Reduction in EI Source Cleaning Frequency: The Benefits of a Novel GC with an Inert Microfluidic Flowpath

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Introduction

Matrix Effects in GC/MS: Flow Path Contamination

Overtime, changes in signal response may occur with the accumulation of contaminants on the ion source chamber surfaces when using GC/MS coupled with electron ionization (EI). Contamination may occur through repeated analysis of samples containing analytes or matrices of low volatility or by the accumulation of stationary phase bleed, resulting in changes in analyte response or peak skewing. When significant levels of contamination are observed, corrective action is necessary to restore initial performance. In some cases, this may require significant time associated with venting, cleaning the source, and restoring vacuum. In this paper, results indicate that the use of a novel GC with an inert microfluidic flow path reduces ion source cleaning frequency compared to traditional GC for the analysis of semivolatile organic compounds (SVOCs).1

Agilent Intuvo 9000 GC

The Agilent Intuvo 9000 GC is a significant advancement in the development of gas chromatographs.2 The Intuvo includes a number of design innovations making it ideally suited for SVOC analysis particularly in high-throughput laboratory settings.

- **Guard chip** – easily replaceable precolumn to prevent particulate and nonvolatile contamination of the flow path, column and detector. The guard chip can be replaced as easily as an inlet liner.

- **Intuvo Flow Technology (IFT)** – modular microfluidic devices used to assemble the flow path from inlet to detector. The flow path is customizable to suit analysis requirements with inlet or detector splitting and mid-column or post-column backflush.

- **No-trim columns** – Intuvo columns are the same columns used in the Agilent 7890 wound into a planar format compatible with the Intuvo direct heating and click and run connections.

- **Click and run connections** – ferrules have been eliminated and replaced with direct face seal connections for easy, fast and reliable maintenance.

- **Direct heating** – unlike air bath ovens, the Intuvo uses direct conductive heating. This enables both fast column heating and faster cool down.

Experimental

Experimental Approach

Matrix studies were conducted concurrently on two separate instrument platforms. One system was an Agilent Intuvo 9000 GC coupled to 5977 GC/MSD and the other system was an Agilent 7890 coupled to a 5977 GC/MSD. Both MSDs were configured with an inert ion source with 6 mm drawout plate. The same instrument methods were used on both systems with the exception of the guard chip which is unique to the Intuvo 9000 GC (Table 1).

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<th>Table 1. Instrument Conditions</th>
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<td><strong>GC Conditions</strong></td>
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<tr>
<td>Liner</td>
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<td>Guard chip (Intuvo only)</td>
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Results and Discussion

Matrix Study

Figure 1 shows the scheme used to track the contamination of the system over the course of matrix study. After calibration and initial system performance check, 20 matrix injections were made. Following the matrix injection, the system performance, calibration and internal standard (ISTD) responses were reevaluated. The maintenance action level for each performance metric was based upon the limits set by USEPA Method 8270D.1 A sequential approach to maintenance was used in order to determine the source of contamination in the instrument flowpath.

![Flowchart showing maintenance action levels for matrix study](image)

Figure 1. Scheme for tracking contamination

Soil Extracts

In order to model the worst-case soil extract typically encountered in an environmental lab, a composite soil extract in methylene chloride was donated from ESC Lab Sciences (Mt. Juliet, TN). The soil extract contained a significant level of particulate soil residue requiring liner replacement after just 20 matrix injections (Figure 2).

![Image of soil extract and liner contamination](image)

Figure 2. Soil extract and liner contamination

7890 GC/5977 MSD Internal Standard Response

In accordance with USEPA 8270D, the threshold criteria for internal standard response was set to a maximum loss of 50% peak area compared to the midpoint calibration standard in the initial calibration. Two replicate studies were carried out starting with new columns and clean sources.

The result of the first matrix study with the 7890/5977 is shown in Figure 3. Area measurements shown were taken after maintenance was performed to comply with the study scheme (Figure 1). Maintenance included liner replacement after each series of 20 matrix injections in order to restore system inertness. Inlet seal replacement and column trimming was required at certain intervals in order to restore calibration. As indicated in Figure 3, the internal area response begins to approach the 50% threshold after 240 matrix injections. Inlet maintenance and column trimming was not able to restore performance. At this point, the column was replaced and the system was retested. However, the internal standard response was not restored. Only after source cleaning did the ISTD responses return to levels close to the initial responses.

![Graph showing normalized ISTD response over matrix injections](image)

Figure 3. First 7890/5977 matrix study. ISTD response over the course of matrix injections.

The result of the second matrix study with the 7890/5977 is shown in Figure 4. After 120 matrix injections, the area response of 1,4-dichlorobenzene-d$_4$ dropped below the 50% threshold. Inlet maintenance, column trimming and column replacement did not return the response to the initial levels. Again, source cleaning was required to return ISTD responses close to the initial levels.
Results and Discussion

7890 GC/5977 MSD Internal Standard Response

![Graph showing ISTD response over the course of matrix injections.]

Figure 4. Second 7890/5977 matrix study. ISTD response over the course of matrix injections.

Intuvo GC/5977 MSD Internal Standard Response

The result of the matrix study on the Intuvo/5977 is shown in Figure 5. After each series of 20 matrix injections, the liner was replaced. Interestingly, the 50% response threshold was not reached over the course of 680 matrix injections. Also, it appears that the variation in ISTD response is uniform for all internal standards and not divergent as observed with the 7890/5977. This indicates that variability in response is likely due to other factors which are not related to detection (e.g. injection variability, solvent evaporation).

![Graph showing ISTD response over the course of matrix injections.]

Figure 5. Intuvo/5977 matrix study. ISTD response over the course of matrix injections.

Current Work

The reason for the stability of internal standard response on the Intuvo/5977 system is currently being investigated in greater detail. The first hypothesis is that the Intuvo flow path in between the distal end of the column and the source is providing some protection from column bleed. This is being tested by replicating the Intuvo flow path in the 7890 oven and measuring the internal standard response after long intervals at which the column is held at maximum temperature. Initial data indicates there is no additional protection comparing measurements with and without the Intuvo flow path in the 7890 oven.

Future Work

The second hypothesis is that the Intuvo guard chip and flow path are providing some protection of the ion source from matrix. Future studies will be carried out comparing the longevity of source response with and without the Intuvo flow path replicated in a 7890 oven for a series of matrix injections.

Conclusions

Matrix Study

In comparing results between the matrix study on the 7890/5977 and Intuvo/5977 it appears that there is a substantial increase in the number of matrix injections that can be performed on the Intuvo before source cleaning is required. Further studies are being conducted in order to verify and elucidate the exact mechanism observed in these initial studies. If verified, this would prove to be of substantial benefit in the analysis of SVOCs particularly for contract environmental laboratories.

References


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