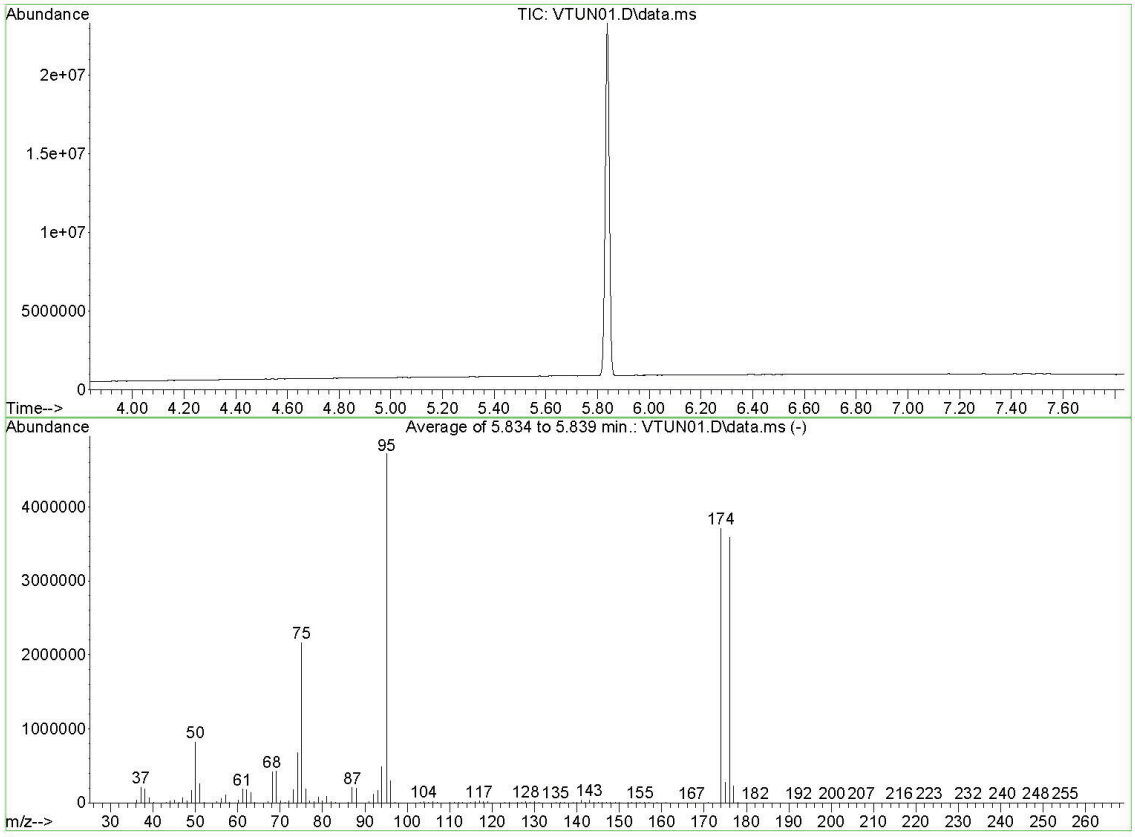
Figure 3. Use of sequence to invoke BFB Autotune.

Method 524.2 - BFB Tune Check

```
Data Path : C:\MSDchem\1\data\
Data File : VTUN01.D
Acq On    : 10 Apr 2012   2:13 pm
Operator  : jsh
Sample    : 2.5 ng OC BFB Tune Check | GF = 5.0
Misc      : 5975C MSD using BFB Autotune
ALS Vial  : 1   Sample Multiplier: 1

Integration File: rteint.p

Method    : C:\MSDchem\1\methods\BFB_524_2.M
Title     : Method 524.2
```



AutoFind: Scans 984, 985, 986; Background Corrected with Scan 970

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	17.3	817820	PASS
75	95	30	80	45.9	2162700	PASS
95	95	100	100	100.0	4716152	PASS
96	95	5	9	6.2	294737	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	78.7	3710849	PASS
175	174	5	9	7.4	273257	PASS
176	174	95	101	96.7	3590014	PASS
177	176	5	9	6.4	230523	PASS

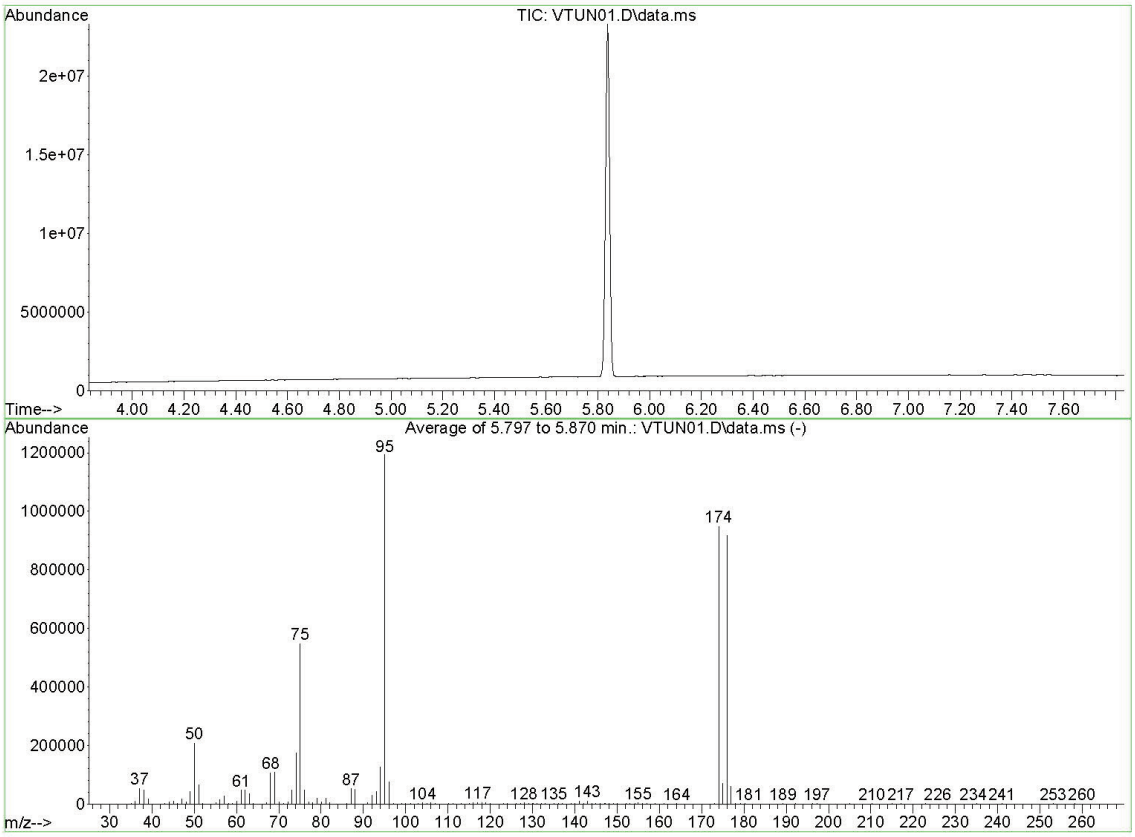
Figure 4. BFB Tune Check report using average of three scans.

Method 524.2 - BFB Tune Check

```
Data Path : C:\MSDchem\1\data\
Data File : VTUN01.D
Acq On    : 10 Apr 2012   2:13 pm
Operator  : jsh
Sample    : 2.5 ng OC BFB Tune Check | GF = 5.0
Misc      : 5975C MSD using BFB Autotune
ALS Vial  : 1   Sample Multiplier: 1

Integration File: rteint.p

Method    : C:\MSDchem\1\methods\BFB_524_2.M
Title     : Method 524.2
```



AutoFind: Averaged scan 971 to 997; Bkg corrected with scan 970)

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	17.4	208090	PASS
75	95	30	80	45.9	547083	PASS
95	95	100	100	100.0	1192636	PASS
96	95	5	9	6.3	75351	PASS
173	174	0.00	2	0.1	781	PASS
174	95	50	100	79.5	948003	PASS
175	174	5	9	7.4	70368	PASS
176	174	95	101	96.8	917661	PASS
177	176	5	9	6.4	58891	PASS

Figure 5. BFB Tune Check report using average across the entire GC peak.

BFB Failures

There are several ways BFB can fail to meet the criteria. Each failure mode indicates a different potential issue. For example, the Gain Factor setting during BFB testing must avoid saturating the detector during acquisition testing of the BFB spectra. The absolute abundance of 95 *m/z* fragment intensity must be below 8.2 million counts in height, preferably under 3 million. If it is too intense, check the actual amount injected and the Gain Factor setting in the mass spectrometer parameters view. Remember that the signal scales proportionately with the Gain Factor. For example, lowering the Gain Factor from 5 to 1 will also reduce the amplification of the ion current to detector signal (peak heights) by a factor of 5 [8].

From an VOC application perspective, the outdated practice of setting the EM voltage in the GC/MS acquisition method as tune voltage plus several hundred volts often produces signal saturation of the aromatics, specifically *m*+*p*-xylene. Proper use of the Gain Factor will consistently eliminate this problem. It should be noted that the same Gain Factor used to acquire the BFB Tune Check Standard should be used to acquire all data acquired under the conditions established for the VOC method.

Failures of BFB to meet the criteria fall broadly into two categories: persistent and occasional failures. In persistent failures, four or five consecutive evaluations of BFB fail the spectral criteria and usually involve failing specifically the 50 *m/z* : 95 *m/z* or 174 *m/z* : 95 *m/z* ratios. To resolve this failure, retune or clean the ion source depending on its history. In occasional failures, typically one or two consecutive spectra will not meet the criteria for any of the ion ratios. This does not warrant retuning or ion source maintenance. However, these failures will be compensated better than 50% using the spectral averaging option allowed by the 524.2 method.

To evaluate a new BFB Autotune, the critical ions present in the spectrum of BFB at of 50 and 174 *m/z* should be tested. It is recommended they are evaluated by the average across the peak approach described above. In the unlikely case of a tune failure, run the BFB Autotune again. If it fails again, an ion source cleaning is required. After cleaning an ion source, replacing a filament, or similar service the best approach is to delete the prior BFB_ATUNE.U file, perform an autotune and check the ATUNE.U report, and perform the BFB Autotune.

Summary of BFB Autotune Operation and Troubleshooting

Although the BFB Autotune is effective, it cannot overcome certain deficiencies in MSD performance. The following is a summary of the autotune operation:

1. Set the ion source and quadrupole to 250 °C and 200 °C (as appropriate to these VOC analysis methods), respectively, in ATUNE.U and wait at least two hours.
2. As recommended, bake out the analyzer at an ion source temperature of 300 °C and the quadrupole temperature at 200 °C for 3 hours prior to use.
3. Click **TUNE** from the menu to execute and evaluate autotune (ATUNE.U).
4. Scan and check the tune report to ensure that the background is low (typically less than 150 ions present in the spectrum of PFTBA) especially in the mass range near 50 *m/z*. If it is not, analyzing the VOC gases will be difficult even if a good BFB Autotune is provided.

Additional indicators pointing to the operational health of the ion source or the electron multiplier are:

- Is the electron multiplier voltage set in tuning to produce 69 *m/z* abundance near the autotune target of 500,000 counts? If this value is too low (< 250,000 counts), an error will be generated during the BFB Autotune and it will not complete until this is remedied.
- Is the electron multiplier voltage setting relatively low; less than 1,500 V? This is a good indicator of ion source cleanliness and the health of the electron multiplier. Electron multiplier voltages in excess of 2,000 V may in part indicate ion source cleaning needs to be performed and possibly replacement of an aged electron multiplier.
- Are the mass peak profiles of the tuning ions clear, well-shaped and intense? If not, clean the ion source properly [9]. Check the calibrant (PFTBA) level in the vial. Consider replacing the EM. Reinstall the ion source. From the **Tune and Vacuum Control** view, click **Execute > Bake out MSD** to bake the ion source and quadrupole for at least 3 hours at 300 °C and 200 °C, respectively.
- After executing the BFB Autotune, did the tune complete without ALERT error flags? There are three alerts that may appear, indicating a problem with the BFB Autotune:
  - **Calibrant intensity is too low**
    As described above, insufficient ion intensity for fragment mass 69 results from too low an EM voltage setting, too little calibrant in the vial, or a dirty ion source.

- **Filament failure**
  This message indicates a very dirty ion source, a warped filament, or a high pressure (column flow) in the ion source. Cleaning the ion source, replacing the flagged filament, or temporarily switching to the other filament may solve the problem. Another approach is to try the process again.

  1. Delete the BFB_Atune.U file.
  2. Retune with Autotune and inspect the tune report.
  3. Tune using BFB Autotune.

- **Exceeded ion focus limits**
  This error can occur if the ion focus and entrance lens leads are incorrectly installed or the ion source is very dirty.

Standard Preparation

The stock standards were acquired from AccuStandard, Inc. of New Haven, CT. The standards listed in Table 5 were used to implement Method 524.2.

Tune Check Standard Prep – BFB

The first step for VOC analysis by GC/MS is to ensure that the MSD is generating spectral data with acceptable fidelity for BFB. After completing a BFB Autotune, inject on-column the amount of BFB specified in the method.

Method 524.2 requires that 25 ng or less of BFB be analyzed and pass the spectral criteria specified in the method. To prepare the appropriate concentration from the Tune Check Standard stock solution listed in Table 4, consider the split ratio specified in the GC method so that, with the sample, the appropriate mass amount of BFB is injected on-column. The suggested split ratio for optimal VOC performance is 150:1. Therefore, dilute the BFB stock solution accurately so that the desired mass amount of BFB is injected on-column. If permissible, adjust the inlet split ratio to yield the desired mass amount of BFB on-column for direct injection. For example, to obtain a mass amount of 25 ng BFB on-column, use a 80:1 split ratio for a 1-µL injection of the stock Tune Check Standard (1 µL of 2,000 ng/µL ÷ 80 = 25 ng/µL or 25 ng on-column).

Initial Calibration Standard Preparation

Careful ICAL standard preparation is critical for successful VOC analysis. The quality of water and methanol used for standard preparation, and the cleanliness of the laboratory and glassware directly impact sub-ppb levels of detection. Always use P&T grade methanol and DI water prepared by reverse osmosis (DI/RO) when preparing dilutions of stock standards. Eliminate or at least minimize exposure to common organic solvents found in the laboratory, such as methylene chloride, or acetone, because they will be detected by the GC/MS system at levels below 1 ppb.

There are at least two different ways to prepare aqueous ICAL standards. Semi-automatically prepared aqueous ICAL standards are used with the Atomx P&T device to make the appropriate dilutions of a manually-prepared calibration level. Manually prepared aqueous ICAL standards are used with the StratUm/AQUATek 70 P&T system. These preparation schemes are listed in Tables 6 and 7 respectively.

Table 5. Stock Standard Solutions from AccuStandard

Name	Part number	Concentration	Description
Tune check standard	CLP-004-80X	2,000 µg/mL	4-Bromofluorobenzene
Fortification solution	M-524-FS	2,000 µg/mL	Fluorobenzene – IS 4-Bromofluorobenzene – SUR 1,2-Dichlorobenzene-d4 - SUR
Target compounds mix 1	M-502-10X	2,000 µg/mL	60 Target compounds
Target compounds mix 2	M-524R-B	2,000 µg/mL	24 Target compounds

Table 6. ICAL, Internal, and Surrogate Standard Preparation for the Atomx

Method 524.2 - ICAL and Fortification Standard Preparation

Using the Atomx

Final concentration	Concentration of ICAL standard	Dilution/Sample size
0.5 µg/L	50 µg/L [1]	100:1
1 µg/L	50 µg/L [1]	50:1
5 µg/L	50 µg/L [1]	10:1
10 µg/L	50 µg/L [1]	5:1
25 µg/L	50 µg/L [1]	2:1
50 µg/L	50 µg/L [1]	1:1
Fortification standard		
1.0 µg/L	5 µL using Atomx [2]	25 mL
5.0 µg/L	5 µL using Atomx [2]	5 mL

AccuStandard p/n M-502-10X	Method 502.2 volatile organic compounds (60 compounds) at a concentration of 2,000 µg/mL (2,000,000 µg/L).
AccuStandard p/n M-524R-B	Additions to Method 524.2 (24 compounds) at a concentration of 2,000 µg/mL (2,000,000 µg/L).
AccuStandard p/n M-524-FS	Method 524.2 fortification stock is at a concentration of 2,000 µg/mL (2,000,000 µg/L).

[1] Combine and dilute the stock target solutions EACH: 500 µL of 2,000 µg/mL (2,000,000 µg/L) in 10 mL MeOH for a 100 µg/mL (100,000 µg/L) working stock (WS) solution. Then, dilute 50 µL of 100 µg/mL (100,000 µg/L) in 10 mL DI water for a 0.05 µg/mL (50 µg/L) ICAL standard.

[2] Dilute stock fortification standard 25 µL of 2,000 µg/mL (2,000,000 µg/L) in 10 mL MeOH for a 5 µg/mL (5,000 µg/L) working stock (WS) solution.

Table 7. ICAL, Internal, and Surrogate Standard Preparation for the StratUm/AQUATek 70

Method 524.2 - ICAL and Fortification Standard Preparation

Using the StratUm and AQUATek 70

Final concentration	Volume of WS	Volume of water
0.5 µg/L	5 µL [1]	200 mL
1 µg/L	5 µL [1]	100 mL
5 µg/L	25 µL [1]	100 mL
10 µg/L	50 µL [1]	100 mL
25 µg/L	125 µL [1]	100 mL
50 µg/L	250 µL [1]	100 mL
Fortification standard		
1.0 µg/L	2 µL using AQUATek 70 [2]	25 mL
5.0 µg/L	2 µL using AQUATek 70 [2]	5 mL

AccuStandard p/n M-502-10X	Method 502.2 volatile organic compounds (60 compounds) at a concentration of 2,000 µg/mL (2,000,000 µg/L).
AccuStandard p/n M-524R-B	Additions to Method 524.2 (24 compounds) at a concentration of 2,000 µg/mL (2,000,000 µg/L).
AccuStandard p/n M-524-FS	Method 524.2 fortification stock is at a concentration of 2,000 µg/mL (2,000,000 µg/L).

[1] Combine and dilute the stock target solution EACH 100 µL of 2,000 µg/mL (2,000,000 µg/L) in 10 mL MeOH for a 20 µg/mL (20,000 µg/L) working stock (WS) solution.

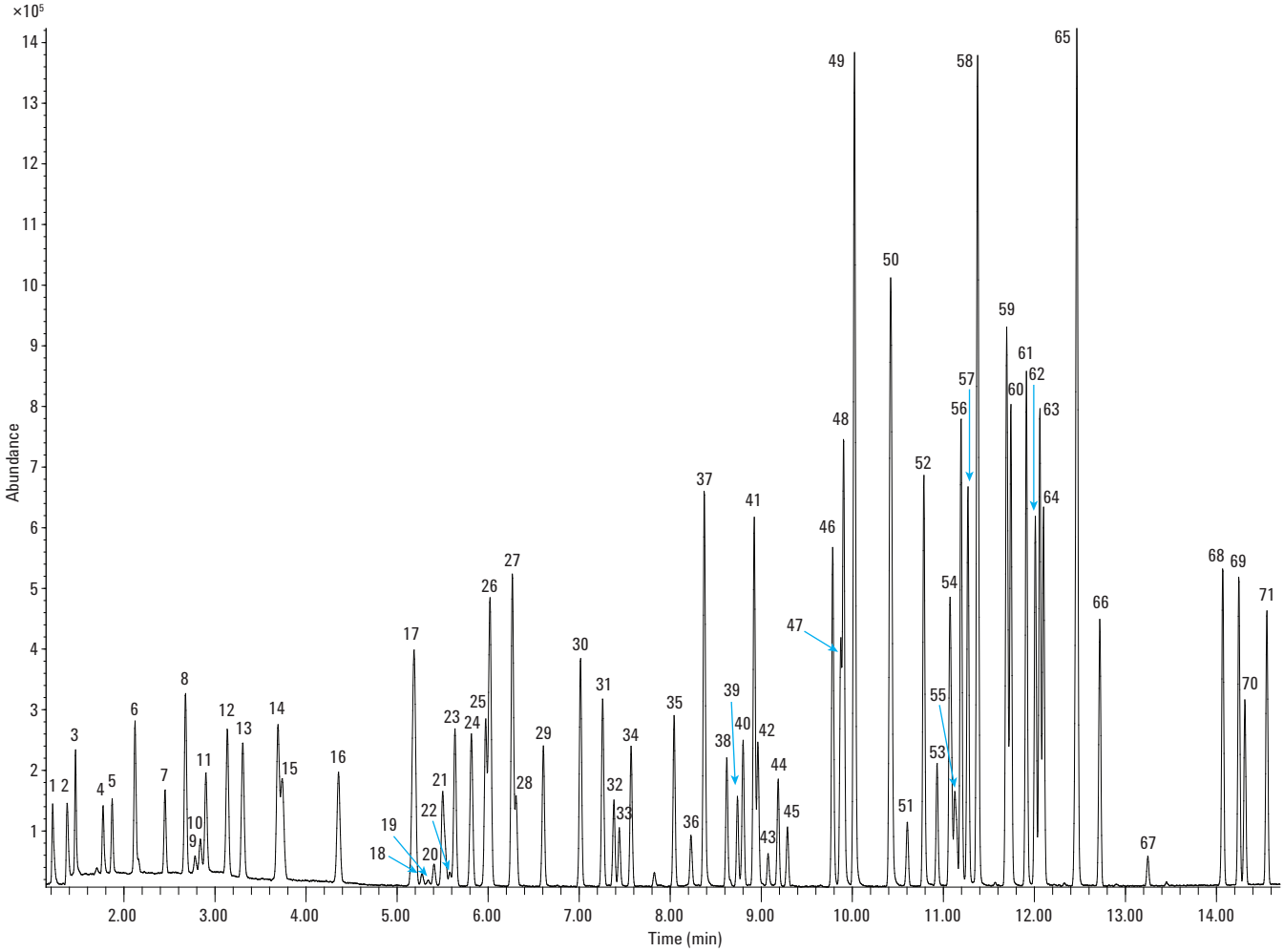
[2] Dilute stock fortification standard 125 µL of 2,000 µg/mL (2,000,000 µg/L) in 20 mL MeOH for a 12.5 µg/mL (12,500 µg/L) working stock (WS) soluton.

Results and Discussion

After a successful BFB injection and spectral evaluation, an ICAL was performed over the specified working range, which is typically from 0.5 – 50 µg/L.

Figure 6 shows a typical chromatogram (TIC) acquired using the GC, MSD, and P&T parameters specified in this analysis. It is important to note that the Gain Factor was used to appropriately scale the dynamic range of the MSD prior to ICAL data acquisition [8]. Typical ICAL results generated using System 1 (Atomx PTC) are given in Table 8 over a concentration range from 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. Although Average Relative Response Factor curve fits are preferred, typically the error involved at low levels of concentration requires linear or quadric curve fits and indicates low response, analyte carryover, or contamination. The capability of extending the quantitation to these lower concentrations indicates the lack of these problems. The results in Table 8 demonstrate that all compounds met the criteria of less than 20% RSD and in most cases are less than 10% RSD. The dilution series for the multilevel ICAL were prepared by the Atomx from the 25 µg/L standard for the 0.25 µg/L level and 50 µg/L standard for all others. This shows the superb dilution capability of the Atomx. The %RSDs for the internal standard and surrogate compounds as introduced by the Atomx PTC are <5% RSD as 5 µL delivered.

The accuracy and precision of most analyses, including VOC analysis, depend upon the success of method implementation, especially at low calibration levels. Both Method 524.2 and 8260B allow for degrees of variance specific to ICAL. Traditionally, calibration curve fit routines specified in a method use an Average Relative Response Factor fit and evaluate %RSD to determine if the linearity for a specific target compound is acceptable. Method 524.2 specifies %RSDs to be 20% or less and Method 8260B specifies %RSDs to be 15% or less if Average Relative Response Factors are used. Both methods allow linear and quadratic curve fits, however, due to the potential error in accuracy, these curve fit routines are avoided as much as possible. The precision of this approach is unparalleled for ICAL curves for the target analytes. Additionally, the precision of the Internal Standard and Surrogate compounds in Method 524.2 demonstrate the high degree of precision achievable with the GC/MS and the P&T instrumentation. It is worth noting that the accuracy and precision when using the Atomx to semi-automatically prepare the ICAL standards and dispense the Internal Standard and Surrogate compounds are very good and can be seen in the %RSD values for both compound types.



1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. Diethyl Ether
8. 1,1-Dichloroethene
9. Acetone
10. Iodomethane
11. Carbon disulfide
12. Allyl chloride
13. Methylene chloride
14. Acrylonitrile, *trans*-1,2-dichloroethene
15. Methyl *tert*-butyl ether (MTBE)
16. 1,1-Dichloroethane
17. 2,2-Dichloropropane, *cis*-1,2-Dichloroethene
18. 2-Butanone (MEK)
19. Propionitrile
20. Methyl acrylate
21. Bromochloromethane, Methacrylonitrile
22. THF
23. Chloroform
24. 1,1,1-Trichloroethane
25. 1-Chlorobutane
26. Carbon tetrachloride, 1,1-Dichloro-1-propene
27. Benzene
28. 1,2-Dichloroethane
29. Fluorobenzene
30. Trichloroethene
31. 1,2-Dichloropropane
32. Dibromomethane
33. Methyl methacrylate
34. Bromodichloromethane
35. *cis*-1,3-Dichloropropene
36. 1,1-Dichloropropanone, 2-Nitropropane, 4-methyl-2-pentanone (MIBK)
37. Toluene
38. *trans*-1,3-Dichloropropene
39. Ethyl methacrylate
40. 1,1,2-Trichloroethane
41. Tetrachloroethene
42. 1,3-Dichloropropane
43. 2-Hexanone
44. Dibromochloromethane
45. 1,2-Dibromoethane
46. Chlorobenzene
47. 1,1,1,2-Tetrachloroethane
48. Ethylbenzene
49. m+p-Xylene
50. o-Xylene, Styrene
51. Bromoform
52. Isopropylbenzene
53. Bromofluorobenzene
54. Bromobenzene, 1,1,2,2-Tetrachloroethane
55. 1,2,3-Trichloropropane, *trans*-1,4-Dichloro-2-butene
56. n-Propylbenzene
57. 2-Chlorotoluene
58. 1,3,5-Trimethylbenzene, 4-Chlorotoluene
59. *tert*-Butylbenzene
60. 1,2,4-Trimethylbenzene
61. *sec*-Butylbenzene
62. 1,3-Dichlorobenzene
63. p-Isoproyltoluene
64. 1,4-Dichlorobenzene
65. 1,2-Dichlorobenzene-d4, 1,2-Dichlorobenzene, n-Butylbenzene
66. Hexachloroethane
67. 1,2-Dibromo-3-chloropropane (DBCP)
68. 1,2,4-Trichlorobenzene
69. Hexachlorobutadiene
70. Naphthalene
71. 1,2,3-Trichlorobenzene

Figure 6. Total Ion Chromatogram of Method 524.2 ICAL standard.

Table 8. ICAL for Method 524.2 from 0.25 – 50 µg/L using the Atomx (continued)

Method 524.2 ICAL	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)	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	1.000	4.0
Dichlorodifluoromethane	0.097	0.105	0.098	0.080	0.081	0.080	0.089	0.090	11.3
Chloromethane	0.167	0.176	0.183	0.153	0.149	0.139	0.147	0.159	10.4
Vinyl chloride	0.174	0.150	0.153	0.142	0.146	0.146	0.162	0.153	7.3
Bromomethane	0.063	0.073	0.070	0.056	0.063	0.064	0.075	0.066	9.8
Chloroethane	0.090	0.081	0.084	0.078	0.081	0.078	0.085	0.083	5.0
Trichlorofluoromethane	0.185	0.189	0.180	0.151	0.156	0.155	0.170	0.169	9.2
Diethyl Ether	0.084	0.078	0.078	0.066	0.070	0.068	0.073	0.074	8.6
1,1-Dichloroethene	0.203	0.182	0.191	0.164	0.172	0.170	0.186	0.181	7.5
Acetone				0.021	0.019	0.015	0.016	0.018	14.0
Iodomethane	0.051	0.059	0.066	0.077	0.079	0.067	0.064	0.066	15.0
Carbon disulfide	0.319	0.271	0.281	0.253	0.274	0.276	0.303	0.282	7.7
Allyl chloride	0.212	0.193	0.202	0.187	0.196	0.192	0.201	0.198	4.2
Methylene chloride	0.188	0.156	0.157	0.143	0.150	0.145	0.156	0.157	9.5
trans-1,2-Dichloroethene	0.185	0.164	0.165	0.152	0.160	0.159	0.179	0.166	7.0
Methyl tert-butyl ether (MTBE)	0.300	0.267	0.247	0.214	0.225	0.219	0.239	0.244	12.5
1,1-Dichloroethane	0.243	0.199	0.217	0.212	0.221	0.214	0.233	0.220	6.6
2,2-Dichloropropane	0.192	0.177	0.176	0.150	0.153	0.147	0.157	0.165	10.4
cis-1,2-Dichloroethene	0.192	0.196	0.210	0.192	0.199	0.198	0.214	0.200	4.3
2-Butanone (MEK)				0.028	0.026	0.024	0.025	0.025	6.9
Bromochloromethane	0.090	0.085	0.095	0.079	0.084	0.081	0.085	0.086	6.2
Chloroform	0.195	0.196	0.201	0.196	0.207	0.202	0.221	0.202	4.4
1,1,1-Trichloroethane	0.187	0.171	0.183	0.171	0.178	0.175	0.192	0.180	4.5
1-Chlorobutane	0.274	0.262	0.254	0.231	0.244	0.242	0.264	0.253	5.9
Carbon Tetrachloride	0.162	0.157	0.169	0.146	0.153	0.153	0.170	0.158	5.5
1,1-Dichloro-1-propene	0.181	0.180	0.172	0.149	0.158	0.158	0.174	0.167	7.4
Benzene	0.509	0.483	0.502	0.467	0.488	0.482	0.528	0.494	4.1
1,2-Dichloroethane	0.127	0.119	0.124	0.109	0.112	0.112	0.120	0.118	5.7
Trichloroethene	0.152	0.132	0.131	0.118	0.122	0.122	0.134	0.130	8.7
1,2-Dichloropropane	0.154	0.128	0.129	0.117	0.124	0.121	0.130	0.129	9.3
Dibromomethane	0.048	0.049	0.049	0.046	0.050	0.048	0.052	0.049	3.8
Bromodichloromethane	0.152	0.141	0.148	0.138	0.151	0.145	0.159	0.148	4.8
cis-1,3-Dichloropropene	0.191	0.182	0.170	0.156	0.168	0.165	0.178	0.173	6.8
4-methyl-2-pentanone (MIBK)				0.074	0.078	0.076	0.081	0.077	4.0
Toluene	0.584	0.528	0.530	0.482	0.510	0.504	0.554	0.527	6.4
trans-1,3-Dichloropropene	0.150	0.133	0.131	0.115	0.120	0.120	0.128	0.128	9.2
1,1,2-Trichloroethane	0.089	0.089	0.079	0.067	0.071	0.068	0.071	0.076	12.4
Tetrachloroethene	0.135	0.176	0.159	0.123	0.130	0.127	0.144	0.142	13.6
1,3-Dichloropropane	0.148	0.145	0.132	0.118	0.124	0.122	0.131	0.132	8.8
2-Hexanone				0.041	0.041	0.039	0.042	0.041	2.5
Dibromochloromethane	0.104	0.098	0.096	0.084	0.091	0.091	0.098	0.094	6.8
1,2-Dibromoethane (EDB)	0.084	0.081	0.073	0.060	0.064	0.063	0.066	0.070	13.6
Chlorobenzene	0.334	0.303	0.319	0.285	0.305	0.301	0.328	0.311	5.6
1,1,1,2-Tetrachloroethane	0.128	0.107	0.107	0.103	0.108	0.106	0.115	0.111	7.8
Ethylbenzene	0.618	0.577	0.577	0.519	0.544	0.549	0.611	0.571	6.3
m+p-Xylene	0.475	0.455	0.435	0.391	0.412	0.418	0.467	0.436	7.2
o-Xylene	0.444	0.435	0.435	0.399	0.418	0.415	0.461	0.430	4.8
Styrene	0.337	0.327	0.332	0.308	0.326	0.327	0.361	0.331	4.8
Bromoform	0.059	0.061	0.053	0.046	0.049	0.048	0.052	0.052	10.6
Isopropylbenzene	0.532	0.494	0.492	0.434	0.448	0.451	0.498	0.479	7.3

Table 8. ICAL for Method 524.2 from 0.25 – 50 µg/L using the Atomx (continued)

Method 524.2 ICAL	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
Bromofluorobenzene (SUR)	0.323	0.332	0.323	0.319	0.332	0.330	0.348	0.330	2.9
Bromobenzene	0.195	0.209	0.233	0.196	0.214	0.211	0.221	0.211	6.3
1,1,2,2-Tetrachloroethane	0.098	0.119	0.102	0.093	0.093	0.081	0.080	0.095	14.0
1,2,3-Trichloropropane	0.031	0.025	0.026	0.020	0.021	0.019	0.020	0.023	18.3
n-Propylbenzene	0.405	0.389	0.378	0.343	0.359	0.357	0.392	0.375	6.0
2-Chlorotoluene	0.409	0.397	0.378	0.343	0.359	0.357	0.392	0.377	6.5
1,3,5-Trimethylbenzene	0.535	0.476	0.460	0.411	0.432	0.431	0.478	0.460	9.0
4-Chlorotoluene	0.479	0.472	0.432	0.381	0.405	0.406	0.450	0.432	8.6
tert-Butylbenzene	0.518	0.470	0.450	0.395	0.419	0.415	0.455	0.446	9.2
1,2,4-Trimethylbenzene	0.498	0.484	0.455	0.403	0.424	0.427	0.472	0.452	7.7
sec-Butylbenzene	0.701	0.681	0.617	0.517	0.549	0.551	0.612	0.604	11.5
1,3-Dichlorobenzene	0.270	0.255	0.244	0.212	0.226	0.223	0.244	0.239	8.4
p-Isopropyltoluene	0.573	0.520	0.477	0.400	0.424	0.430	0.478	0.472	12.7
1,4-Dichlorobenzene	0.259	0.253	0.247	0.208	0.221	0.221	0.242	0.236	8.1
1,2-Dichlorobenzene-d4 (SURR)	0.290	0.275	0.287	0.280	0.288	0.289	0.303	0.287	3.1
1,2-Dichlorobenzene	0.225	0.229	0.213	0.183	0.194	0.192	0.210	0.207	8.5
n-Butylbenzene	0.569	0.540	0.496	0.407	0.430	0.437	0.495	0.482	12.5
Hexachloroethane	0.107	0.104	0.093	0.082	0.087	0.089	0.099	0.095	9.8
1,2-Dibromo-3-chloropropane (DBCP)	0.014	0.015	0.014	0.013	0.013	0.011	0.011	0.013	12.0
1,2,4-Trichlorobenzene	0.180	0.179	0.161	0.132	0.144	0.146	0.162	0.158	11.5
Hexachlorobutadiene	0.126	0.118	0.100	0.089	0.088	0.088	0.098	0.101	15.3
Naphthalene	0.305	0.280	0.262	0.210	0.227	0.229	0.250	0.252	13.1
1,2,3-Trichlorobenzene	0.154	0.140	0.139	0.115	0.124	0.123	0.137	0.133	10.0

Typical reporting limits for most compounds are 0.5 µg/L. However, there are some compounds that have higher reporting limits. For example, the ketones, such as acetone, have reporting limits at 5.0 µg/L. There are regional differences for reporting limits, but generally the limits cited are listed in Table 8.

Figure 7 illustrates an EIC of the gases. The relationship between bromomethane and chloroethane is important. Chloroethane should be present in the EIC abundance (height) at 75% or more, relative to bromomethane. Deficient chloroethane indicates sample pathway or trap activity. Poor water management in the GC and P&T systems manifests itself in the process of dehydrohalogenation. This was reflected in the high %RSD values or highly nonlinear curve fit (R^2 <0.98) obtained by an ICAL and MDL study for 1,1,2,2-tetrachloroethane, tetrachloroethene, trichloroethene, and other VOC's that are susceptible to dehydrohalogenation.

An MDL study was performed after demonstrating acceptable linearity of greater than two orders of magnitude for every target analyte using an Average Relative Response curve fit (all %RSD are < 20%). Seven 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.

Equation 1. Formula for MDL Calculations

$$MDL = s \times t_{(n-1,\ 1\text{-alpha} = 99)} = s \times 3.143$$

Where:

(n-1, 1-alpha) = t value for the 99% confidence level with n-1 degrees of freedom

n = number of trials

s = standard deviation of the seven trials

Table 9 lists the calculated MDLs for Method 524.2, which are typical using the conditions of this analysis. As the low ppt levels indicate, this method has the potential for lower levels of detection.

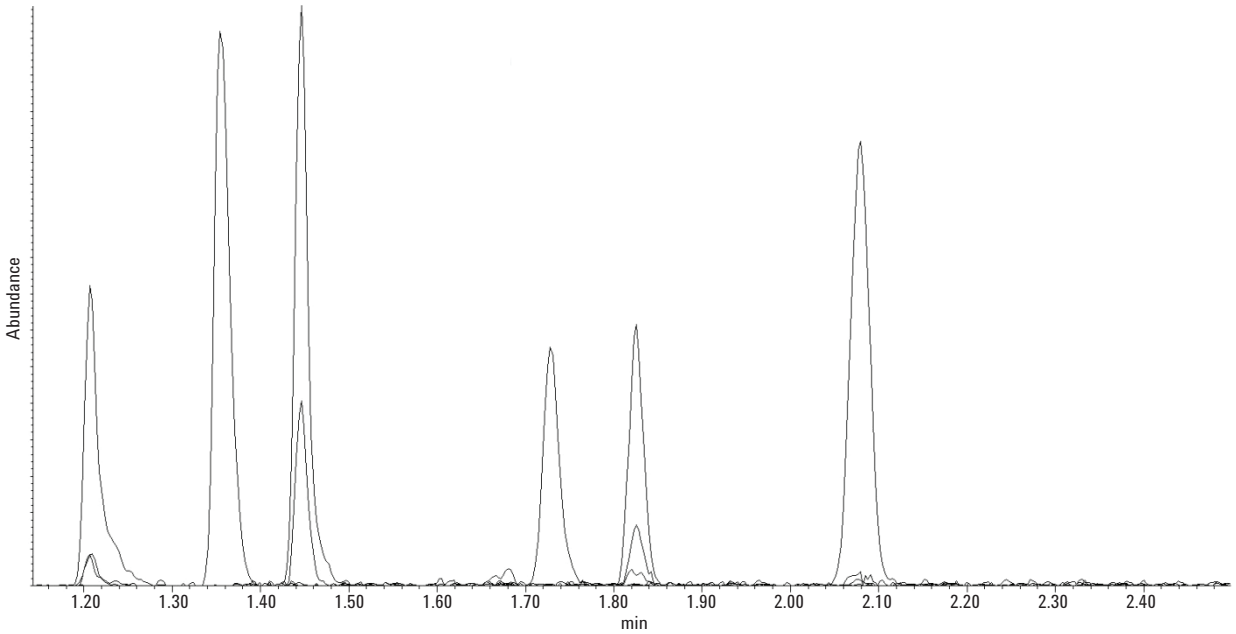


Figure 7. Extracted Ion Chromatogram of the gases from left to right: dichlorodifluoromethane (85 m/z), chloromethane (50 m/z), vinyl chloride (62 m/z), bromomethane (94 m/z), chloroethane (64 m/z), and trichlorofluoromethane (101 m/z) in order of their elution.

Table 9. Calculated MDLs at 0.25 µg/L for Method 524.2 using the Atomx (continued)

Method 524.2 MDL study	Spike µg/L	MDL 1 µg/L	MDL 2 µg/L	MDL 3 µg/L	MDL 4 µg/L	MDL 5 µg/L	MDL 6 µg/L	MDL 7 µg/L	Avg µg/L	SD µg/L	MDL µg/L
Fluorobenzene (ISTD)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	0.000	N/A
Dichlorodifluoromethane	0.25	0.31	0.32	0.28	0.29	0.25	0.26	0.29	0.29	0.025	0.08
Chloromethane	0.25	0.29	0.31	0.28	0.28	0.28	0.27	0.29	0.29	0.013	0.04
Vinyl chloride	0.25	0.23	0.25	0.28	0.28	0.27	0.27	0.25	0.26	0.019	0.06
Bromomethane	0.25	0.30	0.28	0.27	0.27	0.29	0.30	0.32	0.29	0.018	0.06
Chloroethane	0.25	0.23	0.23	0.24	0.24	0.23	0.23	0.28	0.24	0.018	0.06
Trichlorofluoromethane	0.25	0.25	0.28	0.29	0.27	0.28	0.25	0.28	0.27	0.016	0.05
Diethyl Ether	0.25	0.29	0.26	0.29	0.29	0.28	0.29	0.28	0.28	0.011	0.03
1,1-Dichloroethene	0.25	0.25	0.30	0.29	0.30	0.27	0.28	0.29	0.28	0.018	0.06
Acetone	2.00	2.61	2.94	2.90	3.00	2.93	2.76	2.96	2.87	0.138	0.43
Iodomethane	0.25	0.28	0.20	0.27	0.30	0.26	0.17	0.23	0.24	0.046	0.15
Carbon disulfide	0.25	0.25	0.25	0.27	0.26	0.24	0.25	0.27	0.26	0.011	0.04
Allyl chloride	0.25	0.25	0.24	0.28	0.26	0.25	0.27	0.28	0.26	0.016	0.05
Methylene chloride	0.25	0.28	0.28	0.31	0.28	0.27	0.33	0.30	0.29	0.021	0.07
trans-1,2-Dichloroethene	0.25	0.31	0.31	0.29	0.26	0.31	0.27	0.27	0.29	0.022	0.07
Methyl tert-butyl ether (MTBE)	0.25	0.27	0.26	0.28	0.28	0.27	0.28	0.26	0.27	0.009	0.03
1,1-Dichloroethane	0.25	0.25	0.24	0.26	0.27	0.23	0.21	0.25	0.24	0.020	0.06
2,2-Dichloropropane	0.25	0.20	0.07	0.20	0.18	0.22	0.20	0.21	0.18	0.051	0.16
cis-1,2-Dichloroethene	0.25	0.28	0.26	0.28	0.30	0.30	0.26	0.31	0.28	0.020	0.06
2-Butanone (MEK)	1.00	0.92	1.06	0.88	1.01	0.97	1.02	1.06	0.99	0.069	0.22
Bromochloromethane	0.25	0.29	0.30	0.28	0.33	0.28	0.29	0.32	0.30	0.020	0.06
Chloroform	0.25	0.22	0.22	0.23	0.25	0.23	0.24	0.25	0.23	0.013	0.04
1,1,1-Trichloroethane	0.25	0.23	0.24	0.27	0.25	0.24	0.23	0.27	0.25	0.017	0.05
1-Chlorobutane	0.25	0.28	0.28	0.26	0.29	0.28	0.25	0.29	0.28	0.015	0.05
Carbon tetrachloride	0.25	0.22	0.24	0.24	0.25	0.21	0.22	0.24	0.23	0.015	0.05
1,1-Dichloro-1-propene	0.25	0.29	0.29	0.29	0.30	0.28	0.28	0.24	0.28	0.020	0.06
Benzene	0.25	0.28	0.28	0.29	0.30	0.29	0.28	0.28	0.29	0.008	0.02
1,2-Dichloroethane	0.25	0.29	0.31	0.27	0.31	0.25	0.32	0.28	0.29	0.025	0.08
Trichloroethene	0.25	0.23	0.25	0.27	0.25	0.26	0.25	0.24	0.25	0.013	0.04
1,2-Dichloropropane	0.25	0.37	0.39	0.37	0.31	0.32	0.33	0.30	0.34	0.035	0.11
Dibromomethane	0.25	0.23	0.25	0.23	0.28	0.23	0.25	0.26	0.25	0.019	0.06
Bromodichloromethane	0.25	0.23	0.22	0.23	0.25	0.22	0.23	0.23	0.23	0.010	0.03
cis-1,3-Dichloropropene	0.25	0.24	0.26	0.24	0.26	0.23	0.24	0.26	0.25	0.013	0.04
4-methyl-2-pentanone (MIBK)	0.25	0.25	0.34	0.28	0.28	0.38	0.31	0.38	0.32	0.051	0.16
Toluene	0.25	0.25	0.24	0.24	0.26	0.24	0.24	0.24	0.24	0.008	0.02
trans-1,3-Dichloropropene	0.25	0.26	0.27	0.23	0.27	0.27	0.22	0.24	0.25	0.021	0.07
1,1,2-Trichloroethane	0.25	0.34	0.35	0.35	0.39	0.35	0.35	0.35	0.35	0.016	0.05
Tetrachloroethene	0.25	0.28	0.30	0.30	0.33	0.29	0.42	0.31	0.32	0.047	0.15
1,3-Dichloropropane	0.25	0.26	0.30	0.29	0.30	0.29	0.29	0.30	0.29	0.014	0.04
2-Hexanone	1.00	0.79	0.84	0.84	0.87	0.81	0.75	0.79	0.81	0.040	0.13
Dibromochloromethane	0.25	0.25	0.26	0.25	0.28	0.24	0.25	0.24	0.25	0.014	0.04
1,2-Dibromoethane (EDB)	0.25	0.25	0.32	0.27	0.30	0.33	0.26	0.32	0.29	0.033	0.10
Chlorobenzene	0.25	0.23	0.25	0.24	0.25	0.24	0.24	0.24	0.24	0.007	0.02
1,1,1,2-Tetrachloroethane	0.25	0.23	0.23	0.26	0.26	0.22	0.24	0.24	0.24	0.015	0.05
Ethylbenzene	0.25	0.24	0.25	0.24	0.25	0.24	0.23	0.27	0.25	0.013	0.04
m+p-Xylene	0.50	0.48	0.49	0.49	0.53	0.50	0.49	0.50	0.50	0.016	0.05
o-Xylene	0.25	0.26	0.25	0.24	0.26	0.24	0.24	0.24	0.25	0.010	0.03
Styrene	0.25	0.37	0.38	0.37	0.40	0.36	0.35	0.37	0.37	0.016	0.05
Bromoform	0.25	0.25	0.27	0.31	0.27	0.29	0.26	0.30	0.28	0.022	0.07
Isopropylbenzene	0.25	0.23	0.25	0.26	0.28	0.26	0.26	0.28	0.26	0.017	0.05
Bromofluorobenzene (SUR)	1.00	1.00	1.03	1.03	1.05	1.03	1.05	1.05	1.03	0.018	0.06
Bromobenzene	0.25	0.27	0.31	0.29	0.28	0.29	0.29	0.30	0.29	0.013	0.04
1,1,2,2-Tetrachloroethane	0.25	0.28	0.29	0.32	0.31	0.30	0.29	0.28	0.30	0.015	0.05
1,2,3-Trichloropropane	0.25	0.28	0.32	0.35	0.22	0.39	0.31	0.32	0.31	0.053	0.17
n-Propylbenzene	0.25	0.27	0.29	0.29	0.28	0.27	0.26	0.27	0.28	0.011	0.04
2-Chlorotoluene	0.25	0.27	0.28	0.28	0.28	0.27	0.26	0.27	0.27	0.008	0.02
1,3,5-Trimethylbenzene	0.25	0.26	0.26	0.25	0.26	0.25	0.26	0.27	0.26	0.007	0.02
4-Chlorotoluene	0.25	0.25	0.26	0.26	0.29	0.25	0.26	0.27	0.26	0.014	0.04
tert-Butylbenzene	0.25	0.25	0.25	0.25	0.28	0.24	0.23	0.25	0.25	0.015	0.05

Table 9. Calculated MDLs at 0.25 µg/L for Method 524.2 using the Atomx (continued)

Method 524.2 MDL study	Spike µg/L	MDL 1 µg/L	MDL 2 µg/L	MDL 3 µg/L	MDL 4 µg/L	MDL 5 µg/L	MDL 6 µg/L	MDL 7 µg/L	Avg µg/L	SD µg/L	MDL µg/L
1,2,4-Trimethylbenzene	0.25	0.25	0.25	0.26	0.27	0.26	0.25	0.27	0.26	0.009	0.03
sec-Butylbenzene	0.25	0.27	0.28	0.27	0.29	0.28	0.26	0.29	0.28	0.011	0.03
1,3-Dichlorobenzene	0.25	0.27	0.26	0.26	0.27	0.27	0.23	0.28	0.26	0.016	0.05
p-Isopropyltoluene	0.25	0.27	0.28	0.27	0.28	0.27	0.27	0.27	0.27	0.005	0.02
1,4-Dichlorobenzene	0.25	0.25	0.27	0.25	0.28	0.27	0.25	0.27	0.26	0.013	0.04
1,2-Dichlorobenzene-d4 (SURR)	1.00	1.09	1.13	1.08	1.09	1.11	1.10	1.09	1.10	0.017	0.05
1,2-Dichlorobenzene	0.25	0.28	0.31	0.29	0.31	0.30	0.29	0.31	0.30	0.012	0.04
n-Butylbenzene	0.25	0.28	0.28	0.28	0.30	0.29	0.26	0.31	0.29	0.016	0.05
Hexachloroethane	0.25	0.25	0.25	0.26	0.24	0.25	0.24	0.25	0.25	0.007	0.02
1,2-Dibromo-3-chloropropane (DBCP)	0.25	0.24	0.26	0.29	0.36	0.27	0.30	0.32	0.29	0.040	0.13
1,2,4-Trichlorobenzene	0.25	0.29	0.29	0.26	0.29	0.29	0.27	0.28	0.28	0.012	0.04
Hexachlorobutadiene	0.25	0.33	0.28	0.27	0.30	0.30	0.28	0.30	0.29	0.020	0.06
Naphthalene	0.25	0.33	0.33	0.31	0.33	0.30	0.31	0.31	0.32	0.013	0.04
1,2,3-Trichlorobenzene	0.25	0.29	0.28	0.27	0.29	0.27	0.27	0.30	0.28	0.012	0.04

As regulations continue to restrict VOCs, lower detection levels are becoming necessary. Therefore, calibrations at levels below the standard 0.5 or 0.25 µg/L level are necessary, while maintaining relatively higher levels in the calibration range. Throughout the history of the MSD, successfully achieving and maintaining a calibration range of three orders of magnitude has been a challenge, due to target tuning the MSD. Calibration across nontraditional three orders of magnitude is now achievable with the BFB Autotune. Table 10 shows a typical example of ICAL results. This example uses four specific groups of VOCs:

- **Trihalomethanes (THMs**) – These classic VOCs are used for municipal ground water monitoring. Levels of disinfectants and their byproducts are targeted because of their potential impact on the groundwater.
- **Gases** – Analyses for VOCs in gases are challenging due to their volatility and chromatographic properties.
- **Aromatics** –Aromatics typically do not present a problem of detection at low ppt levels because of the intense molecular ions in their spectra. The challenge is to be able to detect extremely low levels of nonaromatics without reaching the point of signal saturation.
- **Dehydrohalogenation** – These compounds indicate inefficient or poor water management with P&T.

Table 10 shows data from an ICAL ranging from 0.1–100 µg/L, which is a full three orders of magnitude. All compounds demonstrate good linearity using an Average Relative Response Factor curve fit. All %RSDs are below the method requirement of <20%; therefore linear and quadratic curve fitting are not required. Water management was proven sufficient since there is minimal evidence of dehydrohalogenation. Methyl tert butyl ether (MTBE), an oxygenate added to gasoline, demonstrated acceptable performance. All %RSDs were less than 20%, indicating that there was no signal saturation. All four THMs demonstrated superb linear performance. The %RSD of 7.7% for the ISTD fluorobenzene illustrates its exceptional performance.

The accompanying MDL study was conducted following the same protocol. The results of this MDL study conducted at 0.1 µg/L are shown in Table 11.

Table 10. ICAL for Method 524.2 from 0.1 – 100 µg/L using the Atomx

Method 524.2 ICAL selected compounds	0.1 µg/L RRF	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	100 µg/L RRF	Avg RRF	%RSD
Fluorobenzene (ISTD)	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	ISTD	1.000	7.7
Vinyl chloride	0.189	0.174	0.150	0.153	0.142	0.146	0.146	0.162	0.149	0.157	10.0
Chloroethane	0.108	0.090	0.081	0.084	0.078	0.081	0.078	0.085	0.075	0.085	11.6
Allyl chloride	0.249	0.212	0.193	0.202	0.187	0.196	0.192	0.201	0.182	0.202	9.8
Methyl tert-butylether (MTBE)	0.334	0.300	0.267	0.247	0.214	0.225	0.219	0.239	0.211	0.250	16.9
Chloroform	0.241	0.195	0.196	0.201	0.196	0.207	0.202	0.221	0.207	0.207	7.2
1,1,1-Trichloroethane	0.210	0.187	0.171	0.183	0.171	0.178	0.175	0.192	0.172	0.182	7.1
Carbon tetrachloride	0.170	0.162	0.157	0.169	0.146	0.153	0.153	0.170	0.149	0.159	5.8
Benzene	0.724	0.509	0.483	0.502	0.467	0.488	0.482	0.528	0.570	0.528	15.1
Trichloroethene	0.156	0.152	0.132	0.131	0.118	0.122	0.122	0.134	0.116	0.131	10.8
Dibromomethane	0.056	0.048	0.049	0.049	0.046	0.050	0.048	0.052	0.046	0.049	6.5
Bromodichloromethane	0.168	0.152	0.141	0.148	0.138	0.151	0.145	0.159	0.148	0.150	6.2
Toluene	0.660	0.584	0.528	0.530	0.482	0.510	0.504	0.554	0.502	0.539	10.1
Tetrachloroethene	0.217	0.148	0.158	0.159	0.142	0.130	0.127	0.144	0.170	0.155	17.5
Dibromochloromethane	0.126	0.104	0.098	0.096	0.084	0.091	0.091	0.098	0.091	0.098	12.3
Chlorobenzene	0.343	0.334	0.303	0.319	0.285	0.305	0.301	0.328	0.295	0.313	6.3
1,1,1,2-Tetrachloroethane	0.130	0.128	0.107	0.107	0.103	0.108	0.106	0.115	0.106	0.112	8.9
Ethylbenzene	0.632	0.618	0.577	0.577	0.519	0.544	0.549	0.611	0.553	0.575	6.6
m+p-Xylene	0.502	0.475	0.455	0.435	0.391	0.412	0.418	0.467	0.405	0.440	8.4
o-Xylene	0.528	0.444	0.435	0.435	0.399	0.418	0.415	0.461	0.419	0.440	8.6
Bromoform	0.070	0.059	0.061	0.053	0.046	0.049	0.048	0.052	0.048	0.054	14.5
Bromofluorobenzene (SURR)	0.351	0.323	0.332	0.323	0.319	0.332	0.330	0.348	0.367	0.336	4.7
1,1,2,2-Tetrachloroethane	0.131	0.098	0.119	0.102	0.093	0.093	0.081	0.080	0.085	0.098	17.7
1,2-Dichlorobenzene-d4 (SURR)	0.326	0.29	0.275	0.287	0.28	0.288	0.289	0.303	0.351	0.299	8.3

Table 11. Calculated MDLs at 0.1 µg/L for Method 524.2 using the Atomx

Method 524.2 MDL study selected compounds	Spike µg/L	MDL 1 µg/L	MDL 2 µg/L	MDL 3 µg/L	MDL 4 µg/L	MDL 5 µg/L	MDL 6 µg/L	MDL 7 µg/L	Avg µg/L	SD µg/L	MDL µg/L
Fluorobenzene (ISTD)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00		
Vinyl chloride	0.10	0.13	0.12	0.14	0.09	0.12	0.10	0.11	0.12	0.017	0.05
Chloroethane	0.10	0.10	0.13	0.11	0.07	0.11	0.16	0.13	0.12	0.028	0.09
Allyl chloride	0.10	0.15	0.12	0.10	0.11	0.11	0.12	0.14	0.12	0.018	0.06
Methyl *tert*-butyl ether (MTBE)	0.10	0.13	0.13	0.13	0.07	0.12	0.11	0.11	0.11	0.021	0.07
Chloroform	0.10	0.09	0.12	0.09	0.10	0.11	0.11	0.10	0.10	0.011	0.03
1,1,1-Trichloroethane	0.10	0.13	0.12	0.14	0.10	0.11	0.11	0.12	0.12	0.013	0.04
Carbon tetrachloride	0.10	0.11	0.11	0.12	0.08	0.11	0.10	0.10	0.10	0.013	0.04
Benzene	0.10	0.13	0.14	0.14	0.13	0.12	0.12	0.14	0.13	0.009	0.03
Trichloroethene	0.10	0.12	0.12	0.13	0.10	0.12	0.12	0.11	0.12	0.010	0.03
Dibromomethane	0.10	0.11	0.11	0.12	0.11	0.09	0.09	0.09	0.10	0.013	0.04
Bromodichloromethane	0.10	0.12	0.11	0.11	0.11	0.10	0.08	0.11	0.11	0.013	0.04
Toluene	0.10	0.11	0.12	0.13	0.11	0.11	0.11	0.12	0.12	0.008	0.02
Tetrachloroethene	0.10	0.12	0.14	0.14	0.12	0.15	0.14	0.14	0.14	0.011	0.04
Dibromochloromethane	0.10	0.12	0.12	0.13	0.10	0.13	0.12	0.12	0.12	0.010	0.03
Chlorobenzene	0.10	0.11	0.11	0.11	0.11	0.10	0.10	0.11	0.11	0.005	0.02
1,1,1,2-Tetrachloroethane	0.10	0.11	0.12	0.13	0.09	0.11	0.13	0.09	0.11	0.017	0.05
Ethyl benzene	0.10	0.11	0.11	0.12	0.11	0.12	0.12	0.12	0.12	0.005	0.02
m+p-Xylene	0.20	0.20	0.20	0.22	0.19	0.20	0.21	0.20	0.20	0.010	0.03
o-Xylene	0.10	0.11	0.12	0.12	0.11	0.10	0.12	0.12	0.11	0.008	0.02
Bromoform	0.10	0.11	0.13	0.11	0.11	0.10	0.13	0.12	0.12	0.011	0.04
Bromofluorobenzene (SURR)	1.00	1.07	1.05	1.01	1.05	1.05	1.05	1.03	1.04	0.019	0.06
1,1,2,2-Tetrachloroethane	0.10	0.12	0.13	0.11	0.12	0.13	0.11	0.11	0.12	0.009	0.03
1,2-Dichlorobenzene-d4 (SURR)	1.00	1.09	1.09	1.09	1.08	1.07	1.11	1.09	1.09	0.012	0.04

All calculated MDLs were in the sub-ppt level and approached the upper ppq levels of detection. Approximately every twelve hours, a calibration check is required to verify that the tune and calibration remain valid. This is called continuing calibration verification (CCV). The new BFB Autotune provides a very high degree of stability and some beta sites have run samples for months without retuning or failing the CCV criteria. Appendix J provides an example of such data for a site applying Method 8260B.

Conclusions

Even though it is a standard analysis in environmental laboratories around the world, VOC analysis by GC/MS with P&T continues to evolve. Requirements for lower and lower levels of detection drive this analysis; therefore, new and improved technologies play a significant part in its success. Column and GC inlet liner technologies such as the Ultra Inert [UI] process provide a higher level of stability and robustness. Improvements in MSD tuning give better sensitivity and stability. The BFB Autotune is one of the most significant enhancements to MSD technology since the introduction of the 5971A MSD. BFB Autotuning has improved sensitivity into the ppq range and allows labs to measure stability in weeks or longer, instead of days. Improvements in P&T technology now bring an unparalleled level of both accuracy and precision yielding superb calibrations across an even wider dynamic working range than has been traditionally expected.

However, the fundamentals of VOC analysis should not be forgotton. The correct parameters for all three components: GC, MSD, and P&T are necessary for success. As improvements in technology allow for lower and lower levels of detection, it is imperative that the proven and sound practices demonstrated since the early 1990's continue to be followed. This practice will lead VOC analyses with GC/MS into the twenty-first century.

References

1. R.D. Dandeneau and E.H. Zerenner, *J. High Resolut. Chromatogr*. 2, 351–356 (1979).
2. R.D. Dandeneau and E.H. Zerenner, LCGC 8(12), 908–912 (1990).
3. L.S. Ettre, *The Evolution of Capillary Columns for Gas Chromatography*, LCGC 19(1), 48-59 (2001).
4. J.S. Hollis, *EPA Method 524.2 by capillary direct split mode using the HP5972A Mass Selective Detector* [MSD], HP Application Note 5962-8659E (1993).
5. J.W. Munch, T.A Bellar, J.W. Eichelberger, W.L. Budde, R.W. Slater, Jr., A. Alford-Stevens, *Method 524.2 – Measurement of Purgeable Organic Compounds In Water by Capillary Column Gas Chromatography/Mass Spectrometry – Revision 4.1*.
6. EPA Publication SW-846 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 8260B – *Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)*.
7. *Enhancements to Gain Normalized Instrument Tuning: Understanding the Benefits and Features*, Agilent Technologies, Inc. Technical Note 5989-7654EN (www.chem.agilent.com).
8. *MSD EI and CI Source Cleaning and Installation*, Agilent Technologies, Inc. Technical Note 5989-5974EN (www.chem.agilent.com).

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Appendix A
Agilent GC/MS method conditions for acquiring BFB

Instrument control parameters: Method BFB_524_2.M

GC Acquisition parameters	
Sample inlet	GC
Injection source	External device
Mass spectrometer	Enabled
Equilibration time	0 minutes
Max temperature	260 °C
Slow fan	Disabled
Oven program	On 40 °C for 1 minutes then 25 °C/min to 200 °C for 0.6 minutes Run time is 8 minutes
Front SS inlet	He
Mode	Split
Heater	200 °C
Pressure	14.517 psi
Total flow	110.7 mL/min
Septum purge flow	5 mL/min
Gas saver	Off
Split ratio	150:1
Split flow	105 mL/min
Thermal Aux 2 (MSD transfer line)	
Heater	On
Temperature	250 °C
Column	Agilent J&W 121-1324UI, DB-624UI, 20 m × 180 µm, 1 µm
Max temperature	260 °C
In	Front SS inlet He
Out	Vacuum
Initial temperature	40 °C
Pressure	14.517 psi
Constant flow	0.7 mL/min
Average velocity	37.157 cm/s
Holdup time	0.8971 minutes
Run time	8 minutes

MS Acquisition parameters	
Tune file	BFB_Atune.u
Acquisition mode	Scan
Solvent delay	3.00 minutes
EMV mode	Gain factor
Gain factor	5.00
Scan parameters	
Low mass	35.0
High mass	260.0
Threshold	100
Sample	2 A/D samples 4
MS zones	
MS source	250 °C
MS quad	200 °C
Trace ion detection	ON

Appendix B
Agilent GC/MS method conditions for VOCs

Instrument control parameters: Method 524_2.M

GC Acquisition parameters	
Sample inlet	GC
Injection source	External device
Mass spectrometer	Enabled
Equilibration time	0 minutes
Max temperature	260 °C
Slow fan	Disabled
Oven program	On 35 °C for 4 minutes then 15 °C/min to 240 °C for 0.3333 minutes Run time is 18 minutes
Front SS inlet	He
Mode	Split
Heater	200 °C
Pressure	14.125 psi
Total flow	110.7 mL/min
Septum purge flow	5 mL/min
Gas saver	Off
Split ratio	150:1
Split flow	105 mL/min
Thermal Aux 2 (MSD transfer line)	
Heater	On
Temperature	250 °C
Column	Agilent J&W 121-1324UI, DB-624UI, 20 m × 180 µm, 1 µm
Max temperature	260 °C
In	Front SS inlet He
Out	Vacuum
Initial temperature	35 °C
Pressure	14.125 psi
Constant flow	0.7 mL/min
Average velocity	37.062 cm/s
Holdup time	0.8994 minutes
Run time	18 minutes

MS Acquisition parameters	
Tune file	BFB_Atune.u
Acquisition mode	Scan
Solvent delay	1.05 minutes
EMV mode	Gain factor
Gain factor	5.00
Scan parameters	
Low mass	35.0
High mass	260.0
Threshold	100
Sample	2 A/D samples 4
MS zones	
MS source	250 °C
MS quad	200 °C
Trace ion detection	ON

Method 524.M compound list with approximate retention times and suggested nominal quantitation ions

Name	Ret Time	Signal	Q1 Signal	Q2 Signal	Q3 Signal
Fluorobenzene (ISTD)	6.61	96	77		
Dichlorodifluoromethane	1.22	85	87	50	
Chloromethane	1.37	50	52	49	
Vinyl chloride	1.47	62	64		
Bromomethane	1.77	94	96	93	79
Chloroethane	1.87	64	66	49	
Trichlorofluoromethane	2.12	101	103	66	105
Diethyl ether	2.45	59	74	45	43
1,1-Dichloroethene	2.68	61	96	98	
Acetone	2.78	43	58		
Iodomethane	2.84	142	127	141	
Carbon disulfide	2.9	76			
Allyl chloride	3.13	41	39	76	78
Methylene chloride	3.31	49	84	86	51
trans-1,2-Dichloroethene	3.69	61	96	98	63
Acrylonitrile	3.69	53	52	51	
Methyl tert-butyl ether (MTBE)	3.74	73	57	43	41
1,1-Dichloroethane	4.36	63	65		
2,2-Dichloropropane	5.18	77	41	79	39
cis-1,2-Dichloroethene	5.19	61	96	98	63
2-Butanone (MEK)	5.27	43	72		
Propionitrile	5.35	54	52		
Methyl acrylate	5.4	55	85	42	
Bromochloromethane	5.5	49	130	128	51
Methacrylonitrile	5.53	41	67	39	51
THF	5.58	42	41	71	
Chloroform	5.64	83	85	47	
1,1,1-Trichloroethane	5.82	97	99	61	
1-Chlorobutane	5.97	56	49		
Carbon Tetrachloride	6.01	117	119	121	82
1,1-Dichloro-1-propene	6.03	75	39	110	77
Benzene	6.26	78	77	51	52.1
1,2-Dichloroethane	6.31	62	64	49	
Trichloroethene	7.02	130	95	132	97
1,2-Dichloropropane	7.26	63	62	41	76
Dibromomethane	7.38	174	93	95	172
Methyl methacrylate	7.44	41	69	39	100
Bromodichloromethane	7.57	83	85	47	48
cis-1,3-Dichloropropene	8.04	75	39	77	110
2-Nitropropane	8.23	43	41	39	42
4-methyl-2-pentanone [MIBK]	8.23	43	58	41	
Toluene	8.37	91	92	65	

Name	Ret Time	Signal	Q1 Signal	Q2 Signal	Q3 Signal
1,1-Dichloropropanone	8.23	43	83		
trans-1,3-Dichloropropene	8.62	75	39	77	77
Ethyl methacrylate	8.74	69	41	39	99
1,1,2-Trichloroethane	8.8	97	83	61	99
Tetrachloroethene	8.92	166	164	129	131
1,3-Dichloropropane	8.96	76	41	78	39
2-Hexanone	9.08	43	58	57	41
Dibromochloromethane	9.19	129	127	131	79
1,2-Dibromoethane	9.29	107	109		
Chlorobenzene	9.78	112	77	114	51
1,1,1,2-Tetrachloroethane	9.87	131	133	119	
Ethylbenzene	9.91	91	106		
m+p-Xylene	10.02	91	106	105	77
o-Xylene	10.41	91	106	105	77
Styrene	10.43	104	103	78	51
Bromoform	10.6	173	171	175	
Isopropylbenzene	10.79	105	120	77	79
Bromofluorobenzene	10.93	95	174	176	75
Bromobenzene	11.07	77	156	158	
1,1,2,2-Tetrachloroethane	11.09	83	85	95	60
1,2,3-Trichloropropane	11.13	75	77		
n-Propylbenzene	11.2	91	120	65	92
trans-1,4-Dichloro-2-butene	11.15	53	89	124	
2-Chlorotoluene	11.27	91	126	89	63
1,3,5-Trimethylbenzene	11.37	105	120	75	77
4-Chlorotoluene	11.38	91	126	63	125
tert-Butylbenzene	11.7	119	91	134	77
1,2,4-Trimethylbenzene	11.74	105	120	77	119
sec-Butylbenzene	11.91	105	134	91	77
1,3-Dichlorobenzene	12.01	146	148	111	75
p-Isoproyltoluene	12.06	119	134	91	117
1,4-Dichlorobenzene	12.1	146	148	111	75
1,2-Dichlorobenzene-d4	12.45	152	115	150	
1,2-Dichlorobenzene	12.47	146	148	111	
n-Butylbenzene	12.47	91	92	134	65
Hexachloroethane	12.72	117	119	201	
1,2-Dibromo-3-chloropropane (DBCP)	13.25	157	75	155	39
1,2,4-Trichlorobenzene	14.07	180	182	145	74
Hexachlorobutadiene	14.25	225	227	223	118
Naphthalene	14.31	128	127	129	
1,2,3-Trichlorobenzene	14.55	180	182	145	74

Appendix C
Agilent GC/MS method conditions for acquiring BFB

Instrument control parameters: Method BFB_8260B.M

GC Acquisition parameters

Sample inlet	GC
Injection source	External device
Mass spectrometer	Enabled
Equilibration time	0 minutes
Max temperature	260 °C
Slow fan	Disabled
Oven program	On 40 °C for 1 minutes then 25 °C/min to 200 °C for 0.6 minutes Run time is 8 minutes
Front SS inlet	He
Mode	Split
Heater	200 °C
Pressure	14.517 psi
Total flow	110.7 mL/min
Septum purge flow	5 mL/min
Gas saver	Off
Split ratio	150:1
Split flow	105 mL/min
Thermal Aux 2 (MSD Transfer Line)	
Heater	On
Temperature	250 °C
Column	Agilent J&W 121-1324UI, DB-624UI, 20 m × 180 µm, 1 µm
Max temperature	260 °C
In	Front SS inlet He
Out	Vacuum
Initial temperature	35 °C
Pressure	14.517 psi
Constant flow	0.7 mL/min
Average velocity	37.157 cm/s
Holdup time	0.8971 minutes
Run time	8 minutes

MS Acquisition parameters

Tune file	BFB_Atune.u
Acquisition mode	Scan
Solvent delay	3.00 minutes
EMV mode	Gain factor
Gain factor	5.00
Scan parameters	
Low mass	35.0
High mass	300.0
Threshold	100
Sample	2 A/D samples 4
MS zones	
MS source	250 °C
MS quad	200 °C
Trace ion detection	ON

Appendix D
Agilent GC/MS method conditions for acquiring VOCs

Instrument control parameters: Method 8260B.M

GC Acquisition parameters

Sample inlet	GC
Injection source	External device
Mass spectrometer	Enabled
Equilibration time	0 minutes
Max temperature	260 °C
Slow fan	Disabled
Oven program	On 35 °C for 4 minutes then 15 °C/min to 240 °C for 0.3333 minutes Run time is 18 minutes
Front SS inlet	He
Mode	Split
Heater	200 °C
Pressure	14.125 psi
Total flow	110.7 mL/min
Septum purge flow	5 mL/min
Gas saver	Off
Split ratio	150:1
Split flow	105 mL/min

Thermal Aux 2 (MSD transfer line)

Heater	On
Temperature	250 °C
Column	Agilent J&W 121-1324UI, DB-624UI, 20 m × 180 µm, 1 µm
Max temperature	260 °C
In	Front SS inlet He
Out	Vacuum
Initial temperature	35 °C
Pressure	14.125 psi
Constant flow	0.7 mL/min
Average velocity	37.062 cm/s
Holdup time	0.8994 minutes
Run time	18 minutes

MS Acquisition parameters

Tune file	BFB_Atune.u
Acquisition mode	Scan
Solvent delay	1.05 minutes
EMV mode	Gain factor
Gain factor	5.00

Scan parameters

Low mass	35.0
High mass	300.0
Threshold	100
Sample	2 A/D samples 4

MS Zones

MS source	250 °C
MS quad	200 °C
Trace ion detection	ON

Method 8260B.M compound list with approximate retention times and suggested nominal quantitation ions

Name	Ret Time	Signal	Q1 Signal	Q2 Signal	Q3 Signal
Fluorobenzene (ISTD)	6.7	96	70	50	77
Chlorodifluoromethane	1.56	51	67	69	
Dichlorodifluoromethane	1.24	85	87	50	
Chloromethane	1.39	50	52	49	
Vinyl chloride	1.5	62	64	61	
1,3-Butadiene	1.87	54	53	51	
Bromomethane	1.81	96	94	81	
Chloroethane	1.92	64	66	49	
Dichlorofluoromethane	2.19	67	69	47	
Trichlorofluoromethane	2.19	101	103	66	
Ethyl ether	2.52	59	45	74	
Acrolein	2.33	56	55	53	
Trichlorotrifluoroethane	2.81	151	101	153	
1,1-Dichloroethene	2.77	96	63	61	
Acetone	2.89	43	58	42	
Iodomethane	2.877	142	127	141	
Carbon disulfide	3.01	76	78	77	
2-Propanol (Isopropyl alcohol)	2.5	45	44	59	
3-Chloro-1-propene	3.01	76	41	78	
Methyl acetate	3.34	43	74	59	
Acetonitrile	3.14	40	39	41	42
Methylene chloride	3.43	84	86	49	51
tert-Butyl alcohol	3.76	59	57	60	
Acrylonitrile	3.82	53	52	51	
Methyl tert-Butyl Ether	3.92	73	57	55	
trans-1,2-Dichloroethene	3.84	96	61	98	
Hexane	4.24	57	56	71	55
Diisopropyl ether	4.65	45	59	87	
1,1-Dichloroethane	4.51	63	65	83	
Vinyl acetate	4.51	86	43		
Chloroprene	4.67	53	88	90	62
tert-Butyl ethyl ether	5.08	59	87	57	
2,2-Dichloropropane	5.32	77	79	97	
cis-1,2-Dichloroethene	5.34	96	98	61	
2-Butanone	5.43	72	43	57	
Ethyl acetate	5.45	61	70	88	
Propionitrile	5.41	54	53	55	50
Methacrylonitrile	5.81	67	66	52	41
Bromochloromethane	5.64	128	49	130	
Tetrahydrofuran	5.77	71	72	42	
Chloroform	5.77	83	85	47	
Cyclohexane	6.01	56	84	69	
1,1,1-Trichloroethane	5.96	97	99	61	
Dibromofluoromethane	5.96	113	111	192	
Carbon tetrachloride	5.96	117	119	121	
1,1-Dichloropropene	6.02	75	110	77	

Name	Ret Time	Signal	Q1 Signal	Q2 Signal	Q3 Signal
Isobutyl alcohol	6.4	43	41	42	
1,2-Dichloroethane-d4	6.35	65	67	51	
Benzene	6.41	78	52	51	
1,2-Dichloroethane	6.44	62	64	49	98
tert-Amyl methyl ether	6.57	55	73	87	
Trichloroethene	7.15	95	132	130	97
Methylcyclohexane	7.39	83	55	98	
1,2-Dichloropropane	7.39	63	62	41	112
Dibromomethane	7.51	93	95	174	172
Methyl methacrylate	7.5	69	100	59	39
1,4-Dioxane	7.6	88	58		
Bromodichloromethane	7.7	83	85	127	
2-Nitropropane	8.28	41	43	39	38
2-Chloroethyl vinyl ether	8.04	63	106	65	
cis-1,3-Dichloropropene	8.17	75	77	39	
4-Methyl-2-pentanone (MIBK)	8.37	58	43	85	100
Toluene-d8	8.44	98	99	70	
Toluene	8.51	92	91	65	
Chlorobenzene-d5 (ISTD)	9.89	82	117	119	
n-Octane	8.51	85	57	71	
trans-1,3-Dichloropropene	8.74	75	77	39	
Ethyl methacrylate	8.78	69	86	99	41
1,1,2-Trichloroethane	8.92	83	97	85	99
Tetrachloroethene	9.06	164	129	131	166
2-Hexanone	9.22	57	43	100	53
1,3-Dichloropropane	9.09	76	78	63	
Dibromochloromethane	9.31	129	206	208	
1,2-Dibromoethane (EDB)	9.42	107	109	188	
1-Chlorohexane	9.91	91	41	69	
Chlorobenzene	9.92	112	114	77	
Ethylbenzene	10.04	106	91	77	
1,1,1,2-Tetrachloroethane	10	131	133	119	
m,p-Xylenes	10.16	106	91	77	
o-Xylene	10.54	106	91	65	
Styrene	10.56	103	104	78	
Bromoform	10.73	173	171	175	254
Isopropylbenzene	10.92	105	120	77	
cis-1,4-Dichloro-2-butene	10.91	89	62	75	124
4-Bromofluorobenzene	11.06	95	174	176	
1,4-Dichlorobenzene-d4 (ISTD)	12.13	152	150	115	
1,1,2,2-Tetrachloroethane	11.21	83	85	131	
trans-1,4-Dichloro-2-bu	91	126	63		
tert-Butylbenzene	11.83	119	91	134	
1,2,4-Trimethylbenzene	11.87	105	120	77	
sec-Butylbenzene	12.05	105	134	91	
p-Isopropyltoluene	12.19	119	134	91	
1,3-Dichlorobenzene	12.15	146	111	148	
1,4-Dichlorobenzene	12.15	146	111	148	

Name	Ret Time	Signal	Q1 Signal	Q2 Signal	Q3 Signal
n-Butylbenzene	12.6	91	92	134	
1,2-Dichlorobenzene	12.61	146	111	148	
1,2-Dibromo-3-chloropropane (DBCP)	13.38	155	157	75	
1,3,5-Trichlorobenzene	13.546	180	182	145	
1,2,4-Trichlorobenzene	14.21	180	182	145	
Hexachlorobutadiene	14.39	225	223	227	
Naphthalene	14.45	128	127	102	
1,2,3-Trichlorobenzene	14.7	180	182	145	

Appendix E
Teledyne Tekmar Teklink methods for the Atomx

Method: Method 524_5 mL - VOCARB

Method type: Water

Instrument: Atomx

Comments: Teklink Method for the Atomx with 5 mL purge vessel using the VOCARB 3000 (#K) Trap

Purge	
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Water heater temperature	80 °C
Sample vial temperature	20 °C
Sample equilibrate time	0.00 minutes
Soil valve temperature	110 °C
Standby flow purge	10 mL/min
Ready temperature condensate	40 °C
Ready temperature	45 °C
Presweep time	0.25 minutes
Prime sample fill volume	3.0 mL
Sample volume	5.0 mL
Sweep sample time	0.25 minutes
Sweep sample flow	100 mL/min
Sparge vessel heater	Off
Sparge vessel temperature	20 °C
Prepurge time	0.00 minutes
Prepurge flow	0 mL/min
Purge time	11.0 minutes
Purge flow	40 mL/min
Purge temperature	20 °C
Condensate purge temperature	20 °C
Dry purge time	2.00 minutes
Dry purge flow	100 mL/min
Dry purge temperature	20 °C

Desorb	
Methanol needle rinse	Off
Methanol needle rinse volume	0.0 mL
Water needle rinse volume	7.0 mL
Sweep needle time	0.25 minutes
Desorb preheat temperature	245 °C
GC start signal	Start of desorb
Desorb time	4.00 minutes
Drain flow	100 mL/min
Desorb temperature	250 °C

Bake	
Methanol glass rinse	Off
Number of methanol glass rinses	0
Methanol glass rinse volume	0.0 mL
Number of water bake rinses	3
Water bake rinse volume	7.0 mL
Bake rinse sweep time	0.40 minutes
Bake rinse sweep flow	100 mL/min
Bake rinse drain time	0.60 minutes
Bake time	6.00 minutes
Bake flow	200 mL/min
Bake temperature	260 °C
Condensate bake temperature	200 °C

Cryo	
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: "Method 524_25 mL - VOCARB"

Method type: Water

Instrument: Atomx

Comments: Teklink Method for the Atomx with 25 mL purge vessel using the VOCARB 3000 (#K) Trap

Purge

Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Water heater temperature	80 °C
Sample vial temperature	20 °C
Sample equilibrate time	0.00 minutes
Soil valve temperature	110 °C
Standby flow purge	10 mL/min
Ready temperature condensate	40 °C
Ready temperature	45 °C
Presweep time	0.25 minutes
Prime sample fill volume	3.0 mL
Sample volume	25.0 mL
Sweep sample time	0.25 minutes
Sweep sample flow	100 mL/min
Sparge vessel heater	Off
Sparge vessel temperature	20 °C
Prepurge time	0.00 minutes
Prepurge flow	0 mL/min
Purge time	11.0 minutes
Purge flow	40 mL/min
Purge temperature	20 °C
Condensate purge temperature	20 °C
Dry purge time	2.00 minutes
Dry purge flow	100 mL/min
Dry purge temperature	20 °C

Desorb

Methanol needle rinse	Off
Methanol needle rinse volume	0.0 mL
Water needle rinse volume	27.0 mL
Sweep needle time	0.25 minutes
Desorb preheat temperature	245 °C
GC start signal	Start of desorb
Desorb time	4.00 minutes
Drain flow	100 mL/min
Desorb temperature	250 °C

Bake

Methanol glass rinse	Off
Number of methanol glass rinses	0
Methanol glass rinse volume	3.0 mL
Number of water bake rinses	3
Water bake rinse volume	27.0 mL
Bake rinse sweep time	0.40 minutes
Bake rinse sweep flow	100 mL/min
Bake rinse drain time	0.60 minutes
Bake time	6.00 minutes
Bake flow	200 mL/min
Bake temperature	260 °C
Condensate bake temperature	200 °C

Cryo

Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: "Method 8260_5 mL - VOCARB"

Method type: Water

Instrument: Atomx

Comments: Teklink Method for the Atomx with 5 mL purge vessel using the VOCARB 3000 (#K) Trap

Purge	
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Water heater temperature	80 °C
Sample vial temperature	20 °C
Sample equilibrate time	0.00 minutes
Soil valve temperature	110 °C
Standby flow purge	10 mL/min
Ready temperature condensate	40 °C
Ready temperature	45 °C
Presweep time	0.25 minutes
Prime sample fill volume	3.0 mL
Sample volume	25.0 mL
Sweep sample time	0.25 minutes
Sweep sample flow	100 mL/min
Sparge vessel heater	Off
Sparge vessel temperature	20 °C
Prepurge time	0.00 minutes
Prepurge flow	0 mL/min
Purge time	11.0 minutes
Purge flow	40 mL/min
Purge temperature	20 °C
Condensate purge temperature	20 °C
Dry purge time	2.00 minutes
Dry purge flow	100 mL/min
Dry purge temperature	20 °C

Desorb	
Methanol needle rinse	Off
Methanol needle rinse volume	0.0 mL
Water needle rinse volume	27.0 mL
Sweep needle time	0.25 minutes
Desorb preheat temperature	245 °C
GC start signal	Start of desorb
Desorb time	0.50 minutes
Drain flow	100 mL/min
Desorb temperature	250 °C

Bake	
Methanol glass rinse	Off
Number of methanol glass rinses	0
Methanol glass rinse volume	0.0 mL
Number of water bake rinses	3
Water bake rinse volume	27.0 mL
Bake rinse sweep time	0.40 minutes
Bake rinse sweep flow	100 mL/min
Bake rinse drain time	0.60 minutes
Bake time	6.00 minutes
Bake flow	200 mL/min
Bake temperature	260 °C
Condensate bake temperature	200 °C

Cryo	
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: "Cold Standby_5 mL - VOCARB"

Method type: Water

Instrument: Atomx

Comments: Teklink Method for the Atomx with 5 mL purge vessel using the VOCARB 3000 (#K) Trap

Purge	
Valve oven temperature	20 °C
Transfer line temperature	20 °C
Sample mount temperature	20 °C
Water heater temperature	80 °C
Sample vial temperature	20 °C
Sample equilibrate time	0.00 minutes
Soil valve temperature	20 °C
Standby flow purge	10 mL/min
Ready temperature condensate	40 °C
Ready temperature	45 °C
Presweep time	0.25 minutes
Prime sample fill volume	3.0 mL
Sample volume	5.0 mL
Sweep sample time	0.25 minutes
Sweep sample flow	100 mL/min
Sparge vessel heater	Off
Sparge vessel temperature	20 °C
Prepurge time	0.00 minutes
Prepurge flow	0 mL/min
Purge time	11.0 minutes
Purge flow	40 mL/min
Purge temperature	20 °C
Condensate purge temperature	20 °C
Dry purge time	2.00 minutes
Dry purge flow	100 mL/min
Dry purge temperature	20 °C

Desorb	
Methanol needle rinse	Off
Methanol needle rinse volume	0.0 mL
Water needle rinse volume	10.0 mL
Sweep needle time	0.25 minutes
Desorb preheat temperature	245 °C
GC start signal	Start of desorb
Desorb time	4.00 minutes
Drain flow	100 mL/min
Desorb temperature	250 °C

Bake	
Methanol glass rinse	Off
Number of methanol glass rinses	0
Methanol glass rinse volume	0.0 mL
Number of water bake rinses	2
Water bake rinse volume	7.0 mL
Bake rinse sweep time	0.40 minutes
Bake rinse sweep flow	100 mL/min
Bake rinse drain time	0.60 minutes
Bake time	4.00 minutes
Bake flow	200 mL/min
Bake temperature	260 °C
Condensate bake temperature	200 °C

Cryo	
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: "Cold Standby_25 mL - VOCARB"

Method type: Water

Instrument: Atomx

Comments: Teklink Method for the Atomx with 25 mL purge vessel using the VOCARB 3000 (#K) Trap

Purge	
Valve oven temperature	20 °C
Transfer line temperature	20 °C
Sample mount temperature	20 °C
Water heater temperature	80 °C
Sample vial temperature	20 °C
Sample equilibrate time	0.00 minutes
Soil valve temperature	20 °C
Standby flow purge	10 mL/min
Ready temperature condensate	40 °C
Ready temperature	45 °C
Presweep time	0.25 minutes
Prime sample fill volume	3.0 mL
Sample volume	25.0 mL
Sweep sample time	0.25 minutes
Sweep sample flow	100 mL/min
Sparge vessel heater	Off
Sparge vessel temperature	20 °C
Prepurge time	0.00 minutes
Prepurge flow	0 mL/min
Purge time	11.0 minutes
Purge flow	40 mL/min
Purge temperature	20 °C
Condensate purge temperature	20 °C
Dry purge time	2.00 minutes
Dry purge flow	100 mL/min
Dry purge temperature	20 °C

Desorb	
Methanol needle rinse	Off
Methanol needle rinse volume	0.0 mL
Water needle rinse volume	10.0 mL
Sweep needle time	0.25 minutes
Desorb preheat temperature	245 °C
GC start signal	Start of desorb
Desorb time	4.00 minutes
Drain flow	100 mL/min
Desorb temperature	250 °C

Bake	
Methanol glass rinse	Off
Number of methanol glass rinses	0
Methanol glass rinse volume	0.0 mL
Number of water bake rinses	2
Water bake rinse volume	27.0 mL
Bake rinse sweep time	0.40 minutes
Bake rinse sweep flow	100 mL/min
Bake rinse drain time	0.60 minutes
Bake time	4.00 minutes
Bake flow	200 mL/min
Bake temperature	260 °C
Condensate bake temperature	200 °C

Cryo	
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Appendix F
Teledyne Tekmar Teklink methods for the StratUm + AquaTEK 70

Method: Method 524_5 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Purge ready temperature	45 °C
Standby flow	10 mL/min
Pressurize time	0.25 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	0.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	40 °C
Condenser purge temperature	20 °C
Rinse loop time	0.25 minutes
Purge loop time	0.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	20 °C
Dry purge flow	100 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	4.00 minutes
Desorb temperature	250 °C
Desorb flow	100 mL/min
Bake rinse	On
Number of bake rinses	3
Bake drain time	0.25 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: Method 524_25 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Purge ready temperature	45 °C
Standby flow	10 mL/min
Pressurize time	0.60 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	1.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	40 °C
Condenser purge temperature	20 °C
Rinse loop time	0.75 minutes
Purge loop time	1.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	20 °C
Dry purge flow	100 mL/min
GC start	Start of desorb
Desorb	Preheat temperature 245 °C
Desorb drain	On
Desorb time	4.00 minutes
Desorb temperature	250 °C
Desorb flow	100 mL/min
Bake rinse	On
Number of bake rinses	3
Bake drain time	0.75 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: 8260_5 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Purge ready temperature	45 °C
Standby flow	10 mL/min
Pressurize time	0.25 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	0.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	40 °C
Condenser purge temperature	20 °C
Rinse loop time	0.25 minutes
Purge loop time	0.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	20 °C
Dry purge flow	100 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	0.50 minutes
Desorb temperature	250 °C
Desorb flow	100 mL/min
Bake rinse	On
Number of bake rinses	3
Bake drain time	0.25 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: 8260_25 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	125 °C
Transfer line temperature	125 °C
Sample mount temperature	40 °C
Purge ready temperature	45 °C
Standby flow	10 mL/min
Pressurize time	0.60 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	1.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	40 °C
Condenser purge temperature	20 °C
Rinse loop time	0.75 minutes
Purge loop time	1.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	20 °C
Dry purge flow	100 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	0.50 minutes
Desorb temperature	250 °C
Desorb flow	200 mL/min
Bake rinse	On
Number of bake rinses	3
Bake drain time	0.75 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: Cold Standby_5 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	20 °C
Transfer line temperature	20 °C
Sample mount temperature	20 °C
Purge ready temperature	20 °C
Standby flow	0 mL/min
Pressurize time	0.25 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	0.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	20 °C
Condenser purge temperature	20 °C
Rinse loop time	0.25 minutes
Purge loop time	0.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	40 °C
Dry purge flow	200 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	4.00 minutes
Desorb temperature	250 °C
Desorb flow	100 mL/min
Bake rinse	On
Number of bake rinses	1
Bake drain time	0.25 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Method: Cold Standby_25 mL - VOCARB

Stratum (with AQUATek 70) Method and VOCARB 3000 (#K) Trap

Parameter	Setpoint
Valve oven temperature	20 °C
Transfer line temperature	20 °C
Sample mount temperature	20 °C
Purge ready temperature	20 °C
Standby flow	0 mL/min
Pressurize time	0.60 minutes
Fill I.S. time	0.04 minutes
Sample transfer time	1.50 minutes
Pre-purge time	0.50 minutes
Pre-purge flow	40 mL/min
Sample heater	Off
Sample preheat time	1.00 minutes
Sample temperature	40 °C
Purge time	11.00 minutes
Purge temperature	0 °C
Purge flow	40 mL/min
Condenser ready temperature	20 °C
Condenser purge temperature	20 °C
Rinse loop time	0.75 minutes
Purge loop time	1.50 minutes
Dry purge time	2.00 minutes
Dry purge temperature	40 °C
Dry purge flow	200 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	4.00 minutes
Desorb temperature	250 °C
Desorb flow	100 mL/min
Bake rinse	On
Number of bake rinses	1
Bake drain time	0.75 minutes
Bake drain flow	400 mL/min
Bake time	6.00 minutes
Bake temperature	260 °C
Bake flow	200 mL/min
Condenser bake temperature	200 °C
Focus temperature	n/a
Inject time	n/a
Inject temperature	n/a
Standby temperature	n/a

Appendix G
Method 8260B-ICAL

Compound	0.25000 Level 1	0.50000 Level 2	1.000 Level 3	4.000 Level 4	20.000 Level 5	40.000 Level 6	60.000 Level 7	80.000 Level 8	RRF	%RSD
8. Dichlorodifluoromethane	0.34054	0.32883	0.31235	0.42528	0.41838	0.40534	0.39518	0.38404	0.37624	11.477
9. Chloromethane (**)	0.50701	0.44283	0.39135	0.43214	0.39398	0.38491	0.37392	0.37243	0.41232	11.183
10. Vinyl chloride (*)	0.37353	0.32909	0.38365	0.41448	0.39844	0.38986	0.37795	0.35849	0.37819	6.862
11. Bromomethane	+++++	0.29372	0.19802	0.23361	0.20475	0.22141	0.21065	0.21717	0.22562	14.261
12. Chloroethane	0.23582	0.19971	0.22573	0.24434	0.21540	0.21818	0.20800	0.21158	0.21985	6.717
13. Trichlorofluoromethane	0.58258	0.59444	0.50895	0.55473	0.53940	0.52228	0.51544	0.49372	0.53894	6.656
15. Trichlorotrifluoroethane	0.33586	0.29722	0.30380	0.32937	0.29805	0.28793	0.28376	0.27627	0.30153	7.011
17. 1,1-Dichloroethene(*)	0.36693	0.30018	0.34161	0.35170	0.32497	0.30486	0.30203	0.29625	0.32357	8.347
18. Acetone	+++++	+++++	+++++	0.20374	0.20235	0.19494	0.18732	0.18789	0.19525	3.964
21. Carbon disulfide	1.09419	1.03008	0.99721	1.07853	0.98246	0.95236	0.94433	0.92458	1.00047	6.244
31. Vinyl acetate	+++++	0.77985	0.71755	0.84031	0.77711	0.73544	0.71843	0.71068	0.75420	6.281
22. Methylene Chloride	0.31745	0.34951	0.35783	0.38429	0.34684	0.33316	0.33926	0.33192	0.34503	5.834
23. t-Butanol	0.14286	0.13429	0.12444	0.13519	0.12231	0.11764	0.11394	0.11315	0.12548	8.687
25. Methyl-t-butyl-ether	0.96439	1.02300	0.95850	1.06853	0.95791	0.91416	0.92313	0.92509	0.96684	5.540
26. *trans*-1, 2-Dichloroethene	0.29336	0.40553	0.37764	0.39099	0.36397	0.35042	0.34776	0.34137	0.35888	9.641
27. Acrylonitrile	0.24006	0.22783	0.23137	0.25382	0.23640	0.22904	0.22784	0.23414	0.23506	3.715
30. 1,1-Dichloroethane (**)	0.69013	0.70848	0.67984	0.72079	0.66661	0.63283	0.63595	0.62699	0.67020	5.340
33. 2,2-Dichloropropane	0.57176	0.59340	0.54055	0.56170	0.51466	0.47754	0.47232	0.45268	0.52308	9.905
35. *cis*-1,2-Dichloroethene	0.47801	0.52877	0.42189	0.43850	0.41024	0.38637	0.39012	0.38440	0.42979	11.851
38. 2-Butanone	+++++	+++++	0.42879	0.34666	0.33205	0.32803	0.32521	0.32447	0.34753	11.691
40. Bromochloromethane	+++++	+++++	0.20272	0.20572	0.19675	0.18114	0.18361	0.18309	0.19217	5.668
41. Chloroform (*)	+++++	0.62212	0.63520	0.67729	0.63984	0.61394	0.61219	0.60416	0.62925	3.926
42. 1,1,1-Trichloroethane	0.61786	0.57410	0.58272	0.61973	0.57669	0.54677	0.54434	0.53548	0.57471	5.580
43. Carbon tetrachloride	0.59278	0.53642	0.53099	0.55774	0.52058	0.50300	0.50351	0.49239	0.52968	6.265
44. 1,1-Dichloropropene	0.65875	0.54076	0.52106	0.57393	0.52341	0.50907	0.50694	0.49943	0.54167	9.759
47. Benzene	1.53791	1.44549	1.40623	1.49558	1.43492	1.40384	1.40025	1.40732	1.44144	3.499
48. 1,2-Dichloroethane	0.50770	0.44387	0.46611	0.46986	0.45238	0.43474	0.44404	0.43859	0.45716	5.239
49. Trichloroethene	0.40789	0.38258	0.41269	0.42306	0.40198	0.38747	0.39061	0.38609	0.39905	3.661
51. 1,2-Dichloropropane (*)	0.35136	0.37070	0.34038	0.37379	0.36086	0.34441	0.34830	0.34800	0.35472	3.479
52. Dibromomethane	0.27656	0.27396	0.23080	0.24449	0.22880	0.22101	0.22154	0.22046	0.23970	9.723
53. 1,4-Dioxane	+++++	0.00665	0.00686	0.00730	0.00658	0.00583	0.00481	+++++	0.00634	14.004
54. Bromodichlorornethane	0.50463	0.48486	0.44358	0.47389	0.45642	0.44111	0.45084	0.44663	0.46274	4.931
55. 2-Chloroethyl vinyl ether	0.25055	0.24377	0.22546	0.23840	0.23473	0.23667	0.23756	0.24129	0.23855	3.045
56. *cis*-1,3-Dichloropropene (xtr)	0.62883	0.57568	0.51216	0.53851	0.52698	0.51680	0.52608	0.52474	0.54372	7.279
57. 4-Methyl-2-pentanone	0.60482	0.64669	0.65514	0.58200	0.57700	0.56518	0.56479	0.55924	0.59436	6.345
58. Toluene (*)	0.94889	0.92042	0.82292	0.90067	0.88503	0.85842	0.87079	0.85925	0.88330	4.485
60. *trans*-1,3-Dichloropropene (xt)	0.55910	0.47078	0.43113	0.48910	0.48072	0.47056	0.47671	0.47433	0.48155	7.416
61. 1,1,2-Trichloroethane	+++++	0.25262	0.30965	0.29899	0.30355	0.28960	0.29366	0.29014	0.29117	6.351
62. Tetrachloroethene	0.42639	0.48062	0.40806	0.44374	0.43233	0.42542	0.41702	0.42540	0.43237	5.110
63. 1,3-Dichloropropane	0.60404	0.51274	0.50988	0.52912	0.53231	0.51849	0.51925	0.53084	0.53209	5.685
64. 2-Hexanone	+++++	0.57036	0.47718	0.51047	0.47741	0.46341	0.45102	0.45284	0.48610	8.688

Compound	0.25000 Level 1	0.50000 Level 2	1.000 Level 3	4.000 Level 4	20.000 Level 5	40.000 Level 6	60.000 Level 7	80.000 Level 8	RRF	%RSD
65. Dibromochloromethane	0.38508	0.33835	0.34641	0.37578	0.38024	0.36437	0.36807	0.37830	0.36707	4.559
66. 1,2-Dibromoethane	0.35967	0.37417	0.35581	0.34281	0.34634	0.33914	0.33794	0.34648	0.35029	3.499
67. 1-Chlorohexane	+++++	0.58814	0.54853	0.55296	0.51070	0.49985	0.48664	0.49088	0.52539	7.287
68. Chlorobenzene (**)	0.96667	0.91848	1.00862	1.01542	0.98865	0.98310	0.97858	0.99366	0.98165	3.053
69. Ethylbenzene (*)	0.48166	0.61758	0.53947	0.58841	0.54732	0.54695	0.53601	0.54320	0.55007	7.233
70. 1,1,1,2-Tetrachloroethane	0.38843	0.37476	0.35974	0.37736	0.36125	0.34829	0.34694	0.35344	0.36378	4.111
71. Xylenes (m,p)	0.68620	0.66850	0.66455	0.70890	0.68194	0.67369	0.66115	0.67500	0.67749	2.244
72. Xylene (o)	0.66009	0.60812	0.63083	0.68003	0.65309	0.63433	0.63067	0.63450	0.64146	3.441
73. Styrene	1.05218	1.01902	1.02298	1.11333	1.06150	1.04165	1.04274	1.06017	1.05170	2.793
74. Bromoform (**)	0.31866	0.33307	0.29824	0.31310	0.30990	0.30272	0.30120	0.30478	0.31021	3.680
75. Isopropylbenzene	1.70424	1.76441	1.76368	1.89950	1.80721	1.76833	1.73658	1.76879	1.77659	3.249
77. Bromobenzene	0.70170	0.74450	0.79777	0.75695	0.73628	0.72449	0.74866	0.75597	0.74579	3.734
78. 1,1,2,2-Tetrachloroethane (**)	0.94965	1.00798	0.87034	0.98105	0.93952	0.90843	0.91815	0.93621	0.93892	4.542
79. n-Propylbenzene	3.72507	3.73928	3.77536	4.00119	3.76395	3.79613	3.84690	3.90084	3.81859	2.451
80. 1,2,3-Trichloropropane	0.36902	0.34779	0.34639	0.31888	0.31096	0.30188	0.29985	0.30156	0.32454	8.122
81. 2-Chlorotoluene	1.99411	2.15852	2.14837	2.17295	2.11289	2.08588	2.11367	2.14066	2.11588	2.679
82. 1,3,5-Trimethylbenzene	2.71917	2.70981	2.59186	2.70105	2.54855	2.55629	2.57620	2.61602	2.62692	2.741
83. 4-Chlorotoluene	2.48753	2.32733	2.37602	2.54098	2.42554	2.39238	2.43979	2.47058	2.43252	2.788
84. t-Butylbenzene	2.40875	2.13786	2.20930	2.40179	2.28049	2.29813	2.30034	2.33033	2.29632	3.956
85. 1,2,4-Trimethylbenzene	2.68061	2.44280	2.41069	2.60898	2.53239	2.53223	2.53100	2.57114	2.53873	3.390
86. *sec*-Butylbenzene	3.32992	3.36793	3.37067	3.64648	3.42537	3.41540	3.41839	3.49025	3.43305	2.875
87. 1,3-Dichlorobenzene	1.33554	1.43268	1.39572	1.47391	1.39238	1.38835	1.41153	1.41364	1.40547	2.818
88. p-Isopropyltoluene	2.59450	2.96811	2.78355	3.03347	2.89197	2.89542	2.86107	2.94415	2.87153	4.680
89. 1,4-Dichlorobenzene	1.54755	1.37173	1.37625	1.52487	1.39961	1.39631	1.42161	1.40908	1.43088	4.700
90. n-Butylbenzene	2.40501	2.58999	2.59695	2.79921	2.66904	2.67884	2.61014	2.68299	2.62902	4.290
91. 1,2-Dichlorobenzene	1.33664	1.32903	1.33766	1.36646	1.30666	1.26875	1.27878	1.29849	1.31531	2.508
92. 1,2-Dibromo-3-chloropropane	0.28997	0.35435	0.26477	0.27844	0.26191	0.25209	0.23719	0.24365	0.27280	13.651
93. 1,2,4-Trichlorobenzene	0.92975	0.80247	0.74702	0.92807	0.89751	0.88191	0.77593	0.82462	0.84841	8.299
94. Hexachlorobutadiene	0.51650	0.48834	0.44919	0.54584	0.51395	0.51651	0.44029	0.46489	0.48194	7.620
95. Naphthalene	2.78606	2.57762	2.71709	2.96419	2.88263	2.74830	2.42534	2.64624	2.71843	6.276
96. 1,2,3-Trichlorobenzene	0.86396	0.70228	0.77820	0.85926	0.83005	0.79633	0.68078	0.72953	0.78005	9.005
M 97. 1,2-Dichloroethene (total)	0.38569	0.46717	0.39978	0.41475	0.38710	0.36840	0.36894	0.36289	0.39434	8.671
M 98. Xylenes (total)	0.67750	0.64838	0.65332	0.69928	0.67232	0.66057	0.65099	0.66150	0.66548	2.555
$ 4. Dibromofluoromethane	+++++	0.40755	0.40799	0.35369	0.34457	0.34025	0.34004	0.34015	0.36203	8.732
$ 5. 1,2-Dichloroethane-d4	+++++	0.43999	0.39597	0.38119	0.36329	0.35701	0.35724	0.35803	0.37896	8.101
$ 6. Toluene-d8	+++++	1.40981	1.40176	1.31237	1.29428	1.29787	1.30131	1.31277	1.33288	3.776
$ 7. 4-Bromofluorobenzene	+++++	0.53168	0.49163	0.45964	0.47071	0.45906	0.45144	0.46149	0.47509	5.913
$ 136 Trifluorotoluene	+++++	0.72900	0.69072	0.63774	0.63783	0.63267	0.63224	0.61097	0.65302	6.336

Average %RSD Results:

Calculated Average %RSD = 6.38856

Maximum Average %RSD = 15.00000

*Passed Average %RSD Test.

Appendix H
Method 8260B - MDL Study

Date Analyzed
Instrument 5975C MSD System
Method ID/Description 8260 AQ
Notes Agilent BFB Autotune – 5 mL – Gain Factor = 5

Analyte	Spike ug/L	MDL #1 ug/L	MDL #2 ug/L	MDL #3 ug/L	MDL #4 ug/L	MDL #5 ug/L	MDL #6 ug/L	MDL #7 ug/L	Avg ug/L	%R	SD ug/L	MDL ug/L	RL ug/L
1,1,1,2-Tetrachloroethane	0.250	0.296	0.254	0.249	0.264	0.256	0.269	0.297	0.269	1.077	0.019	0.061	1.0
1,1,1-Trichloroethane	0.250	0.238	0.212	0.244	0.240	0.259	0.265	0.266	0.246	0.985	0.019	0.060	1.0
1,1,2,2-Tetrachloroethane	0.250	0.262	0.228	0.284	0.271	0.256	0.293	0.257	0.264	1.057	0.021	0.067	1.0
1,1,2-Trichloroethane	0.250	0.253	0.293	0.272	0.266	0.266	0.280	0.252	0.269	1.076	0.014	0.045	1.0
1,1-Dichloroethane	0.250	0.236	0.222	0.229	0.255	0.280	0.275	0.240	0.248	0.993	0.023	0.071	1.0
1,1-Dichloroethene	0.250	0.238	0.224	0.262	0.227	0.281	0.306	0.265	0.257	1.030	0.030	0.094	1.0
1,1-Dichloropropene	0.250	0.235	0.233	0.229	0.248	0.271	0.297	0.263	0.254	1.015	0.025	0.077	1.0
1,2,3-Trichlorobenzene	0.250	0.226	0.236	0.225	0.197	0.246	0.257	0.278	0.238	0.952	0.026	0.081	1.0
1,2,3-Trichloropropane	0.250	0.292	0.323	0.272	0.272	0.272	0.271	0.250	0.279	0.1.116	0.023	0.072	1.0
1,2,4-Trichlorobenzene	0.250	0.271	0.239	0.229	0.188	0.264	0.274	0.254	0.245	0.982	0.030	0.095	1.0
1,2,4-Trimethylbenzene	0.250	0.252	0.274	0.272	0.261	0.264	0.257	0.266	0.264	1.055	0.008	0.024	1.0
1,2-Dibromo-3-chloropropane	0.250	0.335	0.335	0.298	0.214	0.354	0.269	0.366	0.310	1.241	0.054	0.168	2.0
1,2-Dibromoethane	0.250	0.252	0.249	0.228	0.305	0.242	0.247	0.282	0.257	1.030	0.027	0.083	2.0
1,2-Dichlorobenzene	0.250	0.235	0.231	0.267	0.239	0.250	0.255	0.243	0.246	0.982	0.012	0.039	1.0
1,2-Dichloroethane	0.250	0.218	0.266	0.235	0.258	0.269	0.264	0.236	0.249	0.996	0.020	0.062	1.0
1,2-Dichloropropane	0.250	0.236	0.247	0.267	0.267	0.280	0.281	0.253	0.262	1.047	0.017	0.053	1.0
1,3,5-Trimethylbenzene	0.250	0.243	0.246	0.271	0.248	0.280	0.271	0.231	0.256	1.023	0.018	0.057	1.0
1,3-Dichlorobenzene	0.250	0.281	0.258	0.264	0.275	0.265	0.291	0.279	0.273	1.093	0.012	0.037	1.0
1,3-Dichloropropane	0.250	0.288	0.247	0.234	0.231	0.253	0.254	0.257	0.252	1.008	0.019	0.059	1.0
1,4-Dichlorobenzene	0.250	0.291	0.269	0.251	0.265	0.262	0.269	0.254	0.266	1.064	0.013	0.041	1.0
1,4-Dioxane	0.250	5.828	3.240	3.928	4.968	5.902	6.215	5.666	5.107	0.817	1.124	3.531	50.0
1-Chlorohexane	0.250	0.325	0.310	0.347	0.328	0.322	0.288	0.257	0.311	1.244	0.030	0.094	2.0
2,2-Dichloropropane	0.250	0.259	0.206	0.276	0.213	0.265	0.296	0.288	0.257	1.030	0.035	0.110	1.0
2-Butanone	0.250	0.509	0.477	0.552	0.577	0.666	0.525	0.599	0.558	2.231	0.063	0.197	2.0
2-Chloroethyl vinyl ether	0.250	0.229	0.273	0.303	0.295	0.235	0.249	0.242	0.261	1.044	0.029	0.092	2.0
2-Chlorotoluene	0.250	0.265	0.272	0.261	0.264	0.284	0.288	0.258	0.270	1.081	0.012	0.036	1.0
2-Hexanone	0.250	0.254	0.258	0.306	0.286	0.289	0.283	0.306	0.283	1.132	0.020	0.064	2.0
4-Chlorotoluene	0.250	0.271	0.284	0.266	0.244	0.267	0.249	0.273	0.265	1.059	0.014	0.044	1.0
4-Methyl-2-pentanone	0.250	0.253	0.251	0.198	0.251	0.266	0.280	0.239	0.248	0.993	0.025	0.081	2.0
Acetone	2.000	2.456	2.542	2.523	2.451	2.274	2.332	2.085	2.380	1.190	0.162	0.509	10.0
Acrylonitrile	2.500	2.437	2.302	2.299	2.298	2.580	2.456	2.226	2.371	0.948	0.124	0.388	20.0
Benzene	0.250	0.237	0.236	0.255	0.247	0.259	0.247	0.238	0.246	0.982	0.009	0.029	1.0
Bromobenzene	0.250	0.278	0.291	0.257	0.276	0.235	0.280	0.259	0.268	1.072	0.019	0.059	1.0
Bromochloromethane	0.250	0.228	0.281	0.267	0.293	0.325	0.278	0.262	0.276	0.1.104	0.030	0.093	1.0
Bromodichloromethane	0.250	0.251	0.232	0.258	0.232	0.217	0.243	0.259	0.242	0.967	0.016	0.049	1.0
Bromoform	0.250	0.217	0.214	0.229	0.260	0.206	0.225	0.230	0.226	0.904	0.017	0.054	1.0
Bromomethane	0.250	0.370	0.211	0.206	0.230	0.324	0.397	0.290	0.290	1.160	0.077	0.243	1.0
Carbon disulfide	0.250	0.274	0.265	0.288	0.286	0.327	0.335	0.286	0.295	1.179	0.026	0.083	2.0

Analyte	Spike ug/L	MDL #1 ug/L	MDL #2 ug/L	MDL #3 ug/L	MDL #4 ug/L	MDL #5 ug/L	MDL #6 ug/L	MDL #7 ug/L	Avg ug/L	SD %R	MDL ug/L	RL ug/L	ug/L
Carbon tetrachloride	0.250	0.203	0.258	0.269	0.268	0.276	0.283	0.263	0.260	1.040	0.026	0.083	1.0
Chlorobenzene	0.250	0.242	0.237	0.275	0.238	0.248	0.244	0.263	0.250	0.999	0.014	0.045	1.0
Chloroethane	0.250	0.245	0.187	0.298	0.211	0.284	0.348	0.168	0.249	0.995	0.065	0.204	1.0
Chloroform	0.250	0.269	0.275	0.236	0.262	0.259	0.234	0.248	0.254	1.018	0.016	0.050	1.0
Chloromethane	0.250	0.209	0.199	0.216	0.205	0.282	0.255	0.251	0.231	0.924	0.032	0.099	1.0
cis-1,2-Dichloroethene	0.250	0.256	0.263	0.272	0.262	0.250	0.274	0.249	0.261	1.043	0.010	0.031	1.0
cis-1,3-Dichloropropene	0.250	0.244	0.237	0.238	0.224	0.221	0.242	0.232	0.234	0.936	0.009	0.028	1.0
Dibromochloromethane	0.250	0.244	0.258	0.223	0.222	0.253	0.253	0.279	0.247	0.988	0.020	0.063	1.0
Dibromomethane	0.250	0.206	0.237	0.252	0.242	0.254	0.255	0.233	0.240	0.960	0.017	0.055	1.0
Dichlorodifluoromethane	0.250	0.184	0.1774	0.210	0.181	0.215	0.185	0.172	0.189	0.756	0.017	0.059	1.0
Ethylbenzene	0.250	0.270	0.259	0.245	0.264	0.270	0.255	0.270	0.262	1.047	0.010	0.030	1.0
Hexachlorobutadiene	0.250	0.225	0.210	0.192	0.196	0.277	0.307	0.300	0.244	0.975	0.050	0.156	1.0
Isopropylbenzene	0.250	0.231	0.236	0.239	0.243	0.252	0.261	0.263	0.246	0.985	0.012	0.039	1.0
Methylene chloride	0.250	0.230	0.322	0.310	0.297	0.302	0.222	0.277	0.280	1.119	0.039	0.124	1.0
Methyl-t-butyl ether	0.250	0.240	0.241	0.258	0.252	0.265	0.274	0.245	0.253	1.014	0.013	0.041	2.0
Naphthalene	0.250	0.219	0.223	0.239	0.215	0.262	0.276	0.272	0.244	0.974	0.026	0.081	1.0
n-Butylbenzene	0.250	0.231	0.229	0.231	0.242	0.267	0.270	0.244	0.245	0.979	0.017	0.054	1.0
n-Propylbenzene	0.250	0.253	0.243	0.277	0.257	0.282	0.264	0.256	0.262	1.047	0.014	0.043	1.0
p-Isopropyltoluene	0.250	0.262	0.248	0.257	0.241	0.272	0.259	0.249	0.255	1.021	0.011	0.033	1.0
sec-Butylbenzene	0.250	0.268	0.243	0.254	0.245	0.270	0.270	0.248	0.257	1.027	0.012	0.038	1.0
Styrene	0.250	0.247	0.255	0.245	0.264	0.247	0.249	0.223	0.247	0.989	0.012	0.039	1.0
t-Butanol	6.250	6.041	6.209	6.315	5.583	6.312	6.667	6.212	6.191	0.329	1.039	1.033	50.0
t-Butylbenzene	0.250	0.248	0.255	0.245	0.248	0.267	0.290	0.257	0.258	1.034	0.016	0.050	1.0
Tetrachloroethene	0.250	0.253	0.258	0.248	0.250	0.259	0.289	0.254	0.259	1.034	0.014	0.044	1.0
Toluene	0.250	0.253	0.289	0.260	0.268	0.262	0.267	0.243	0.263	1.053	0.014	0.045	1.0
trans-1,2-Dichloroethene	0.250	0.285	0.262	0.290	0.266	0.268	0.278	0.269	0.274	1.096	0.011	0.033	1.0
trans-1,3-Dichloropropene	0.250	0.235	0.239	0.269	0.229	0.237	0.212	0.228	0.235	0.942	0.017	0.054	1.0
Trichloroethene	0.250	0.254	0.266	0.257	0.281	0.257	0.271	0.215	0.257	1.029	0.021	0.066	1.0
Trichlorofluoromethane	0.250	0.228	0.225	0.236	0.211	0.259	0.277	0.218	0.236	0.945	0.024	0.074	1.0
Trichlorotrifluoroethane	0.250	0.204	0.222	0.221	0.231	0.124	0.046	0.250	0.185	0.741	0.073	0.230	2.0
Vinyl acetate	0.250	0.246	0.232	0.264	0.267	0.234	0.266	0.227	0.248	0.992	0.018	0.055	2.0
Vinyl chloride	0.250	0.215	0.218	0.263	0.276	0.296	0.326	0.263	0.205	1.061	0.040	0.125	1.0
Xylene (o)	0.250	0.240	0.246	0.284	0.240	0.280	0.262	0.279	0.262	1.046	0.020	0.062	1.0
Xylenes (m,p)	0.500	0.492	0.494	0.523	0.528	0.543	0.524	0.489	0.513	1.026	0.021	0.067	2.0

Appendix I
Method 8260B – ICV

Compound	RRF/Amount	RF20	CCAL RRF20	Min RRF	%D/%Drift	Max %D/%Drift	Curve type
$ 4. Dibromofluoromethane	0.36203	0.31146	0.31146	0.010	-13.96831	40.00000	Averaged
$ 5. 1,2-Dichloroethane-d4	0.37896	0.32994	0.32994	0.010	-12.93545	40.00000	Averaged
$ 6. Toluene-d8	1.33288	1.14283	1.14283	0.010	-14.25917	40.00000	Averaged
$ 7. 4-Bromofluorobenzene	0.47509	0.47790	0.47790	0.010	0.59161	40.00000	Averaged
$ 136 Trifluorotoluene	0.65302	0.59608	0.59608	0.010	-8.72073	40.00000	Averaged
8. Dichlorodifluoromethane	0.37624	0.39228	0.39228	0.010	4.26154	40.00000	Averaged
9. Chloromethane (**)	0.41232	0.38716	0.38716	0.100	-6.10304	40.00000	Averaged
10. Vinyl chloride (*)	0.37819	0.41265	0.41265	0.010	9.11202	20.00000	Averaged
11. Bromomethane	0.22562	0.23083	0.23083	0.010	2.30808	40.00000	Averaged
12. Chloroethane	0.21985	0.22480	0.22480	0.010	2.25157	40.00000	Averaged
13. Trichlorofluoromethane	0.53894	0.52253	0.52253	0.010	-3.04429	40.00000	Averaged
15. Trichlorotrifluoroethane	0.30153	0.31627	0.31627	0.010	4.88828	40.00000	Averaged
17. 1,1-Dichloroethene (*)	0.32357	0.32182	0.32182	0.010	-0.54020	20.00000	Averaged
18. Acetone	0.19525	0.19889	0.19889	0.010	1.86219	40.00000	Averaged
21. Carbon disulfide	1.00047	1.00882	1.00882	0.010	0.83467	40.00000	Averaged
31. Vinyl acetate	0.75420	0.59999	0.59999	0.010	-20.44608	40.00000	Averaged
22. Methylene chloride	0.34503	0.35196	0.35196	0.010	2.00644	40.00000	Averaged
23. t-Butanol	0.12548	0.10968	0.10968	0.001	-12.58837	40.00000	Averaged
25. Methyl-t-butyl-ether	0.96684	0.95800	0.95800	0.010	-0.91396	40.00000	Averaged
26. *trans*-1,2-Dichloroethene	0.35888	0.36820	0.36820	0.010	2.59783	40.00000	Averaged
27. Acrylonitrile	0.23506	0.22755	0.22755	0.010	-3.19806	40.00000	Averaged
30. 1,1-Dichloroethane (**)	0.67020	0.64941	0.64941	0.100	-3.10245	40.00000	Averaged
33. 2,2-Dichloropropane	0.52308	0.47549	0.47549	0.010	-9.09677	40.00000	Averaged
35. *cis*-1,2-Dichloroethene	0.42979	0.38896	0.38896	0.010	-9.50003	40.00000	Averaged
38. 2-Butanone	0.34753	0.32082	0.32082	0.010	-7.68757	40.00000	Averaged
40. Bromochloromethane	0.19217	0.18758	0.18758	0.010	-2.39117	40.00000	Averaged
41. Chloroform (*)	0.62925	0.62438	0.62438	0.010	-0.77408	20.00000	Averaged
42. 1,1,1-Trichloroethane	0.57471	0.56931	0.56931	0.010	-0.93907	40.00000	Averaged
43. Carbon tetrachloride	0.52968	0.51289	0.51289	0.010	-3.16859	40.00000	Averaged
44. 1,1-Dichloropropene	0.54167	0.52360	0.52360	0.010	-3.33563	40.00000	Averaged
47. Benzene	1.44144	1.43003	1.43003	0.010	-0.79210	40.00000	Averaged
48. 1,2-Dichloroethane	0.45716	0.43019	0.43019	0.010	-5.90039	40.00000	Averaged
49. Trichloroethene	0.39905	0.40022	0.40022	0.010	0.29331	40.00000	Averaged
51. 1,2-Dichloropropane (*)	0.35472	0.34473	0.34473	0.010	-2.81753	20.00000	Averaged
52. Dibromomethane	0.23970	0.22263	0.22263	0.010	-7.12274	40.00000	Averaged
53. 1,4-Dioxane	0.00634	0.00461	0.00461	0.000	-27.26887	40.00000	Averaged
54. Bromodichloromethane	0.46274	0.44347	0.44347	0.010	-4.16635	40.00000	Averaged
55. 2-Chloroethyl vinyl ether	0.23855	0.23628	0.23628	0.010	-0.95187	40.00000	Averaged
56. *cis*-1,3-Dichloropropene (xt)	0.54372	0.53208	0.53208	0.010	-2.14022	40.00000	Averaged
57. 4-Methyl-2-pentanone	0.59436	0.56758	0.56758	0.010	-4.50512	40.00000	Averaged
58. Toluene (*)	0.88330	0.85362	0.85362	0.010	-3.36029	20.00000	Averaged
60. *trans*-1,3-Dichloropropene (x)	0.48155	0.47230	0.47230	0.010	-1.92272	40.00000	Averaged

Compound	RRF/Amount	RF20	CCAL RRF20	Min RRF	%D/%Drift	Max %D/%Drift	Curve type
61. 1,1,2-Trichloroethane	0.29117	0.29250	0.29250	0.010	0.45508	40.00000	Averaged
62. Tetrachloroethene	0.43237	0.40519	0.40519	0.010	-6.28733	40.00000	Averaged
63. 1,3-Dichloropropane	0.53209	0.49650	0.49650	0.010	-6.68691	40.00000	Averaged
64. 2-Hexanone	0.48610	0.44823	0.44823	0.010	-7.79089	40.00000	Averaged
65. Dibromochloromethane	0.36707	0.35325	0.35325	0.010	-3.76495	40.00000	Averaged
66. 1,2-Dibromoethane	0.35029	0.33132	0.33132	0.010	-5.41790	40.00000	Averaged
67. 1-Chlorohexane	0.52539	0.48746	0.48746	0.010	-7.21841	40.00000	Averaged
68. Chlorobenzene (**)	0.98165	0.94114	0.94114	0.300	-4.12685	40.00000	Averaged
69. Ethylbenzene (*)	0.55007	0.52974	0.52974	0.010	-3.69605	20.00000	Averaged
70. 1,1,1,2-Tetrachloroethane	0.36378	0.34747	0.34747	0.010	-4.48263	40.00000	Averaged
71. Xylenes (m,p)	0.67749	0.65347	0.65347	0.010	-3.54641	40.00000	Averaged
72. Xylene (o)	0.64146	0.62527	0.62527	0.010	-2.52332	40.00000	Averaged
73. Styrene	1.05170	1.02909	1.02909	0.010	-2.14974	40.00000	Averaged
74. Bromoform (**)	0.31021	0.28663	0.28663	0.010	-7.60009	40.00000	Averaged
75. Isopropylbenzene	1.77659	1.72665	1.72665	0.010	-2.81090	40.00000	Averaged
77. Bromobenzene	0.74579	0.72435	0.72435	0.010	-2.87530	40.00000	Averaged
78. 1,1,2,2-Tetrachloroethane (*)	0.93892	0.88917	0.88917	0.300	-5.29862	40.00000	Averaged
79. n-Propylbenzene	3.81859	3.75426	3.75426	0.010	-1.68474	40.00000	Averaged
80. 1,2,3-Trichloropropane	0.32454	0.29340	0.29340	0.010	-9.59680	40.00000	Averaged
81. 2-Chlorotoluene	2.11588	2.09275	2.09275	0.010	-1.09318	40.00000	Averaged
82. 1,3,5-Trimethylbenzene	2.62692	2.53695	2.53695	0.010	-3.42492	40.00000	Averaged
83. 4-Chlorotoluene	2.43252	2.36035	2.36035	0.010	-2.96675	40.00000	Averaged
84. T-Butylbenzene	2.29632	2.27240	2.27240	0.010	-1.04174	40.00000	Averaged
85. 1,2,4-Trimethylbenzene	2.53873	2.52059	2.52059	0.010	-0.71440	40.00000	Averaged
86. *sec*-Butylbenzene	3.43305	3.41617	3.41617	0.010	-0.49163	40.00000	Averaged
87. 1,3-Dichlorobenzene	1.40547	1.38582	1.38582	0.010	-1.39822	40.00000	Averaged
88. P-Isopropyltoluene	2.87153	2.80363	2.80363	0.010	-2.36437	40.00000	Averaged
89. 1,4-Dichlorobenzene	1.43088	1.38580	1.38580	0.010	-3.15027	40.00000	Averaged
90. n-Butylbenzene	2.62902	2.58521	2.58521	0.010	-1.66640	40.00000	Averaged
91. 1,2-Dichlorobenzene	1.31531	1.29441	1.29441	0.010	-1.58919	40.00000	Averaged
92. 1,2-Dibromo-3-chloropropane	0.27280	0.24717	0.24717	0.010	-9.39397	40.00000	Averaged
93. 1,2,4-Trichlorobenzene	0.84841	0.77579	0.77579	0.010	-8.55990	40.00000	Averaged
94. Hexachlorobutadiene	0.49194	0.45576	0.45576	0.010	-7.35499	40.00000	Averaged
95. Naphthalene	2.71843	2.51048	2.51048	0.010	-7.64987	40.00000	Averaged
96. 1,2,3-Trichlorobenzene	0.78005	0.69988	0.69988	0.010	-10.27695	40.00000	Averaged
M 97. 1,2-Dichloroethene (Total)	0.39434	0.37858	0.37858	0.010	-3.99575	40.00000	Averaged
M 98. Xylenes (Total)	0.66548	0.64407	0.64407	0.010	-3.21804	40.00000	Averaged

Average %D/Drift Results.

Calculated Average %D/Drift = 4.64924

Maximum Average %D/Drift = 15.00000

* Passed Average %D/Drift Test.

Appendix J
Method 8260B - CCV

Compound	RRF/Amount	RF20	CCAL RRF20	MIN RRF	%D/%Drift	Max%D/$Drift	Curve Type
$ 4. Dibromofluoromethane	0.36203	0.30793	0.30793	0.010	-14.94400	40.00000	Averaged
$ 5. 1,2-Dichloroethane-d4	0.37896	0.33032	0.33032	0.010	-12.83532	40.00000	Averaged
$ 6. Toluene-d8	1.33288	1.14804	1.14804	0.010	-13.86768	40.00000	Averaged
$ 7. 4-Bromofluorobenzene	0.47509	0.50135	0.50135	0.010	5.52653	40.00000	Averaged
$ 136 Trifluorotoluene	0.65302	0.56932	0.56932	0.010	-12.81811	40.00000	Averaged
8. Dichlorodifluoromethane	0.37624	0.36928	0.36928	0.010	-1.85114	40.00000	Averaged
9. Chloromethane (**)	0.41232	0.40187	0.40187	0.100	-2.53470	40.00000	Averaged
10. Vinyl chloride (*)	0.37819	0.40335	0.40335	0.010	6.65290	20.00000	Averaged
11. Bromomethane	0.22562	0.21861	0.21861	0.010	-3.10544	40.00000	Averaged
12. Chloroethane	0.21985	0.21516	0.21516	0.010	-2.13141	40.00000	Averaged
13. Trichlorofluoromethane	0.53894	0.50758	0.50758	0.010	-5.81909	40.00000	Averaged
15. Trichlorotrifluoroethane	0.30153	0.30394	0.30394	0.010	0.79949	40.00000	Averaged
17. 1,1-Dichloroethene (*)	0.32357	0.29751	0.29751	0.010	-8.05290	20.00000	Averaged
18. Acetone	0.19525	0.19197	0.19197	0.010	-1.67941	40.00000	Averaged
21. Carbon disulfide	1.00047	1.09580	1.09580	0.010	9.52828	40.00000	Averaged
31. Vinyl acetate	0.75420	0.73831	0.73831	0.010	-2.10624	40.00000	Averaged
22. Methylene Chloride	0.34503	0.32268	0.32268	0.010	-6.47865	40.00000	Averaged
23. t-Butanol	0.12548	0.11780	0.11780	0.001	-6.12193	40.00000	Averaged
25. Methyl-t-butyl-ether	0.96684	0.95180	0.95180	0.010	-1.55603	40.00000	Averaged
26. *trans*-1,2-Dichloroethene	0.35888	0.33219	0.33219	0.010	-7.43626	40.00000	Averaged
27. Acrylonitrile	0.23506	0.23304	0.23304	0.010	-0.85920	40.00000	Averaged
30. 1,1-Dichloroethane (**)	0.67020	0.60576	0.60576	0.100	-9.61609	40.00000	Averaged
33. 2,2-Dichloropropane	0.52308	0.45812	0.45812	0.010	-12.41860	40.00000	Averaged
35. *cis*-1,2-Dichloroethene	0.42979	0.37067	0.37067	0.010	-13.75593	40.00000	Averaged
38. 2-Butanone	0.34753	0.33514	0.33514	0.010	-3.56773	40.00000	Averaged
40. Bromochloromethane	0.19217	0.17708	0.17708	0.010	-7.85416	40.00000	Averaged
41. Chloroform (*)	0.62925	0.58323	0.58323	0.010	-7.31386	20.00000	Averaged
42. 1,1,1-Trichloroethane	0.57471	0.50924	0.50924	0.010	-11.39145	40.00000	Averaged
43. Carbon tetrachloride	0.52968	0.47581	0.47581	0.010	-10.17013	40.00000	Averaged
44. 1,1-Dichloropropene	0.54167	0.48439	0.48439	0.010	-10.57539	40.00000	Averaged
47. Benzene	1.44144	1.33789	1.33789	0.010	-7.18415	40.00000	Averaged
48. 1,2-Dichloroethane	0.45716	0.40972	0.40972	0.010	-10.37868	40.00000	Averaged
49. Trichloroethene	0.39905	0.36261	0.36261	0.010	-9.13196	40.00000	Averaged
51. 1,2-Dichloropropane (*)	0.35472	0.32903	0.32903	0.010	-7.24287	20.00000	Averaged
52. Dibromomethane	0.23970	0.21247	0.21247	0.010	-11.35898	40.00000	Averaged
53. 1,4-Dioxane	0.00634	0.00653	0.00653	0.000	2.95924	40.00000	Averaged
54. Bromodichloromethane	0.46274	0.41117	0.41117	0.010	-11.14472	40.00000	Averaged
55. 2-Chloroethyl vinyl ether	0.23855	0.23045	0.23045	0.010	-3.39685	40.00000	Averaged
56. *cis*-1,3-Dichloropropene (xt)	0.54372	0.48707	0.48707	0.010	-10.41955	40.00000	Averaged
57. 4-Methyl-2-pentanone	0.59436	0.55308	0.55308	0.010	-6.94548	40.00000	Averaged
58. Toluene (*)	0.88330	0.81971	0.81971	0.010	-7.19920	20.00000	Averaged
60. trans-1,3-Dichloropropene (x)	0.48155	0.44470	0.44470	0.010	-7.65230	40.00000	Averaged
61. 1,1,2-Trichloroethane	0.29117	0.27479	0.27479	0.010	-5.62833	40.00000	Averaged

Compound	RRF/Amount	RF20	CCAL RRF20	MIN RRF	%D/%Drift	Max%D/$Drift	Curve Type
62. Tetrachloroethene	0.43237	0.42795	0.42795	0.010	-1.02370	40.00000	Averaged
63. 1,3-Dichloropropane	0.53209	0.52244	0.52244	0.010	-1.81311	40.00000	Averaged
64. 2-Hexanone	0.48610	0.48886	0.48886	0.010	0.56779	40.00000	Averaged
65. Dibromochloromethane	0.36707	0.35835	0.35835	0.010	-2.37560	40.00000	Averaged
66. 1,2-Dibromoethane	0.35029	0.34097	0.34097	0.010	-2.66247	40.00000	Averaged
67. 1-Chlorohexane	0.52539	0.52604	0.52604	0.010	0.12412	40.00000	Averaged
68. Chlorobenzene (**)	0.98165	0.97600	0.97600	0.300	-0.57501	40.00000	Averaged
69. Ethylbenzene (*)	0.55007	0.54904	0.54904	0.010	-0.18763	20.00000	Averaged
70. 1,1,1,2-Tetrachloroethane	0.36378	0.34529	0.34529	0.010	-5.08217	40.00000	Averaged
71. Xylenes (m,p)	0.67749	0.67448	0.67448	0.010	-0.44459	40.00000	Averaged
72. Xylene (o)	0.64146	0.64343	0.64343	0.010	0.30744	40.00000	Averaged
73. Styrene	1.05170	1.01640	1.01640	0.010	-3.35604	40.00000	Averaged
74. Bromoform (**)	0.31021	0.29775	0.29775	0.010	-4.01697	40.00000	Averaged
75. Isopropylbenzene	1.77659	1.75886	1.75886	0.010	-0.99792	40.00000	Averaged
77. Bromobenzene	0.74579	0.75576	0.75576	0.010	1.33683	40.00000	Averaged
78. 1,1,2,2-Tetrachloroethane(*	0.93892	0.97358	0.97358	0.300	3.69186	40.00000	Averaged
79. n-Propylbenzene	3.81859	3.89748	3.89748	0.010	2.06583	40.00000	Averaged
80. 1,2,3-Trichloropropane	0.32454	0.31495	0.31495	0.010	-2.95645	40.00000	Averaged
81. 2-Chlorotoluene	2.11588	2.17685	2.17685	0.010	2.88168	40.00000	Averaged
82. 1,3,5-Trimethylbenzene	2.62692	2.60407	2.60407	0.010	-0.86973	40.00000	Averaged
83. 4-Chlorotoluene	2.43252	2.47865	2.47865	0.010	1.89658	40.00000	Averaged
84. T-Butylbenzene	2.29632	2.38196	2.38196	0.010	3.72921	40.00000	Averaged
85. 1,2,4-Trimethylbenzene	2.53873	2.59629	2.59629	0.010	2.26723	40.00000	Averaged
86. sec-Butylbenzene	3.43305	3.46989	3.46989	0.010	1.07319	40.00000	Averaged
87. 1,3-Dichlorobenzene	1.40547	1.45803	1.45803	0.010	3.73953	40.00000	Averaged
88. p-Isopropyltoluene	2.87153	2.89984	2.89984	0.010	0.98605	40.00000	Averaged
89. 1,4-Dichlorobenzene	1.43088	1.43038	1.43038	0.010	-0.03493	40.00000	Averaged
90. N-Butylbenzene	2.62902	2.67808	2.67808	0.010	1.86624	40.00000	Averaged
91. 1,2-Dichlorobenzene	1.31531	1.30647	1.30647	0.010	-0.67216	40.00000	Averaged
92. 1,2-Dibromo-3-chloropropane	0.27280	0.25308	0.25308	0.010	-7.22822	40.00000	Averaged
93. 1,2,4-Trichlorobenzene	0.84841	0.80204	0.80204	0.010	-5.46552	40.00000	Averaged
94. Hexachlorobutadiene	0.49194	0.45181	0.45181	0.010	-8.15651	40.00000	Averaged
95. Naphthalene	2.71843	2.54349	2.54349	0.010	-6.43538	40.00000	Averaged
96. 1,2,3-Trichlorobenzene	0.78005	0.72478	0.72478	0.010	-7.08498	40.00000	Averaged
97. 1,2-Dichloroethene (Total)	0.39434	0.35143	0.35143	0.010	-10.88090	40.00000	Averaged
98. Xylenes (Total)	0.66548	0.66413	0.66413	0.010	-0.20330	40.00000	Averaged

Average %D/Drift Results.

Calculated Average %D/Drift = 4.76143

Maximum Average %D/Drift = 15.00000

* Passed Average %D/Drift Test.

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