Validation of USEPA Method 8260C
Using Teledyne Tekmar Atomx, and
Perkin-Elmer Clarus 600 GC/MS

Application Note

By Tyler Trent

Abstract

In order to determine the concentration of volatile organic compounds (VOCs) in water and soil matrices the USEPA developed Method 8260C\(^1\) in conjunction with preparative Methods 5030\(^2\) and 5035\(^3\). Water and soil samples were analyzed in this study. A working linear calibration curve and Method Detection Limits (MDLs) will be demonstrated for the target compound list. The water study used a 5mL sample volume, while the soil study utilized an in-vial purge. Following the conditions of USEPA Method 8260C, an Atomx Automated VOC Sample Prep system in conjunction with a GC/MS was used to validate the method.

Introduction

Teledyne Tekmar has developed the Atomx, an Automated VOC Sample Prep system that integrates a purge and trap concentrator with a multi-matrix autosampler. This “all-in-one” set up allows for increased throughput and efficiency through the features that it provides, such as the three standard addition vessels and 80-postion autosampler tray. Also, the Atomx allows for sampling of multiple matrices, water, soil and methanol extraction in a single run.

Using the PerkinElmer Clarus 600/600T GCMS, a linear calibration curve and MDLs were performed for both water and soil matrices. The water samples employed a calibration range from 0.5-200ppb, while the soil calibration curve ranged from 1.0-200ppb. Both water and soil methods require either a 5mL or 5g sample size. Analysis strictly followed the criteria outlined in the USEPA Method 8260C.

Experimental-Instrument Conditions

The Atomx, equipped with a #9 adsorbent trap, and a PerkinElmer Clarus 600 GC with a Clarus 600T MS were utilized for this study. Tables 1-4 show the CG/MS and purge and trap conditions for both water and soil applications.

<table>
<thead>
<tr>
<th>GC Parameters</th>
<th>MSD Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC: Perkin-Elmer Clarus 600 Gas Chromatograph</td>
<td>MSD: Clarus 600T Quadrupole Mass Spectrometer</td>
</tr>
<tr>
<td>Column: Restek RTX-VMS 20m x 0.18mmID x 1um</td>
<td>Source: 200 °C</td>
</tr>
<tr>
<td>Oven Program: 40 °C for 4 min; 16 °C/min to 100 °C for 0 min; 30 °C /min to 200 °C for 4 min, 15.083 min runtime</td>
<td>Transfer Line Temp: 200 °C</td>
</tr>
<tr>
<td>Inlet: 220 °C</td>
<td>Solvent Delay: 0.5 min</td>
</tr>
<tr>
<td>Column Flow: 0.9mL/min</td>
<td>Scan Range: 35-270 m/z</td>
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<tr>
<td>Gas: Helium</td>
<td>Scan Time: 0.2 sec</td>
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<tr>
<td>Split: 80:1</td>
<td>Inter-scan Delay: 0.1 sec</td>
</tr>
<tr>
<td>Pressure: 18.4psi</td>
<td>Ionization Mode: EI+</td>
</tr>
<tr>
<td>Inlet: Split/Split less</td>
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Tables 1 & 2: GC and MSD Parameters
### Atomx Water Parameters

<table>
<thead>
<tr>
<th>Variable</th>
<th>Value</th>
<th>Variable</th>
<th>Value</th>
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<tr>
<td>Valve Oven Temp</td>
<td>140 °C</td>
<td>Dry Purge Flow</td>
<td>100mL/min</td>
</tr>
<tr>
<td>Transfer Line Temp</td>
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<td>Dry Purge Temp</td>
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</tr>
<tr>
<td>Sample Mount Temp</td>
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<td>Methanol Needle Rinse</td>
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</tr>
<tr>
<td>Water Heater Temp</td>
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</tr>
<tr>
<td>Sample Vial Temp</td>
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<td>Water Needle Rinse Volume</td>
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</tr>
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<td>Sample Equilibrate Time</td>
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<td>Sweep Needle Time</td>
<td>0.50 min</td>
</tr>
<tr>
<td>Soil Valve Temp</td>
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<td>Desorb Preheat Time</td>
<td>245 °C</td>
</tr>
<tr>
<td>Standby Flow</td>
<td>10mL/min</td>
<td>GC Start Signal</td>
<td>Start of Desorbs</td>
</tr>
<tr>
<td>Purge Ready Temp</td>
<td>40 °C</td>
<td>Desorb Time</td>
<td>2.00 min</td>
</tr>
<tr>
<td>Condensate Ready Temp</td>
<td>45 °C</td>
<td>Drain Flow</td>
<td>300mL/min</td>
</tr>
<tr>
<td>Presweep Time</td>
<td>0.25 min</td>
<td>Desorb Temp</td>
<td>250 °C</td>
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<td>Prime Sample Fill Volume</td>
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<td>Sweep Sample Flow</td>
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<td>Bake Rinse Sweep Flow</td>
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<td>Bake Flow</td>
<td>200mL/min</td>
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<td>Bake Temp</td>
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*Table 3: Atomx Water Parameters (Parameters highlighted in yellow were not used.)*
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<th>Value</th>
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<tr>
<td>Transfer Line Temp</td>
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<td>Sample Mount Temp</td>
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<td>Purge Temp</td>
<td>20 °C</td>
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<td>Water Heater Temp</td>
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<td>Condensate Purge Temp</td>
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<td>Sample Vial Temp</td>
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<td>Dry Purge Time</td>
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<tr>
<td></td>
<td></td>
<td>Dry Purge Flow</td>
<td>100mL/min</td>
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<tr>
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<td>Standby Flow</td>
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<td>Sweep Needle Time</td>
<td>0.25 min</td>
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<td>Purge Ready Temp</td>
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<td>Start of Desorbs</td>
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</table>

Table 4: Atomx Soil Parameters (Parameters highlighted in yellow were not used.)

 Calibration and Minimum Detection Limits

A 50ppm working calibration standard was prepared in methanol. Calibration standards were made in volumetric flasks using de-ionized water. The water calibration ranged from 0.5-200ppb, while the soil ranged from 1-200ppb. A 25ppm internal standard (IS) was prepared in methanol and transferred to one of the three standard addition vessels on the Atomx. Using the standard addition feature, the Atomx transferred the IS in 5μL aliquots to the sample providing a constant 25ppb final concentration in 5mL.

TurboMass software was used to process the calibration and MDL data. The relative response factors (RRF) of all target analytes were evaluated for average Response Factor (RF) and calibration percent relative standard deviation (%RSD). Both water and soil calibration curves met the USEPA 8260C1 performance criteria with results listed in Table 5. Total Ion Chromatograms (TIC) for 25ppb water and soil standard can be found in Figures 1-2.

Method detection limits (MDLs) were established for all compounds by analyzing seven replicates at 1.0ppb for water and 5.0ppb for soil. The MDLs for each matrix can be found in Table 5.
<table>
<thead>
<tr>
<th>Compound</th>
<th>Water</th>
<th>Soil</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Spike Level (ppb)</td>
<td>MDL</td>
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<tr>
<td>Pentfluorobenzene (IS)</td>
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<tr>
<td>Dichlorodifluoromethane</td>
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<td>0.132</td>
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<td>Soil</td>
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Table 5 Continued
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<th>MDL</th>
<th>Water Compo. Level (ppb)</th>
<th>MDL</th>
<th>Soil Compo. Level (ppb)</th>
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Table 5: Experimental Results For USEPA Method 8260C Water and Soil

* Compound was linear regressed

Figure 1: TIC of a 25ppb Water Standard for Method 8260C
Conclusions

This study demonstrates the capability of the Atomx Automated VOC Sample Prep system in conjunction with a PerkinElmer Clarus 600 GC/MS in regards to USEPA Method 8260C. Calibration and MDL data met all performance criteria of the method. By completely automating the sample preparation of multiple matrices, efficiency and throughput can be greatly increased, saving time and money.

References

1. USEPA Method 8260C Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Revision 3, August 2006
2. USEPA Method 5030 Purge-And-Trap For Aqueous Samples Revision 3, May 2003
3. USEPA Method 5035 Closed-System Purge-And Trap and Extractions For Volatile Organics In Soil and Waste Samples Revision 1, July 2002