# Analysis of Semivolatile Organic Compounds Using the Agilent Intuvo 9000 Gas Chromatograph

## Application Note

**Author**

Matthew Giardina, Ph.D.,
Applications Chemist,
Gas Phase Separations Division,
Agilent Technologies, Inc.

## Abstract

Gas chromatography/mass spectrometry is a workhorse for the analysis of semivolatile organic compounds in environmental samples. The Agilent Intuvo 9000 GC represents a significant advancement in gas chromatography, and provides a number of advantages for semivolatile analysis. This Application Note demonstrates the performance of the Intuvo 9000 GC for the analysis of semivolatiles based upon the specifications of USEPA method 8270D. Included in this study is an evaluation of the Intuvo during repetitive injections of a composite soil extract to gauge instrument durability under conditions modeling a high-throughput environmental laboratory.

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## Introduction

Gas chromatography/mass spectrometry (GC/MS) is widely regarded as the analytical technique of choice for the analysis of semivolatile organic compounds (SVOCs)[1]. A number of compounds within the class of SVOCs are considered environmental pollutants. Government agencies with regulatory authority have established methods and set performance criteria for the measurement SVOCs in a number of environmental and industrial matrices. For example, United States Environmental Protection Agency (USEPA) method 8270D contains a list of 243 compounds that are suitable for analysis by GC/MS in solid waste, soil, air and water extracts[2]. Method 8270D contains detailed specifications and requirements for the quantitative analysis of SVOCs.

The durability and data quality from the Agilent 7890 GC has set the standard for the analysis of SVOCs and these instruments are used in environmental testing laboratories throughout the world. With the development of new technology, it is of critical importance to demonstrate that the next generation of GC instrumentation can meet the rigors of SVOC analysis for environmental testing.

The Agilent Intuvo 9000 GC is a step forward in the advancement of gas chromatography, and includes a number of design innovations making it ideally suited for SVOC analysis[3]. Intuvo Flow Technology (IFT) incorporates the use of an easy to install and replace Intuvo Column and Guard Chip which acts as a precolumn to prevent particulate and nonvolatile contamination of the column and flow path[4]. Direct heating technology reduces power requirements, and facilitates more rapid column cooling for faster cycle time.

This Application Note demonstrates that the Intuvo 9000 GC can easily achieve the rigorous performance specifications established in USEPA method 8270D, while maintaining equivalent retention time, resolution, and analyte response of the benchmark 7890 GC. In addition, the system durability is demonstrated by the repetitive injection of a composite soil extract.

# Experimental

## Standards and samples

A stock standard containing 77 target compounds and surrogates was purchased from AccuStandard (New Haven, CT). The standard was selected to provide a representative mixture of acids, bases, and neutrals. The stock standard was diluted in dichloromethane containing six internal standards purchased from Supelco (Bellefonte, PA). The nominal concentration of targets and surrogates in the calibration standards were 0.1, 0.2, 0.8, 1, 1.6, 2, 5, 10, 15, 20, 35, 50, 75, and 100 µg/mL. The concentration of the internal standards in each calibration standard was 40 µg/mL. Table 1 lists the compounds used in the study. The compound numbers in Table 1 were assigned based upon retention order of the targets and surrogates with the internal standards listed at the end of the table out of retention order. Compound numbers were assigned to reduce complexity of the graphs.

The tuning standard, containing a mixture of benzidine, pentachlorophenol, 4,4′-dichlorodiphenyltrichloroethane (4,4′-DDT), and decafluorotriphenylphosphine (DFTPP), was purchased from AccuStandard. The tuning standard was diluted in dichloromethane to a final concentration 25 µg/mL.

A composite mixture of soils extracted with dichloromethane prepared for method 8270 was donated from ESC Lab Sciences (Mt. Juliet, TN). The extracts selected for the composite mixture contained the heaviest matrix residue typically encountered in their laboratory.

Table 1. Target compounds, surrogates and internal standards.

| No. | Compound |
|---|---|
| 1 | N-Nitrosodimethylamine |
| 2 | Pyridine |
| 3 | 2-Fluorophenol (surrogate) |
| 4 | Phenol-$d_5$ (surrogate) |
| 5 | Phenol |
| 6 | Aniline |
| 7 | *Bis*(2-chloroethyl) ether |
| 8 | 2-Chlorophenol |
| 9 | 1,3-Dichlorobenzene |
| 10 | 1,4-Dichlorobenzene |
| 11 | Benzyl alcohol |
| 12 | 1,2-Dichlorobenzene |
| 13 | 2-Methylphenol |
| 14 | *Bis*(2-chloroisopropyl) ether |
| 15 | 4-Methylphenol |
| 16 | N-Nitrosodi-*n*-propylamine |
| 17 | Hexachloroethane |
| 18 | Nitrobenzene-$d_5$ (surrogate) |
| 19 | Nitrobenzene |
| 20 | Isophorone |
| 21 | 2-Nitrophenol |
| 22 | 2,4-Dimethylphenol |
| 23 | Benzoic acid |
| 24 | *Bis*(2-chloroethoxy)methane |
| 25 | 2,4-Dichlorophenol |
| 26 | 1,2,4-Trichlorobenzene |
| 27 | Naphthalene |
| 28 | 4-Chloroaniline |
| 29 | Hexachlorobutadiene |
| 30 | 4-Chloro-3-methylphenol |
| 31 | 2-Methylnaphthalene |
| 32 | Hexachlorocyclopentadiene |
| 33 | 2,4,6-Trichlorophenol |
| 34 | 2,4,5-Trichlorophenol |
| 35 | 2-Fluorobiphenyl (surrogate) |
| 36 | 2-Chloronaphthalene |
| 37 | 2-Nitroaniline |
| 38 | Dimethyl phthalate |
| 39 | 2,6-Dinitrotoluene |
| 40 | Acenaphthylene |
| 41 | 3-Nitroaniline |
| 42 | Acenaphthene |
| 43 | 2,4-Dinitrophenol |
| 44 | 4-Nitrophenol |
| 45 | 2,4-Dinitrotoluene |
| 46 | Dibenzofuran |
| 47 | Diethyl phthalate |
| 48 | 4-Chlorophenyl-phenyl ether |
| 49 | Fluorene |
| 50 | 4-Nitroaniline |
| 51 | 4,6-Dinitro-2-methylphenol |
| 52 | N-Nitrosodiphenylamine |
| 53 | Azobenzene |
| 54 | 2,4,6-Tribromophenol (surrogate) |
| 55 | 4-Bromophenyl phenyl ether |
| 56 | Hexachlorobenzene |
| 57 | Pentachlorophenol |
| 58 | Phenanthrene |
| 59 | Anthracene |
| 60 | Carbazole |
| 61 | Di-*n*-butylphthalate |
| 62 | Fluoranthene |
| 63 | Benzidine |
| 64 | Pyrene |
| 65 | *p*-Terphenyl-$d_{14}$ |
| 66 | Butylbenzylphthalate |
| 67 | 3,3′-Dichlorobenzidine |
| 68 | Benzo[a]anthracene |
| 69 | *Bis*(2-ethylhexyl)phthalate |
| 70 | Chrysene |
| 71 | Di-*n*-octyl phthalate |
| 72 | Benzo[b]fluoranthene |
| 73 | Benzo[k]fluoranthene |
| 74 | Benzo[a]pyrene |
| 75 | Indeno[1,2,3-cd]pyrene |
| 76 | Dibenzo[a,h]anthracene |
| 77 | Benzo[g,h,i]perylene |
| 78 | 1,4-Dichlorobenzene-$d_4$ (internal standard) |
| 79 | Naphthalene-$d_8$ (internal standard) |
| 80 | Acenaphthalene-$d_{10}$ (internal standard) |
| 81 | Phenanthrene-$d_{10}$ (internal standard) |
| 82 | Chrysene-$d_{12}$ (internal standard) |
| 83 | Perylene-$d_{12}$ (internal standard) |

### Instrument methods

The Agilent Intuvo 9000 GC was configured with a single MS flow path for interfacing to an Agilent 5977B MSD with an inert EI ion source and a 30 m Intuvo DB-5ms Ultra Inert column. For comparative data, an Agilent 7890 GC was also interfaced to a 5977B MSD with an inert EI ion source and a 30 m Agilent J&W DB-5ms Ultra Inert column. The same method parameters were used on both the Intuvo 9000 GC and the 7890 GC, with the exception of the Intuvo Guard Chip, which was not present on the 7890 GC. Table 2 lists the instrument conditions used during the study.

## Results and Discussion

### Equivalency to an Agilent 7980 GC

In addition to the many innovations in flow path and column heating technology that have been incorporated into the Intuvo 9000 GC, it has been designed to provide equivalent performance to the 7890 GC, with respect to chromatographic retention and response. This is particularly important for the analysis of SVOCs in environmental testing where the 7890 GC has set the performance benchmark.

As an initial validation of equivalency, a standard prepared with a target and surrogate concentration of 20 µg/mL, and an internal standard concentration of 40 µg/mL was injected on a 7890 GC coupled to a 5977B MSD and a 30 m Agilent J&W DB-5ms Ultra Inert column, and an Intuvo 9000 GC coupled to a 5977B MSD and a 30 m Intuvo DB-5ms Ultra Inert column. The same column temperature program and detector conditions were used for analysis (Table 2). Figure 1 shows a normalized total ion chromatogram obtained on both systems. The chromatograms are virtually indistinguishable with only slight differences in regions of high peak density (12.5 and 16.5 minutes).

Table 2. Common GC/MS Parameters.

| Parameter | Value |
|---|---|
| Injection volume | 1 µL |
| Inlet | Split/Splitless 300 °C<br>Pulsed splitless 60 psi to 0.5 minutes<br>Purge 50 mL/min at 0.5 minutes<br>Septum purge switched flow mode 3 mL/min |
| Liner | Agilent Ultra Inert splitless single taper liner with glass wool (p/n 5190-2293) |
| Intuvo Guard Chip | 60 °C for 2 minutes,<br>20 °C/min to 260 °C,<br>6 °C/min to 330 °C,<br>Hold 1.333 minutes (standards) or 10.333 minutes (soil extract) |
| Column | Agilent J&W DB-5ms and Intuvo DB-5ms UI 30 m × 0.25 mm, 0.5 µm (p/n 122-5536UI for 7890 and 122-5536UI-INT for Intuvo) |
| Flow | 2 mL/min constant flow |
| Column temperature | 40 °C for 2 minutes,<br>20 °C/min to 260 °C,<br>6 °C/min to 330 °C,<br>Hold 1.333 minutes (standards) or 10.333 minutes (soil extract) |
| Transfer line temperature | 330 °C |
| Drawout plate | 6 mm (optional) |
| Ion source temperature | 330 °C |
| Quadrupole temperature | 200 °C |
| Scan | 35 to 550 *m/z* |
| Gain factor | 1 |
| Threshold | 50 |
| A/D samples | 2 |

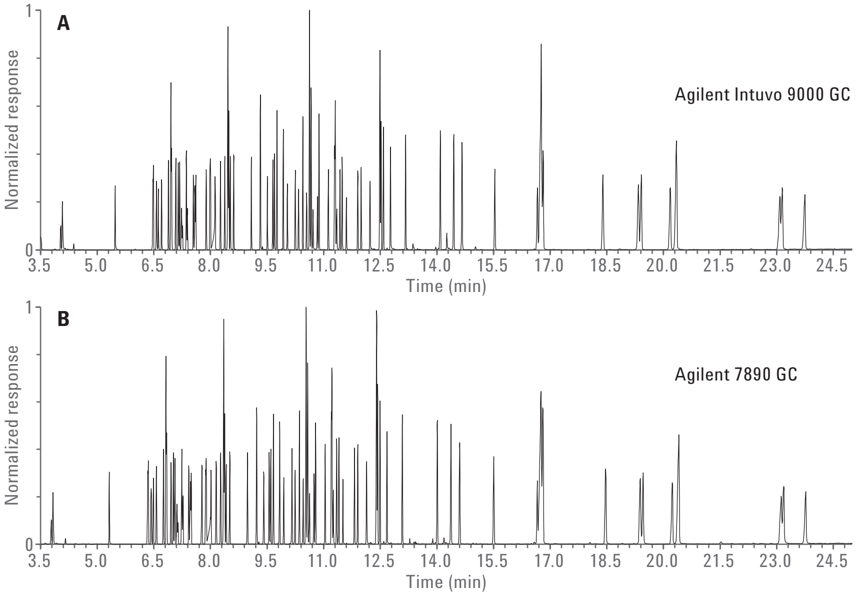

Figure 1. Comparison of SVOC chromatograms generated with an Agilent Intuvo 9000 GC (A) and an Agilent 7890 GC (B).

To provide a more quantitative comparison of retention, Figure 2 shows a plot of the relative retention times (that is, ratio of target and surrogate retention times to the internal standards) for the Intuvo 9000 GC and 7890 GC. The agreement between the two instruments was quite good. The average difference in relative retention time between the two instruments was 0.0006.

Figure 2 shows that the first two eluting compounds, N-nitrosodimethylamine and pyridine, show a greater degree of difference in relative retention time. This is a result of a small amount of precolumn volume introduced by the Intuvo Guard Chip. Figure 3 shows, total ion chromatograms for N-nitrosodimethylamine and pyridine on an expanded axis for the 7890 GC and Intuvo 9000 GC. As expected, the additional volume causes a slight increase in retention time compared to 7890 GC. However, the effect on peak shape was negligible.

Method 8270D stipulates that for reporting close eluting structural isomers as separate analytes, the valley between the two peaks cannot be greater than 50 % of the average maximum height of the isomers. Benzo[b]fluoranthene and benzo[k]fluoranthene are generally selected as a measure of the system's ability to resolve isomers. Figure 4 shows the resolution achieved on the 7890 GC and Intuvo GC. In both cases, the valley between the isomers was well below 50 %.

As with many GC methods, quantitation with method 8270D is based upon the relative response with respect to the internal standards. Figure 5 shows a comparison of response factors for the 7890 GC and Intuvo 9000 GC for an injection of the 20 µg/mL standard. Again, agreement was very good. The average difference in response factors across all the target compounds was 4.6 %.

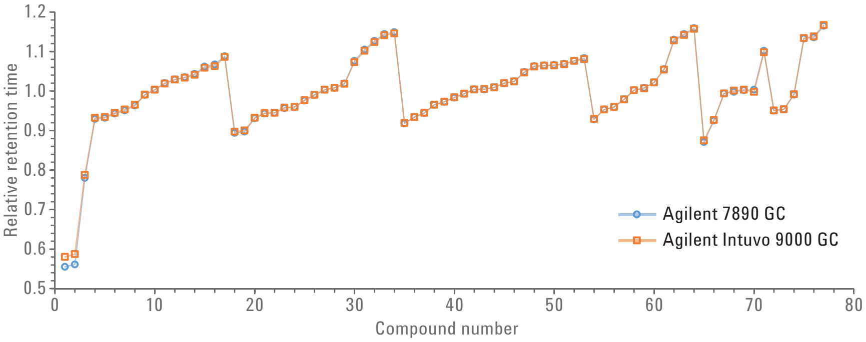

Figure 2. Comparison of relative retention times for SVOCs on an Agilent Intuvo 9000 GC and an Agilent 7890 GC.

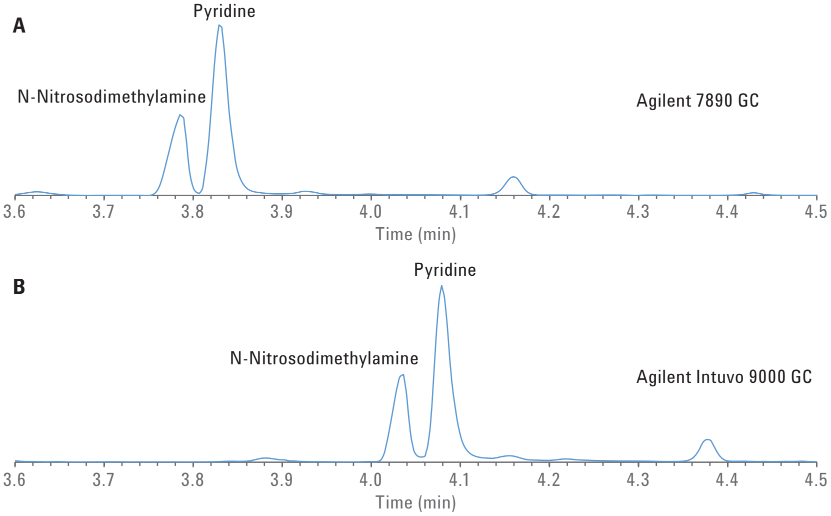

Figure 3. Comparison of first eluting SVOCs on an Agilent 7890 GC (A) and an Agilent Intuvo 9000 GC (B).

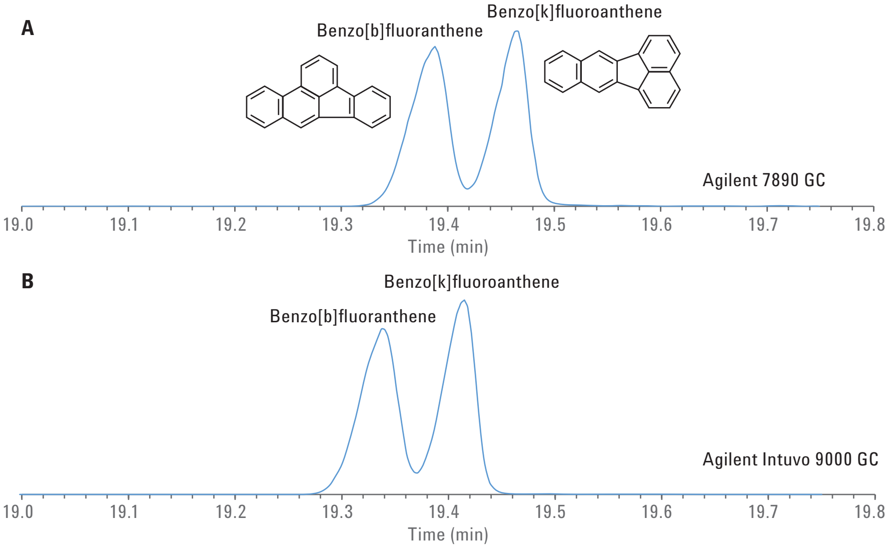

Figure 4. Resolution of isomers on an Agilent 7890 GC (A) and an Agilent Intuvo 9000 GC (B).

In Figure 5, note that the relative responses of the last eluting polyaromatic hydrocarbons (PAHs) indeno[1,2,3-cd] pyrene, dibenzo[a,h]anthracene, and benzo[g,h,i]perylene was slightly greater on the Intuvo 9000 GC compared to the 7890 GC. This demonstrates that the thermal profile across the Intuvo 9000 GC flow path is consistent, allowing the higher boiling point PAHs to pass through the flow path, and maintain recovery and peak shape (Figure 1).

## Method 8270D suitability

For the analysis of SVOCs by GC/MS, it is of paramount importance to verify the system is suitable for quantitative analysis before the collection of data, particularly when data are used for regulatory reporting. Method 8270D specifies the use of a tuning or control standard for verifying performance of the GC/MS. The standard consists of a mixture of DFTTP, 4,4′-DDT, pentachlorophenol, and benzidine to test MS tuning and inertness.

The DFTPP is used to verify suitable ionization and detection of the mass spectrometer. The 4,4′-DDT is used to gauge system inertness by determination of the breakdown products 4,4′-DDD and 4,4′-DDE. Benzidine is used as a probe for base activity, and pentachlorophenol is used as a probe for acid activity. If the minimum performance criteria as established by the method cannot be achieved, the system is deemed unsuitable for analysis.

Figure 6 shows a chromatogram of the tuning standard at a concentration of 25 µg/mL. Method 8270D suggests a concentration of 50 µg/mL with a caveat that lower concentrations may be used to accommodate instruments with greater sensitivity. In this case, 25 µg/mL was selected as not to overload the column, and bias peak symmetry measurements.

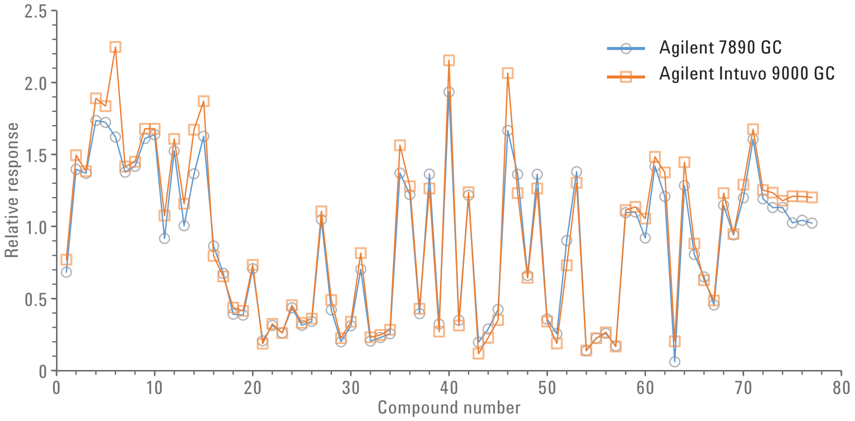

Figure 5. Comparison of relative retention times for SVOCs on an Agilent Intuvo 9000 GC and an Agilent 7890 GC.

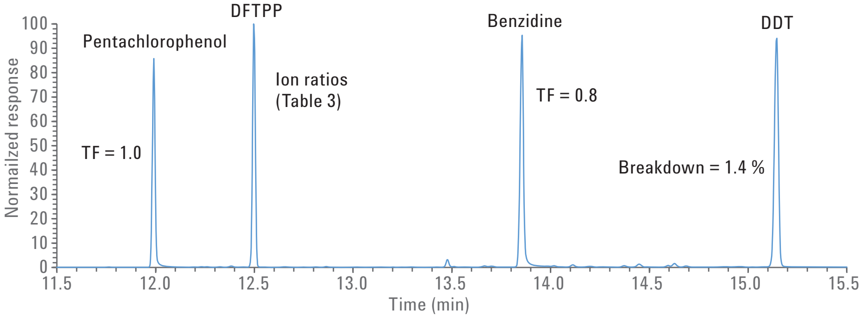

Figure 6. Chromatogram of a method 8270D tuning mix on the Agilent Intuvo 9000 GC.

Tailing factor (TF) was used as the determinant of acid/base activity of pentachlorophenol and benzidine. Based upon method 8270D requirements, the TF measured at 10 % peak height for the extracted quantitation ion should be no greater than 2. For pentachlorophenol and benzidine, the measured tailing factors were 1.0 and 0.8, respectively.

A percent breakdown of 4,4′-DDT was used to determine system inertness. According to method 8270D, the combined area sum of the extracted ions for 4,4′-DDD and 4,4′-DDE should not exceed 20 %. The percent breakdown was measured at 1.4 % on the Intuvo 9000 GC.

Table 3 lists the measured DFTPP ion ratios along with the specified ratios and ranges for method 8270D. All measured ratios are well within the required limits.

The Intuvo 9000 GC easily passed the system suitability metrics as specified in method 8270D.

## Calibration requirements

Calibration is, perhaps, the most challenging requirement to achieve and maintain with method 8270D. The target list comprises a range of acidic, basic, and neutral molecules. The type of calibration and calibration range for a selected analyte is largely based upon the sensitivity of the instrument and the nature of the compound. Some compounds are more sensitive to surface activity, thermal conditions, or detection efficiency. Because of this, several methods of calibration are acceptable for quantitation.

The simplest and most widely applied calibration is based upon average response factor. According to the method, a minimum of five standard levels must be used, and the relative standard deviation (RSD) in response factors should be within ±20 %. Figure 7 shows the percent RSD achieved for 57 of the 77 compounds over a range of 0.1 to 100 µg/mL using 14 calibration levels (with the exception of number 23, benzoic acid, with a calibration from 4 to 100 µg/mL over eight levels). A total of 14 calibration levels were used To precisely determine the linear range of each analyte. The average RSD for the 57 compounds was 4.98 %.

Table 3. DFTPP tuning check.

| Target mass | Relative to mass | Lower limit % | Upper limit % | Rel. abn % | Pass/Fail |
|---|---|---|---|---|---|
| 51 | 442 | 10 | 80 | 31.1 | Pass |
| 68 | 69 | 0 | 2 | 0 | Pass |
| 70 | 69 | 0 | 2 | 0.4 | Pass |
| 127 | 442 | 10 | 80 | 39.4 | Pass |
| 197 | 442 | 0 | 2 | 0 | Pass |
| 198 | 442 | 50 | 100 | 84 | Pass |
| 199 | 198 | 5 | 9 | 6.1 | Pass |
| 275 | 442 | 10 | 60 | 22.4 | Pass |
| 365 | 198 | 1 | 100 | 4.2 | Pass |
| 441 | 442 | 0 | 24 | 15.4 | Pass |
| 442 | 442 | 100 | 100 | 100 | Pass |
| 443 | 442 | 15 | 24 | 18.7 | Pass |

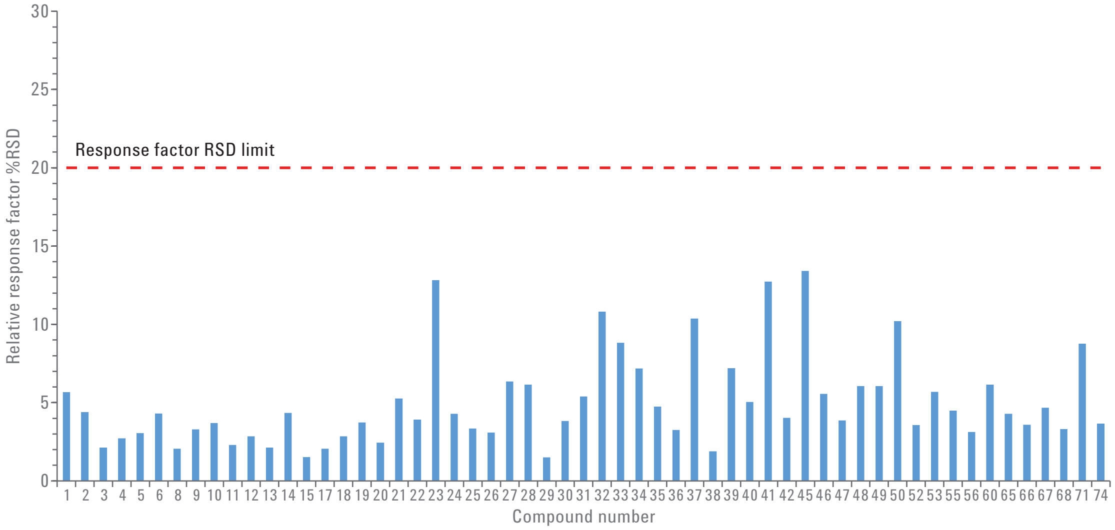

Figure 7. Percent RSD in average response factor for calibration up to 100 µg/mL.

Certain compounds with higher sensitivity such as PAHs tend to overload the stationary phase and saturate the detector at high concentration. In this case, the upper calibration limit was lowered to 50 µg/mL to achieve the calibration criteria using average response factors. Figure 8 shows the percent RSD for 14 of the 77 compounds over a range of 0.1 to 50 µg/mL using 12 calibration levels (with the exception of number 69, benz[a]anthracene, with a calibration from 0.8 to 50 µg/mL over 10 levels). The average RSD for the 14 compounds was 4.10 %.

Some compounds that are active or labile tend to have response factors that vary as a function of concentration. For these analytes, method 8270D allows curve fitting for calibration. The method stipulates that the correlation coefficient (R) must be greater than 0.99, and the calculated concentration of the lowest standard must be within ±30 % of the actual concentration. Table 4 lists the calibration results for the remaining six of the 77 compounds using a weighted linear least squares regression with a 1/x weighting factor. In all the cases, the specified calibration criteria were achieved. Note that the calibration range was selected to achieve the widest dynamic range while satisfying the calibration criteria using a linear model. The percent deviation of the lowest level standard would be closer to zero if the dynamic range were narrowed, or a higher order calibration model were used.

## Matrix study

To gauge the durability of the Intuvo 9000 GC, an iterative cycle of matrix injections and performance checks was carried out. Typically, environmental testing laboratories perform preventative maintenance (for example, liner replacement, column clipping) at regular intervals. This is done to maintain system suitability and calibration integrity over an extended period of time by preempting column and source contamination.

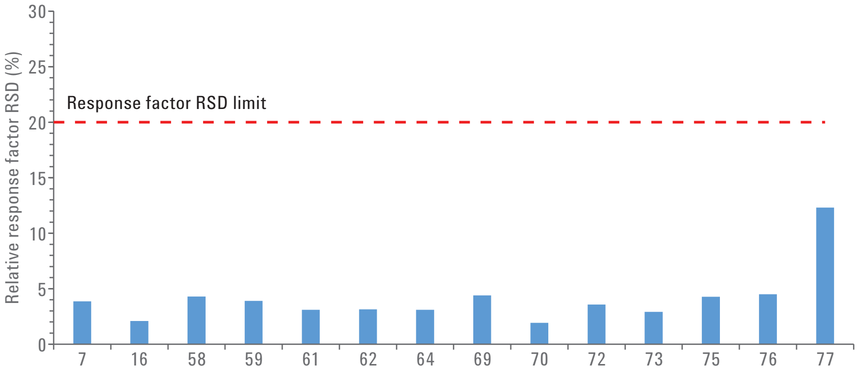

Figure 8. Percent RSD in average response factor for calibration up to 50 µg/mL.

Table 4. Calibration results using weighted least squares regression.

| Compound no. | Compound | $R^2$ | Calibration range (µg/mL) | Percent difference of lowest level standard (±30 % Required) |
|---|---|---|---|---|
| 43 | 2,4-Dinitrophenol | 0.9984 | 1.6–100 | 23.9 |
| 44 | 4-Nitrophenol | 0.9994 | 0.8–100 | 22.9 |
| 51 | 4,6-Dinitro-2-methylphenol | 0.9991 | 0.8–100 | -1.3 |
| 54 | 2,4,6-Tribromophenol | 0.9997 | 0.8–100 | 12.4 |
| 57 | Pentachlorophenol | 0.9992 | 0.8–100 | 23.4 |
| 63 | Benzidine | 0.9966 | 4–100 | 16.5 |

However, in this study, a test strategy was used where matrix samples were injected until suitability or calibration failure, then returned to performance through corrective maintenance. Figure 9 shows a flowchart illustrating the testing scheme.

The test study was gated by performance checks in between

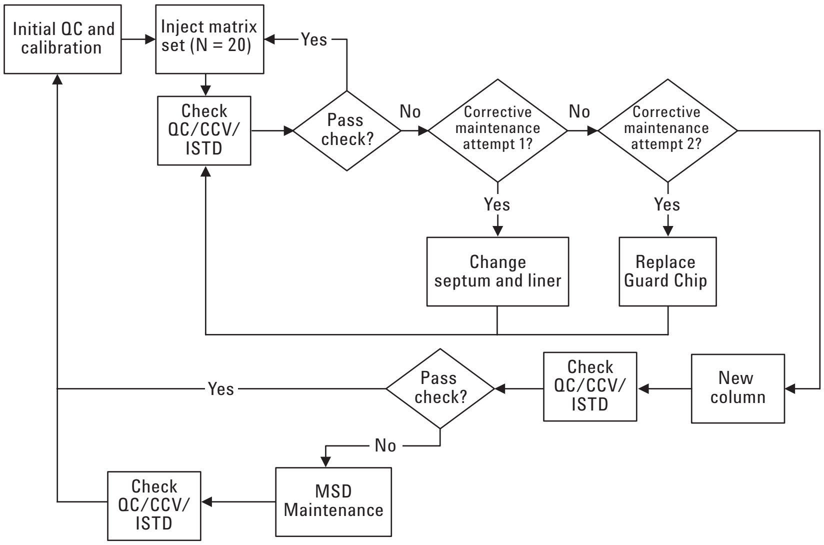

Figure 9. Matrix study testing scheme.

20 matrix injections. The check consisted of three measurements related to the specifications listed in method 8270D including:

- **QC** – Correct DFTPP tuning ratios, tailing factor for benzidine and pentchlorphenol less than 2, and percent breakdown for 4,4′-DDT of less than 20 %
- **CCV** – Midpoint calibration drift is within ±20 % for greater than 10 % of the target compounds
- **ISTD** – Verify the area of internal standard peak area drift is within ±50 %

To begin the study, the system was calibrated using the compounds listed in Table 1, and the method 8270D parameters listed in Table 2. The matrix sample was supplied by ESC Lab Sciences (Mt. Juliet, TN), and was composed of multiple soil samples extracted with dichloromethane and combined. The composite extract was representative of the heaviest matrix sample typically encountered in their laboratory. Figure 10 shows that the extract was opaque, and contained significant matrix residue.

Figure 10. Dichloromethane soil extract

## Study results

### QC results

A total of 680 matrix injections were performed over the course of the study. After each interval of 20 sample injections, the matrix load was sufficient to almost cause a complete breakdown of 4,4′-DDT. Following the testing protocol, the liner and septum were replaced, and the system was retested. After the liner replacement, the percent breakdown dropped below 20 % (Figure 11).

Figure 12 shows a liner after 20 injections of the soil extract. Clearly visible is a layer of soil residue deposited on the glass wool. This residue was likely the cause of the 4,4′-DDT breakdown since replacement of the liner restored breakdown to below 20 %. The glass wool packing was sufficient to protect the system from particulate contamination.

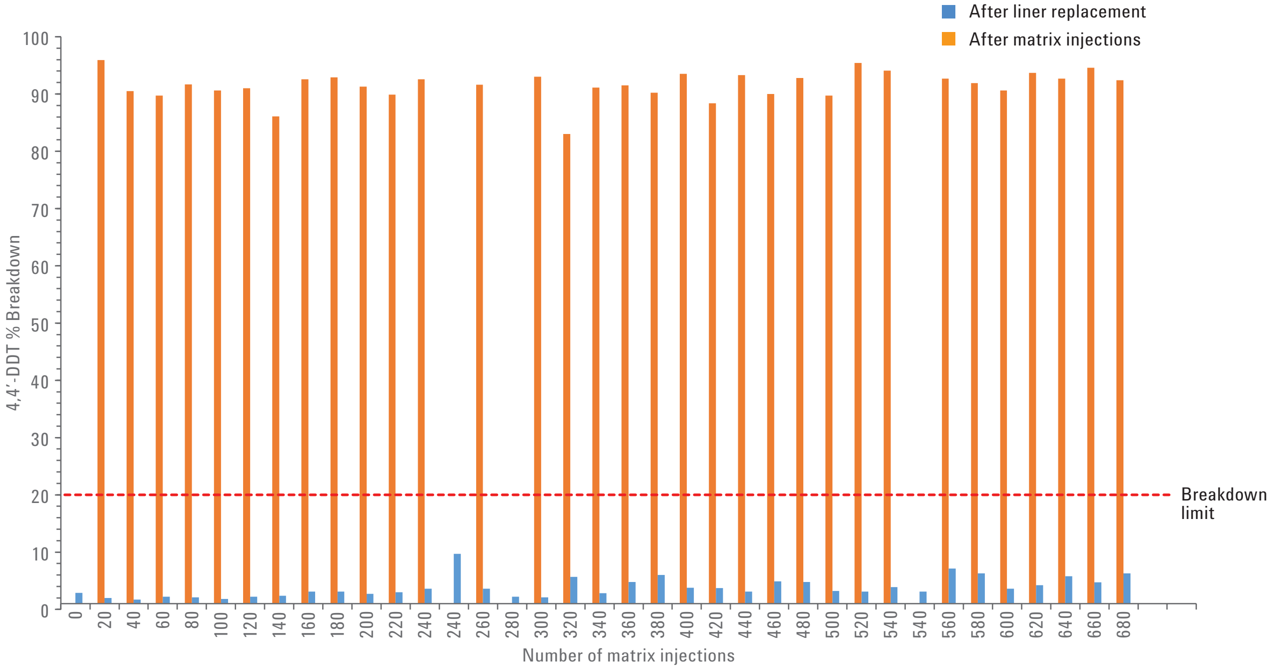

Figure 11. Breakdown and recovery of DDT after liner change (blue) and after matrix injection (orange).

In addition to 4,4′-DDT, the QC sample contains pentachlorophenol and benzidine. Figure 13 shows the tailing factor for benzidine and pentachlorophenol measured after liner replacement. From 180 to 240 matrix injections, the benzidine tailing factor increased from 1.0 to 1.8 close to the limit of 2. Liner replacement was not sufficient to reduce the tailing factor. Following the study protocol, the Intuvo Guard Chip was replaced, and the system was retested, then the tailing factor of benzidine dropped to 1.2. After 520 injections, the benzidine tailing factor increased to 1.7. In this case, liner replacement was sufficient to reduce the tailing factor to 1.

## CCV Results

According to method 8270D, every 12 hours the calibration needs to be verified by injecting a standard at the midpoint of the calibration range. The calculated concentration must be within ± 20 % of the actual concentration for the curve to be validated. If more than 20 % of the compounds fail the ± 20 % calibration check, the system is deemed unsuitable, and corrective action must be taken. In this study, a corrective action limit was set to be more stringent at a failure rate of 10 % for the 77 targets and surrogates (that is, 7 compounds failing CCV). Figure 14 shows the CCV result. After every 20 matrix injections, the number of compounds failing calibration exceeds the 10 % limit. After a liner change, the number of compounds failing the calibration dropped to below 10 %.

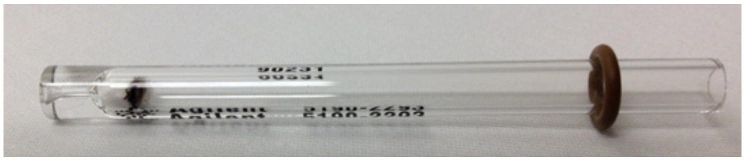

Figure 12. Glass wool packed liner after 20 matrix injections.

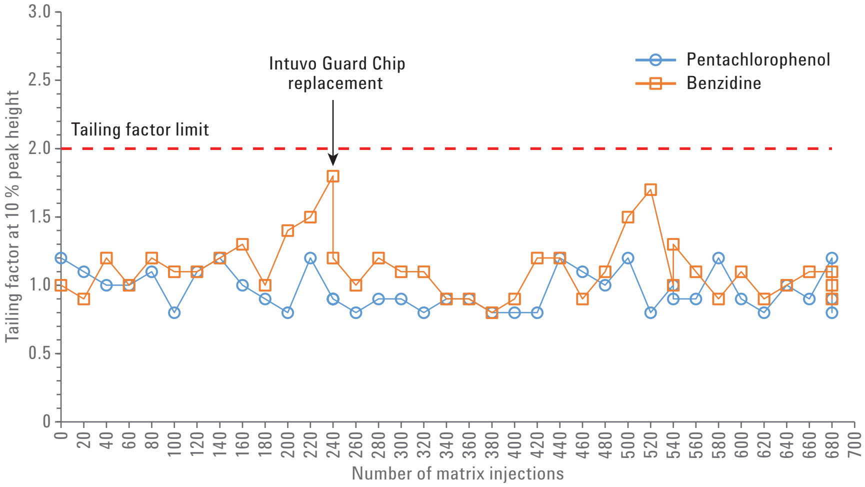

Figure 13. Tailing factor measurements after liner replacement for pentachlorophenol and benzidine.

Figure 14 also shows replacement of the Intuvo Guard Chip after 240 matrix injections triggered by the benzidine tailing factor (Figure 13). As indicated in Figure 14, one compound, hexachlorocyclopentadiene, failed the CCV check even after Intuvo Guard Chip replacement. Although the percent error dropped from –31.3 % to –26.7 %, the Intuvo Guard Chip replacement was not sufficient to reduce the percent error below 20 %. The sensitivity of this compound to matrix can be used as an indicator for determining Intuvo Guard Chip replacement frequency. Figure 15 shows a plot of the CCV checks for hexachlorocyclopentadiene as a function of the number of matrix injections.

Based on this data, a preventative maintenance cycle can be estimated of one Intuvo Guard Chip replacement after every 60 matrix injections. Considering that the extract contained significant matrix residue, it may be surmised that this Intuvo Guard Chip replacement frequency would be sufficient for most soil matrices.

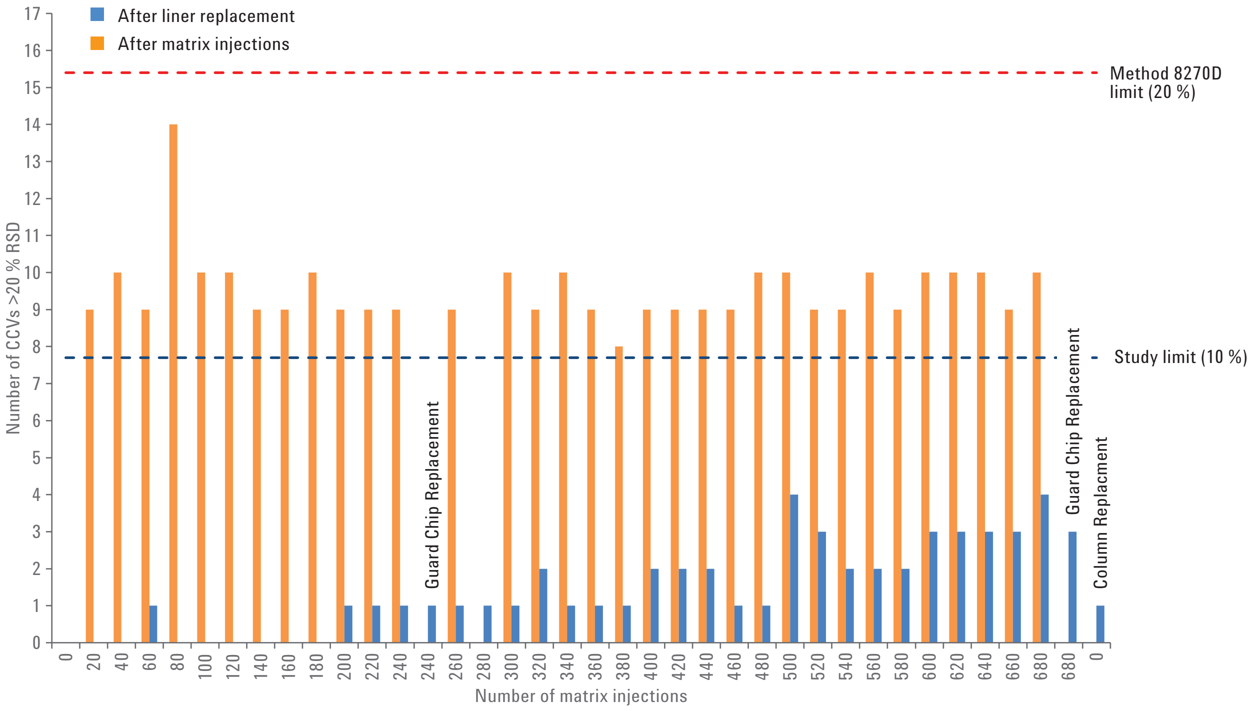

Figure 14. Number of CCV failures after liner change (blue), and after matrix injections (orange).

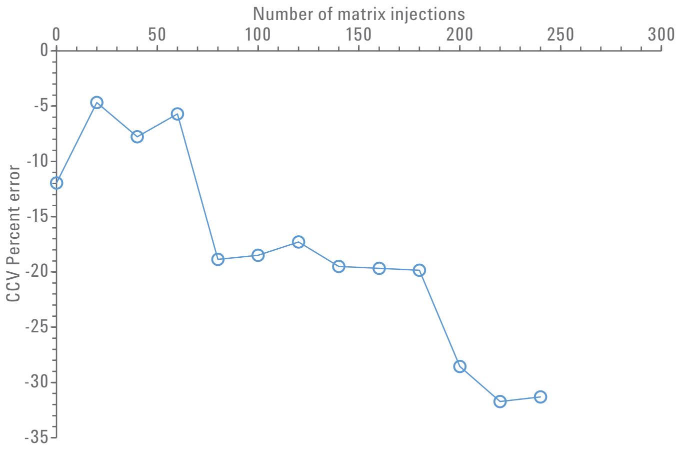

Figure 15. CCV check percent error for hexachlorocyclopentadiene.

To gauge the level of system contamination as a result of a heavy matrix load, matrix injections were continued. Although the 10 % threshold was not reached within the 680 injections, the study was concluded. Figure 14 shows that liner replacement after the 680th injection resulted in 10 CCV failures dropping to four. Intuvo Guard Chip replacement resulted in a further reduction from four to three CCV failures. Column replacement resulted in complete system restoration. The only failing compound after column replacement was benzidine which gave a response greater than the initial calibration (Figure 16). This indicates that the cause of these CCV failures was isolated to the column, and not the flow path or ion source.

## ISTD Results

Method 8270D requires that the variation of the area of internal standards should not exceed a factor of 2. If it does, the system is not suitable for analysis, and corrective action must be taken. Typically, loss in response in the internal standard is a result of ion source contamination. Figure 17 shows the normalized area for the internal standards over 680 injections after liner replacement. Throughout the study, the internal area was within the specified range.

## Comparison to an Agilent 7890A GC

As a point of reference, three similar matrix studies were carried out on the 7890A GC coupled to a 5977 MSD using the protocol shown in Figure 9. Table 5 lists the results. Interestingly, the behavior of the 7890A GC combined with an 5977 MSD appeared significantly different from the Intuvo 9000 GC. For column numbers 1 and 3, the mechanism of the failure was a combination of ISTD responses falling below 50 % (which were restored with ion source cleaning), and greater than 10 % failure of the CCV checks (which were not restored by inlet maintenance or column trimming). For column 2, the mechanism of failure was a result of greater than 10 % failure of the CCV checks.

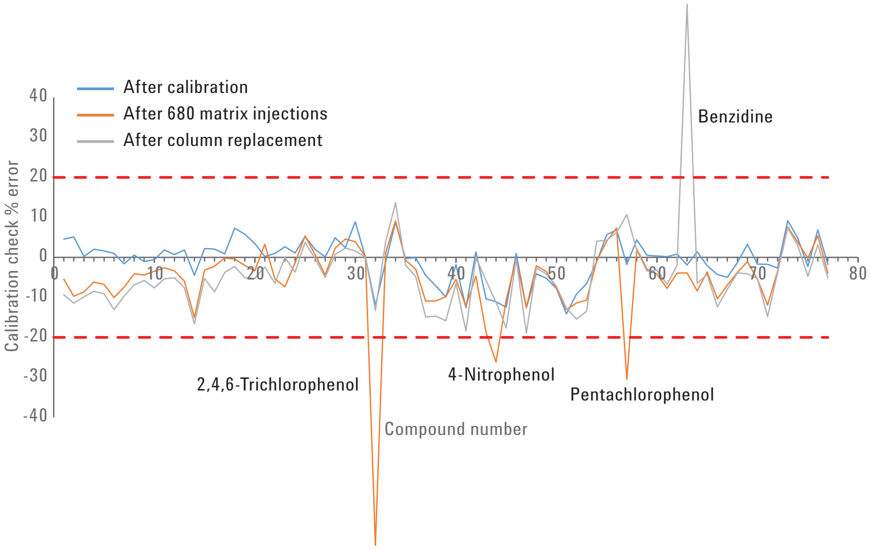

Figure 16. Calibration checks after initial calibration, after 680 matrix injections, and after column replacement.

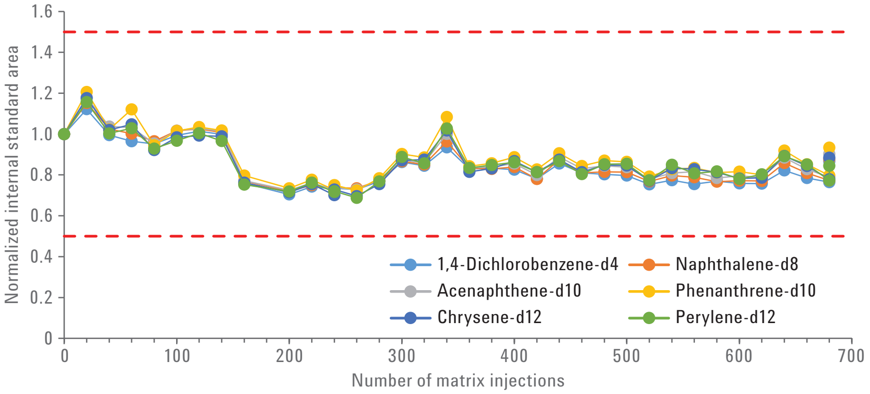

Figure 17. Normalized internal standard peak area across 680 matrix injections.

Table 5. Results of the Agilent 7890A GC combined with an Agilent 5977 MSD study.

| Column number | Number of liners and septa replaced | Number of inlet seals replaced | Number of column trims (30.5 cm each) | Total number of matrix injections |
|---|---|---|---|---|
| 1 | 12 | 2 | 6 | 240 |
| 2 | 4 | 2 | 5 | 80 |
| 3 | 6 | 3 | 4 | 120 |

Another difference in performing maintenance on the 7890 GC compared to Intuvo 9000 GC is that the Intuvo Guard Chip can be replaced more quickly and more assuredly on the Intuvo 9000 GC compared to column trimming on the 7890 GC. In addition, column trimming on the 7890 GC required readjustment of the retention time windows after a sufficient portion of the column was removed. This did not occur with the replacement of the Intuvo Guard Chip. Figure 18 shows the change in retention time after replacement of an Intuvo Guard Chip on the Intuvo 9000 GC compared to trimming 30.5 cm of the column on a 7890 GC. The shift in retention times are clearly visible after trimming on the 7890 GC compared to the Intuvo Guard Chip replacement on the Intuvo 9000 GC where the overlays are virtually identical.

## Conclusion

This study demonstrates the suitability of the Agilent Intuvo 9000 GC for the analysis of SVOCs. The Intuvo 9000 GC can easily meet the performance requirements as specified by USEPA method 8270D. Compared to the Agilent 7890 GC, the Intuvo 9000 GC provided equivalent results in terms of relative retention time and relative response. In addition, repetitive injections of a soil extract illustrated the resilience of the Intuvo 9000 GC to a substantial matrix challenge, and it was easier to maintain compared to the 7890 GC. Intuvo Guard Chip replacement was more expedient than column trimming in terms of maintenance time, and did not require retention time adjustment.

## Acknowledgement

The author would like to thank John Romesburg for his significant contribution to this study, ESC Lab Sciences for donation of matrix extracts, and Michael Szelewski for his expertise in environmental analysis.

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Published in the USA, September 1, 2016
5991-7256EN