Developing an eMethod for the Analysis of Volatile Organic Compounds in Water Using Purge and Trap/GC with Agilent's New 5975 inert Mass Spectrometer

Application

Environmental

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Abstract

Using Agilent's new G1701DA (Version 2.0.00) software, it takes only a few steps to package a gas chromatography/mass spectrometry (GC/MS) method into one that is easily transferred to other similarly configured GC/MS systems. eMethods are truly portable, making it easy to share methods between instruments worldwide without the tedious process of entering all the parameters each time. Thanks to Retention Time Locking (RTL), calibration files (complete with retention time windows) or complex SIM (Selected Ion Monitoring) methods can be transferred without the need for retention time edits. This application note describes a method for the analysis of volatile organic compounds (VOCs) in water samples according to US Environmental Protection Agency (USEPA) method 8260B. Though framed around Method 8260B, it can be used for most VOC analyses requiring Purge and Trap (P&T)/GC/MS instrumentation. The eMethod can be downloaded directly from the Agilent Web site at the following location: www.agilent.com/chem/eMethods. Using the new "eMethod Import" ChemStation feature, one can load the method and all of its parameters. Although the purge-and-trap parameters are not automatically installed, the P&T method is included with the eMethod.

Introduction

GC/MS methods are often developed on one instrument, published, and then replicated on other instruments in different laboratories. In the past, this required new users to input all of the GC and MS parameters, including calibration or SIM tables. Even then, retention times (RTs) would generally differ among instruments so calibration and/or SIM tables would have to be redone. While it has been possible to copy a method to electronic media and copy it to another system, any differences in instrument configuration complicated the process.

A novel feature of the new Agilent G1701DA (Version 2.0.00) software is the ability to export and import complete GC/MS methods. All electronic parameters, including all GC and MS setpoints, calibration tables, SIM tables, and RTL calibration files, are exported as part of the eMethod. eMethods can be distributed over the Web, by e-mail, or on storage media. Installation takes just a minute or so. Normally, new users would first relock the method and then recalibrate. There is a "Notes" section for the method developer to specify nonelectronic parameters such as the type of inlet liner used.

This application note describes an eMethod for the analysis of 60 VOCs in water using a Velocity XPT purge-and-trap (P&T) sample concentrator together with an Agilent 6890N GC and new 5975 inert MSD. It includes a calibration table in the scan mode with appropriate target and qualifier ions, locked RTs for all analytes, RTL calibration files, and a complementary SIM method for use in the synchronous SIM/scan mode.

U.S. EPA Method 8260B [1] is a general purpose method for the analysis of VOCs in matrices such as ground and surface water, sludges, soils and sediments, filter cakes, spent carbons, and spent catalysts. This method is only used for the analyses of target VOCs by GC with mass spectral detection (GC/MS). It refers analysts to other U.S. EPA sample introduction methods that are appropriate for the matrix to be analyzed. Method 8260B is widely used in environmental laboratories with P&T for the analysis of VOCs in surface, ground, and wastewater samples. A similar method for the analysis of drinking water is described in EPA Method 524.2 [2].

Previous application notes in this series have discussed procedures for tuning to the USEPA's BFB requirements [3] and techniques for optimizing P&T/GC/MS methods [4]. This application note includes some additional insights into method optimization, as well as more recent calibration data that are included with the eMethod.

Experimental

Chemical Standards, Reagents, and Vials

High-purity B&J brand methanol was obtained from Honeywell Burdick & Jackson Co. (Muskegon, MI). Standard mixtures used for the preparation of calibration samples, spiking solutions, tune evaluation, and stability test samples were purchased from AccuStandard (New Haven, CT). These include the following: Part No. M-502-10X-Pak containing 60 VOC target analytes at 2000 µg/mL each in methanol; and p/n M-8260A/B-IS/SS-10X-PAK containing p-bromofluorobenzene, chlorobenzene-d5, dibromofluoromethane, 1,4-dichlorobenzene-d4, 1,2-dichloroethane-d4, fluorobenzene, and toluene-d8 at 2000 µg/mL each in methanol. VOC-free water was used for the preparation of standards and test samples. TraceClean 40-mL (nominal volume, actual volume is 43 mL) VOA vials (p/n 15900-022) were purchased from VWR Scientific (West Chester, PA).

Preparation of Calibration and Spiking Solutions

Secondary spiking solutions were prepared in methanol for each calibration level so that a 100-mL volumetric flask could be spiked with 25 µL of the calibration solution (containing 60 VOCs) and 25 µL of the internal standard/surrogate mixture. Each volumetric flask was inverted five times to mix the solution, which was then carefully poured into two 43-mL VOA vials.

Table 1 provides details on how the seven calibration standards were prepared. The combined internal standard and surrogate spiking solution was prepared by diluting 40 µL of the 2000 µg/mL standard to 1.0 mL with methanol. Each sample and standard was spiked at 20 µg/L with this solution.

Instrumentation and Analytical Conditions

The P&T instrumentation and setpoints are listed in Table 2. Since the P&T instrument is controlled by separate software, its parameters cannot be set

Table 1. Procedure for Preparing Calibration Samples

A Calibration level (µg/L)*	B Volume of 2000 µg/mL VOC standard (µL)**	C Diluted to this volume in methanol (mL)***	D Results in this secondary concentration (ng/µL)****	E Amount to spike into 100 mL volumetric flask (µL)*****
1	50	25	4	25
2	40	10	8	25
5	50	5	20	25
20	40	1	80	25
50	40	0.4	200	25
100	40	0.2	400	25
200	40	0.1	800	25

* Concentration of each analyte in the final aqueous calibration solution.

** Volume of the 2000 µg/mL 60-component VOC standard solution, which was diluted to the volume shown in column C.

*** Final volume of VOC solution after dilution in methanol.

**** Concentration of the calibration spiking solution prepared by diluting the amount of 2000 µg/mL standard in column B to the volume shown in column C.

***** Amount of the secondary standard solution (column D) added to 100-mL of water to prepare the calibration standard at the level shown in column A.

while importing this eMethod and must be entered manually. The following P&T options were not used: DryFlow trap, automatic internal standard addition, sample heating, dry purging, and sample cryofocusing. The method shown in Table 2 was originally derived using the wizard that is provided in the TekLink 2.4 P&T control software. Minor modifications were made.

As shown in Table 3, toluene-d8 was used as the RTL compound. Its RT was locked to 7.405 min in the constant flow mode. The constant flow mode was chosen for this application because it helps heavier compounds elute faster and at lower temperatures, which makes the method cycle shorter. While RTL works well in the constant flow mode, it does not compensate for large differences in column length as well as it does in the constant pressure mode. Therefore, it is best to install a new 20-m × 0.18-mm × 1.0 µm DB-VRX column or one that has not been subjected to frequent column cutting. Once installed for this application, there is little need for column maintenance.

Table 2. Purge and Trap Instrumentation and Setpoints

P&T instrument	**Teledyne Tekmar Velocity XPT**
Automatic sampler	**Teledyne Tekmar Aquatek 70**
Software control	**Teledyne Tekmar VOC Teklink version 2.4**
Trap	Vocarb 3000 (Agilent p/n 5182-0775)
P&T-GC interface	P&T transfer line spliced into the GC split/splitless inlet carrier gas line and GC carrier gas plumbed to the Velocity XPT
Sample size	5 mL
Valve oven temperature	150 °C
Transfer line temperature	150 °C
Sample mount temp	90 °C
Purge ready temp	45 °C
DryFlow standby temperature	60 °C
Standby flow	20 mL/min
Pressurize time	0.25 min
Fill IS time	0.00 (internal standards added by hand)
Sample transfer time	0.35 min
Pre-purge time	0.00 min
Pre-purge flow	40 mL/min
Sample heater	Off (Samples not heated)
Sample preheat time	1.00 min
Preheat temperature	40 °C
Purge time	11.00 min
Purge temperature	0 °C (that is, less than the purge ready temp of 45 °C)
Purge flow	40 mL/min
Purge rinse time	0.40 min
Purge line time	0.35 min
Dry purge time	0.00 min (Dry purge not used)
Dry purge temp	40 °C
Dry purge flow	200 mL/min
GC start	Start of desorb
Desorb preheat temperature	245 °C
Desorb drain	On
Desorb time	2.00 min
Desorb temperature	250 °C
Desorb flow	300 mL/min
Bake rinse	On
Number of bake rinses	3
Bake drain time	0.50 min
Bake drain flow	400 mL/min
Bake time	3.00 min
Bake temperature	270 °C
DryFlow bake temperature	300 °C
Bake flow	400 mL/min
Focus temperature	Not used
Inject time	1.00 min
Inject temperature	180 °C
Standby temperature	100 °C

Table 3. Lists the Conditions for the GC/MS System

Gas chromatograph	**Agilent 6890N**
Inlet	Split/Splitless
Inlet liner	Single taper, deactivated (Agilent p/n 5181-3316)
Inlet temperature	150 °C
Split ratio	50:1
Column	20 m × 0.18 mm × 1.0 µm DB-VRX (Agilent p/n 121-1524)
Carrier gas	Nominal helium flow at 1.0 mL/min constant flow
RTL	Toluene-d8 retention time locked to 7.405 min
Oven temperature program	40 °C (3 min), 10 °C/min to 100 °C (0 min), 25 °C/min to 225 °C (3 min)
Mass spectrometer	**Agilent 5975 inert MSD**
Transfer line temperature	225 °C
Quad temperature	150 °C
Source temperature	230 °C
EM voltage	1200 volts
Scan range	35–260 u
Threshold	150
Samples	2
Solvent delay	0 min
Software	MSD Productivity ChemStation Software (p/n G1701DA version D.02.00)

Results and Discussion

According to section 1.3 of the method, 8260B can be used to quantify most VOCs that have boiling points below 200 °C. It lists 123 compounds that can be determined by the method using various sample prep and sample introduction methods. Of these, seven are internal standards or surrogates, nine are not recommended for P&T sample introduction, and three must be purged at 80 °C for efficient recovery. For this study, the 60 VOCs listed in EPA Method 502.2 [5] were analyzed along with three internal standards and four surrogates (Table 4).

Calibration Results

Many laboratories employing Method 8260B or similar methods generate five-point calibrations curves between 5 and 200 µg/L. In a previous application note [4], calibration from 1 to 300 µg/L gave response factor (RF) %RSDs less than 15% for all but eight compounds, while calibrations from 1–200 µg/L all fell under 15%. For the eMethod described in this application note, calibration standards were run at 1, 2, 5, 20, 50, 100, and 200 µg/L. The signals for all analytes at 1 µg/L were sufficient to allow calibration at even lower levels. Figure 1 shows a total ion chromatogram (TIC) of the targets, surrogates, and internal standards at 20 µg/L each.

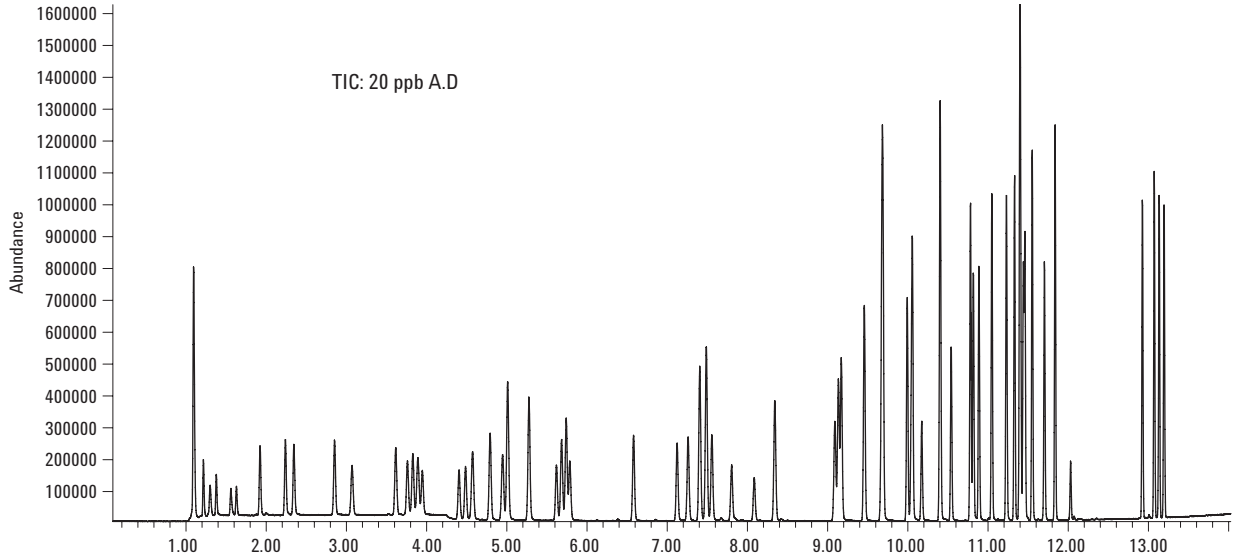

Figure 1. Chromatogram of the 60 VOCs, three internal standards, and four surrogates listed in Table 4. The standard was analyzed using the eMethod described herein.

The average RF and %RSD of the RFs were calculated for each compound over the 1–200 µg/L range. As seen in Table 4, all five of the system performance check compounds (SPCCs) exceeded their minimum RFs by a comfortable margin. In addition, all six of the continuing calibration compound (CCC) RF RSDs were significantly less than the 30% limit specified in EPA Method 8260B. The RF RSDs for all target compounds fell well below 15% over the entire 1–200 µg/L calibration range, allowing the use of average RF values for calibration. The average RF for all compounds was 4.3% over the entire 1–200 µg/L range.

Table 4. Compound List with Average RF and the RF %RSDs for a Seven-Level Calibration from 1 to 200 µg/L

Type*	Compound	RT (min)	Minimum average RF**	Minimum %RSD of Calib. RF***	Average RF 1–200 µg/L	RF %RSD 1–200 µg/L
ISTD	Fluorobenzene	5.277		15		
T	Dichlorodifluoromethane	1.219		15	0.283	7.00
T,SPCC	Chloromethane	1.301	0.1	15	0.259	8.15
T,CCC	Vinyl chloride	1.379		30	0.240	3.36
T	Bromomethane	1.557		15	0.144	10.74
T	Ethyl chloride	1.63		15	0.151	3.54
T	Trichloromonofluoromethane	1.928		15	0.374	2.98
T,CCC	1,1-dichloroethene	2.24		30	0.321	3.14
T	Methylene chloride	2.346		15	0.306	9.32
T	1,2-dichloroethene (E)	2.857		15	0.322	2.45
T,SPCC	1,1-dichloroethane	3.074	0.1	15	0.406	2.94
T	*cis*-1,2-Dichloroethene	3.615		15	0.313	3.23
T	Bromochloromethane	3.757		15	0.198	3.13
T,CCC	Chloroform	3.833		30	0.399	3.62
T	2,2-Dichloropropane	3.891		15	0.339	7.88
Sur	1,2-Dichloroethane-d4	4.404		15	0.288	1.26
Sur	Dibromofluoromethane	3.947		15	0.225	0.77
T	1,2-Dichloroethane	4.491		15	0.346	3.32
T	1,1,1-Trichloroethane	4.574		15	0.398	3.18
T	1,1-Dichloro-1-propene	4.789		15	0.346	3.08
T	Carbon tetrachloride	4.948		15	0.359	4.91
T	Benzene	5.012		15	1.038	2.75
T	Dibromomethane	5.626		15	0.216	2.90
T,CCC	1,2-Dichloropropane	5.682		30	0.262	3.38
T	Trichloroethylene	5.743		15	0.305	3.45
T	Bromodichloromethane	5.787		15	0.328	4.46
T	1,3-Dichloropropene (Z)	6.579		15	0.407	3.84
T	1,3-Dichloropropene (E)	7.126		15	0.357	4.89
T	1,1,2-Trichloroethane	7.263		15	0.196	3.87
Sur	Toluene-d8	7.407		15	0.941	0.37
T,CCC	Toluene	7.489		30	1.109	3.20
T	1,3-Dchloropropane	7.558		15	0.421	3.54
T	Dibromochloromethane	7.804		15	0.269	6.57
T	1,2-Dibromoethane	8.088		15	0.259	3.52
T	Tetrachloroethylene	8.345		15	0.357	3.57
T	1,1,1,2-Tetrachloroethane	9.092		15	0.269	4.84
ISTD	Chlorobenzene-d5	9.134		15		
T,SPCC	Chlorobenzene	9.173	0.3	15	0.984	5.09
T,CCC	Ethylbenzene	9.46		30	1.618	5.14
T	m- and p-Xylene	9.683		15	2.644	3.87
T,SPCC	Bromoform	9.669	0.1	15	0.288	11.12
T	Styrene	9.993		15	1.034	6.37

Table 4. Compound List with Average RF and the RF %RSDs for a Seven-Level Calibration from 1 to 200 µg/L (continued)

Type*	Compound	RT (min)	Minimum average RF**	Minimum %RSD of Calib. RF***	Average RF 1–200 µg/L	RF %RSD 1–200 µg/L
T,SPCC	1,1,2,2-Tetrachloroethane	10.043	0.3	15	0.435	4.64
T	o-Xylene	10.057		15	1.324	4.84
T	1,2,3-Trichloropropane	10.174		15	0.378	4.18
Sur	p-Bromofluorobenzene	10.399		15	0.482	2.51
T	Isopropylbenzene	10.405		15	1.591	5.33
T	Bromobenzene	10.539		15	0.628	4.55
T	n-Propylbenzene	10.782		15	2.039	4.88
T	2-Chlorotoluene	10.815		15	1.163	5.20
T	4-Chlorotoluene	10.885		15	1.234	4.73
T	1,3,5-Trimethylbenzene	11.047		15	1.438	5.61
T	*tert*-Butylbenzene	11.228		15	1.371	4.90
T	1,2,4-Trimethylbenzene	11.331		15	1.513	5.55
T	sec-Butylbenzene	11.395		15	2.026	4.29
T	1,3-Dichlorobenzene	11.407		15	0.925	4.36
ISTD	1,4-Dichlorobenzene-d4	11.44		15		
T	1,4-Dichlorobenzene	11.46		15	1.530	3.04
T	p-isopropyltoluene	11.552		15	2.843	3.48
T	1,2-Dichlorobenzene	11.705		15	1.459	3.19
T	Butylbenzene	11.836		15	2.648	3.56
T	1,2-Dibromo-3-chloropropane	12.031		15	0.235	4.94
T	1,2,4-Trichlorobenzene	12.926		15	1.361	3.25
T	Naphthalene	13.071		15	3.267	3.66
T	Hexachlorobutadiene	13.136		15	0.939	3.20
T	1,2,3-Trichlorobenzene	13.194		15	1.311	2.57
	Average %RSD of Targets					**4.31**

* Compound designations as follows: T (target); SPCC (system performance check compound); CCC (calibration check compound); Surr (surrogate); ISTD (internal standard). Target compounds may also be designated as SPCCs or CCCs.

** The minimum average RF that must be met for the SPCCs.

*** The minimum %RSD of the RFs. If any one or more of the CCC RF RSDs exceeds 30%, instrument maintenance is required. If the RF %RSD for any target compound exceeds 15%, other curve fits must be substituted for the average RF.

The Importance of an Inert Flow Path

The initial calibration for this method resulted in 6 compounds having relative RF %RSDs greater than 15% and a total of 15 with double digit %RSDs. The worst performers were (E) and (Z) 1,3-dichloropropene; dibromochloromethane; bromoform; 1,1,2,2-tetrachloroethane; and 1,2-dibromo-3-chloropropane with RRF RSDs averaging 28%. After replacing the old GC column and inlet liner (descriptions in Table 3), the average RSD fell to 6% for these difficult compounds. All RF %RSDs were well under 15% with an average of 4.3% for all 59 calibrated peaks (m- and p-xylene calibrated together). The results are shown in Table 4. A distribution of the %RSD values is shown graphically in Figure 2.

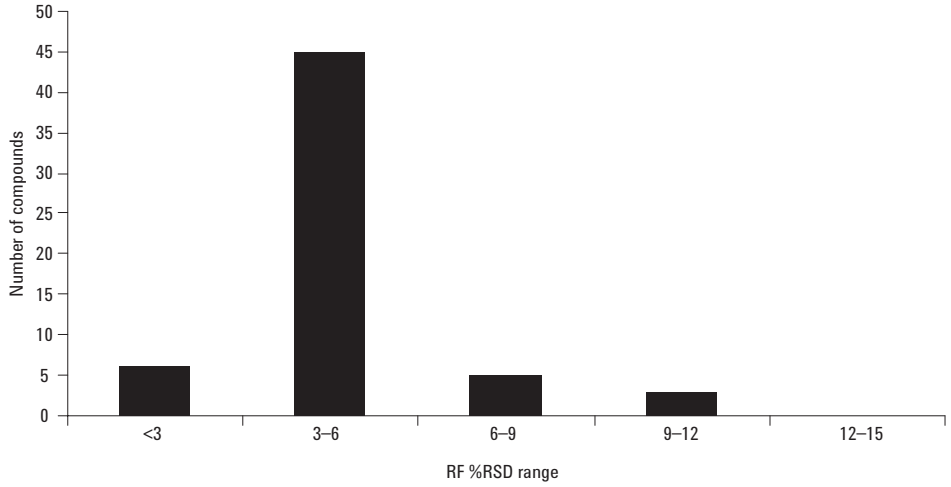

Figure 2. Distribution of the RF RSDs for the 59 calibrated peaks (m- and p-xylene are not resolved).

As discussed, some VOCs are particularly susceptible to active sites in the flow path. One manifestation of this problem is that the calibration curve is nonlinear (Figure 3). Ideally, the RRF should be identical at each calibration level, giving a straight horizontal line. However, when an analyte is adsorbed or decomposed by active sites, the RFs fall off as the concentration goes down.

An easy way to visualize this problem is to consider the illustration in Figure 4. First, assume that there are five active sites somewhere in the sample flow path (Figure 4a). Let us also assume that a high-level calibration standard contains 500 molecules of compound X and that "X" is susceptible to adsorption or decomposition by these active sites. In this case, there is a risk of losing 5 of the 500 molecules, which would reduce the RF by just 1%. Next, assume that a low-level calibration standard contains just 10 molecules of compound X. In this case, one could lose as much as 50% of the analyte, cutting the RF in half (Figure 4b). Thus, as the calibration level for compound X goes down, the response factor falls off, leading to a nonlinear calibration curve such as the bromoform curve shown in Figure 3.

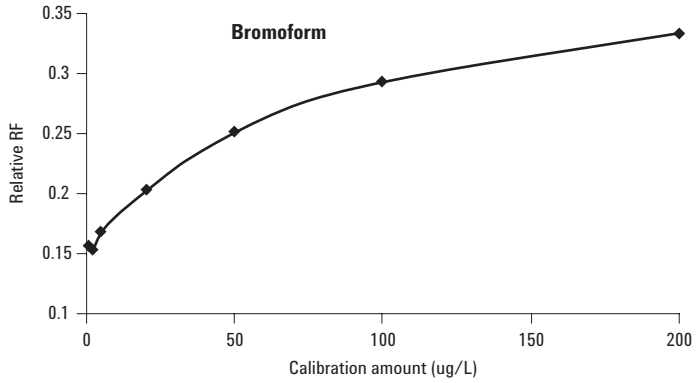

Figure 3. An example of a nonlinear calibration curve for bromoform caused by adsorption by active sites in the P&T/GC/MS flow path.

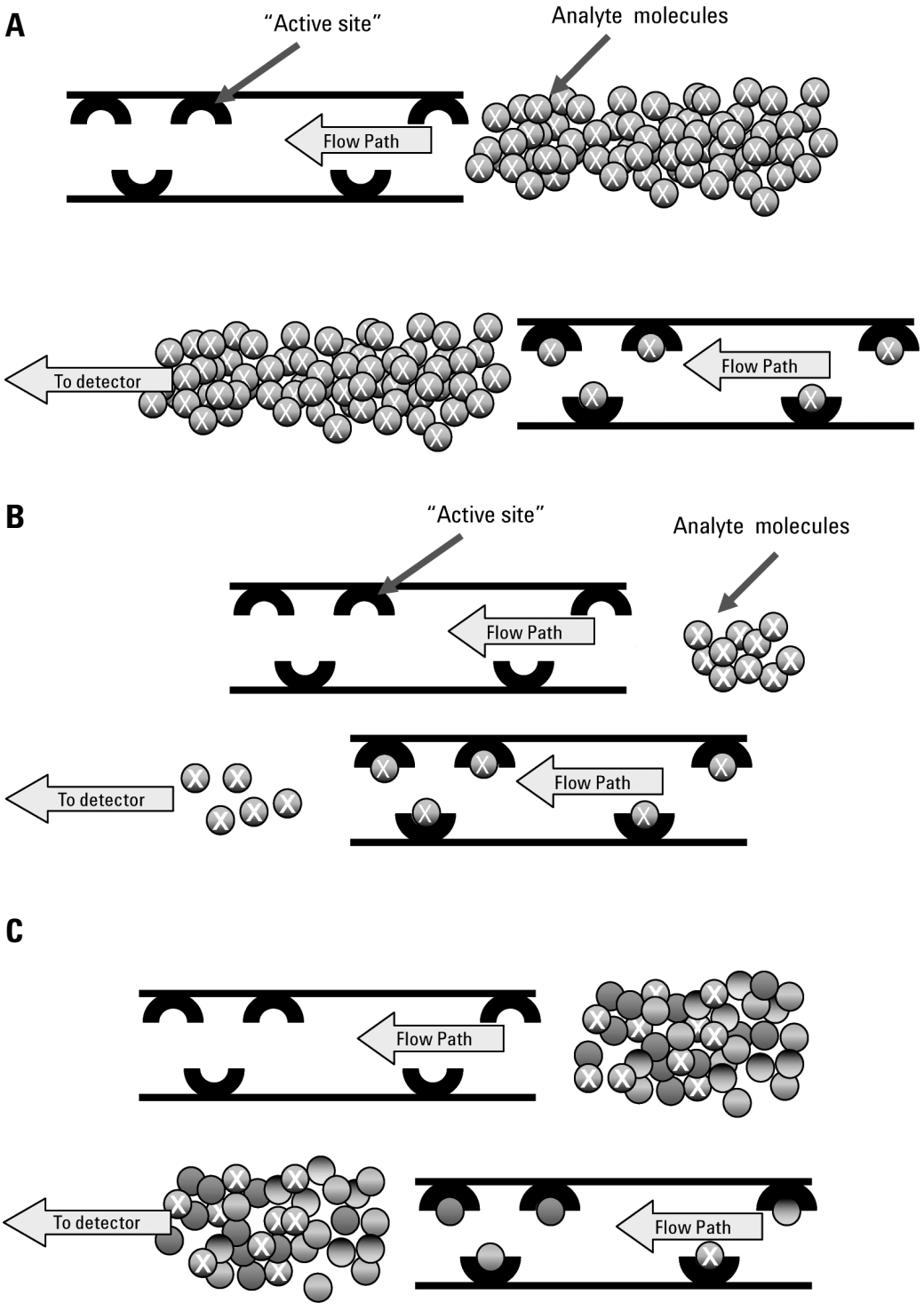

Figure 4. **An illustration showing how "active sites" in the P&T/GC/MS flow path can affect the amount of analyte (compound "X") reaching the detector. A) An example showing how just a small fraction of a high concentration analyte is lost to a small number of active sites. B) An example showing how a large fraction of a low concentration analyte can be lost to a small number of active sites. C) An example showing how analytes may compete for active sites. Very little of compound X is lost due to competition from high concentrations of other analytes.**

Figure 4c illustrates another manifestation of these active site problems. Assume that there are still five active sites and 10 molecules of compound X. However, this time there are other molecules present at high levels, which compete for the same active sites. With a limited number of active sites and competition from other compounds, most of the X molecules reach the detector. This explains why analyte or surrogate recoveries are sometimes low in relatively clean samples and higher in the presence of other analytes.

Active sites can appear anywhere in the flow path of the P&T/GC/MS system. If compounds begin to adsorb, the solution is to replace those components of the flow path that cause the problem. In this case, replacing the column and inlet liner restored instrument performance.

SIM/Scan Method

EPA methods for volatiles analysis do not mention the use of synchronous SIM/scan methods. However, it is possible to obtain SIM and scan data in the same analysis with virtually no sacrifice in sensitivity in each mode. After creating the scan method, a SIM method was created using the ChemStation's AutoSIM tool. The default AutoSIM settings were used.

Figure 5 shows extracted ion chromatograms (EICs) (SIM and scan from a SIM/scan run) for the six gases that are part of many VOC methods. In general, these six compounds give the lowest average RFs among the 60 analytes used for this work. Of these, ethyl chloride is often the least responsive.

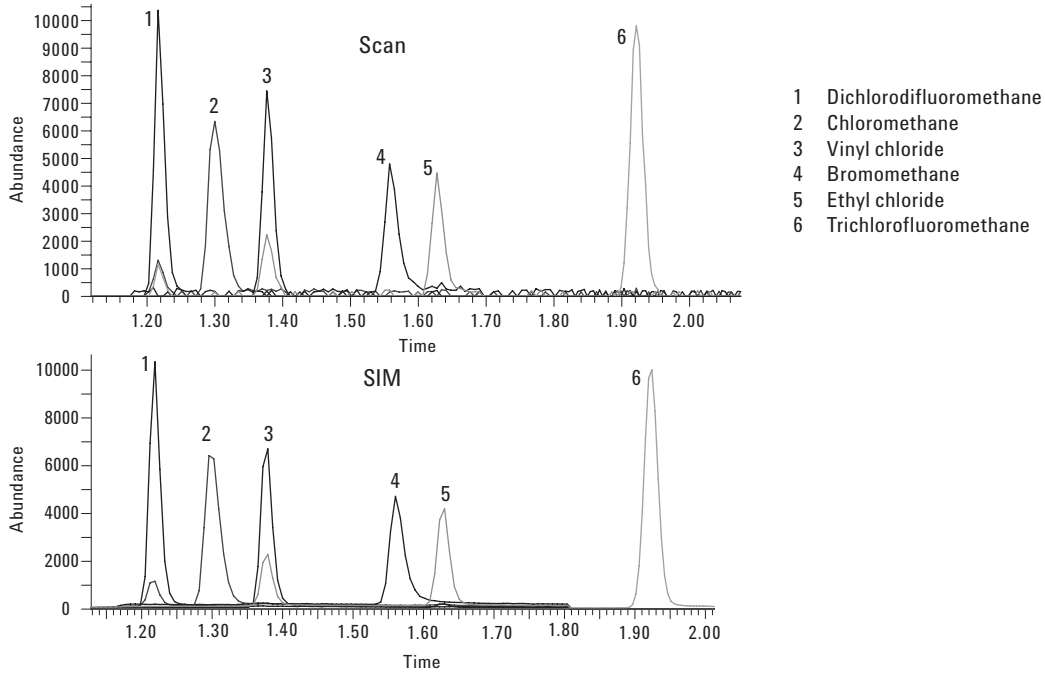

Figure 5. EICs for the six analytes that are gases at standard temperature and pressure. Both the scan and SIM chromatograms were obtained in a single synchronous SIM/scan run.

The sensitivity of the scan-only method was compared to the SIM/scan mode by measuring the signal-to-noise (S/N) ratio for ethyl chloride. As seen in Table 5, the S/N ratio for ethyl chloride is approximately the same for scan runs, whether run alone or as part of a SIM/scan acquisition. A sampling rate of 4 (n = 2) was used in each case. The only difference between these two scan runs is that the scan rate is slower in the SIM/scan mode than in the scan-only mode. Nevertheless, all peaks were defined by at least eight scans, making accurate quantitation possible. As expected, SIM provided a 10-fold improvement in sensitivity over scan. Using the Agilent 5975 inert MSD, one can run in the SIM/scan mode with no loss of scan sensitivity and obtain a SIM chromatogram with 10X greater sensitivity "for free". The only trade-off is in the scan rate, but the rate is still sufficient for quantification. The number of SIM and Scan acquisitions across each peak can be increased without loss of sensitivity by reducing the sample rate from 2^2 to 2^1.

Table 5 shows a comparison of the signal-to-noise (S/N) ratio for ethyl chloride (using *m/z* 64) analyzed by a scan method and a synchronous SIM/scan method. The S/N ratio has been calculated using two different noise measurement methods – root mean squared (RMS) and peak-to-peak.

Table 5. S/N Comparison Between SIM and Scan in SIM/Scan Run

	RMS S/N	Peak/Peak S/N
SIM (SIM/Scan)	749	254
Scan (SIM/Scan)	75	20
Scan (Scan only)	73	17

Figure 6 compares the total ion current chromatograms for the synchronous SIM and scan analysis of the 60 target VOCs (1 ppb each), internal standards (20 ppb), and surrogates (20 ppb) shown in Table 4.

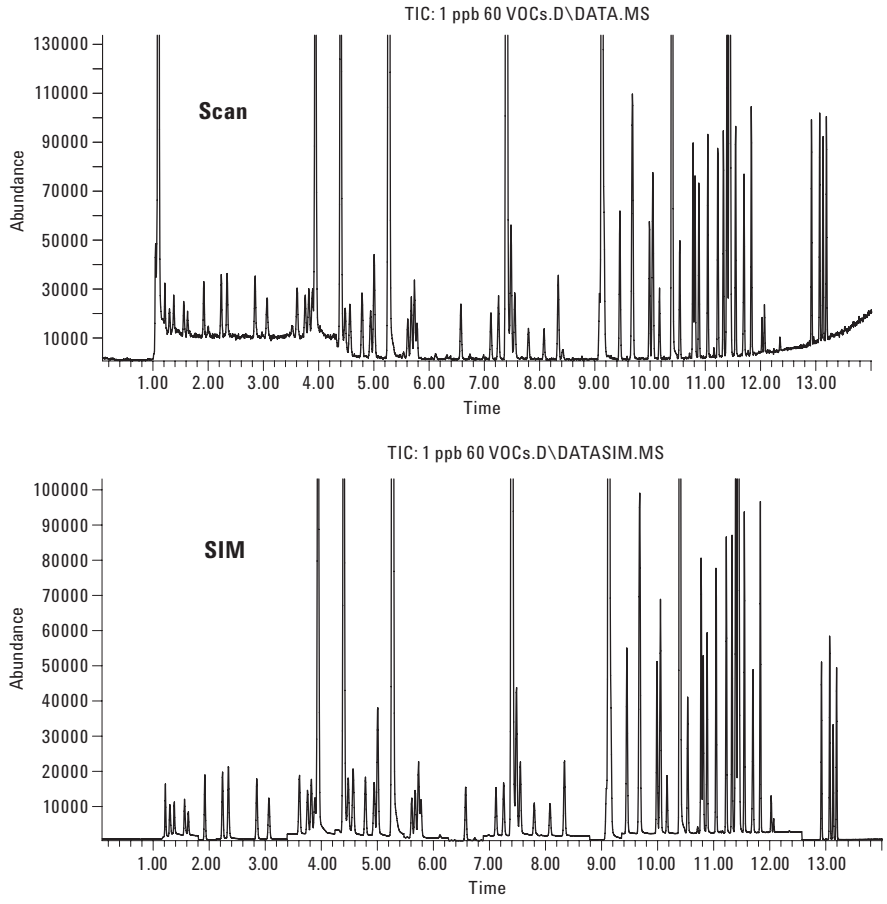

Figure 6. TICs for the SIM and scan chromatograms obtained from a single synchronous SIM/scan analysis of the 67 compounds shown in Table 4.

Conclusions

While the benefit of sharing GC/MS methods is clear, the process of replicating them on various instruments has been tedious and time-consuming. Now, the Agilent eMethod software with its "Method Export" and "Method Import" wizards make GC/MS method transfer a trivial process.

Anyone interested in replicating this method can download all of the parameters from the Agilent Web site (www.agilent.com/chem/eMethods). Using the Method Import function of the Agilent G1701DA (version 2.0.00 or newer), the method can be installed immediately, complete with calibration tables and RTL calibration. The user would have to relock the method using toluene-d8 as the locking compound and run new calibration standards. Although the P&T setpoints are not automatically installed, they are included with the eMethod in the "notes" section.

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