Analysis of Semivolatile Organic Compounds Using Hydrogen Carrier Gas and the Agilent HydroInert Source by Gas Chromatography/Mass Spectrometry

Abstract

Gas chromatography/mass spectrometry (GC/MS) is integral to the analysis of semivolatile organic compounds (SVOCs) in environmental matrices. Recent pressure on the helium (He) supply has required organizations to actively investigate hydrogen (H₂) carrier gas, but most GC/MS analyses have reduced sensitivity and hydrogenation or dechlorination in the sources. The Agilent HydroInert source retains the ability to analyze a wide calibration range (0.1 to 100 µg/mL) and meet the U.S. Environmental Protection Agency (EPA) method 8270 calibration criteria when using H₂ carrier gas.
Introduction

GC/MS is regarded as the select analytical technique for the analysis of SVOCs. Governmental regulatory authorities have established methods and performance criteria for the measurement of SVOCs identified as pollutants in environmental and industrial matrices. For example, the U.S. EPA method 8270 (versions 8270D and 8270E) contains a list of over 200 compounds suitable for analysis by GC/MS in solid waste, soil, air, and water extracts. Method 8270 contains SVOCs across several analyte class types from acids, bases, neutral compounds, and polyaromatic hydrocarbons (PAHs); this method also has detailed specifications and requirements for the Quantitative Analysis of SVOCs.

The availability of helium (He) has been a concern for several years, but interest in transitioning to alternative carrier gases, such as hydrogen (H₂) has significantly increased in recent years. However, existing MS systems have issues with hydrogenation of some functional groups, such as nitro compounds, or dechlorination of heavily chlorinated compounds; these issues would alter the mass spectra of a peak in the total ion chromatogram (TIC) and lead to potential misidentification of compounds. A newly designed extractor source for the Agilent 5977B Inert Plus GC/MSD addresses these H₂-related issues and helps improve performance with H₂ carrier gas in GC/MS. The HydroInert source with H₂ carrier gas retains mass spectral fidelity and can allow users to continue to use existing He-based mass spectral libraries and quantitative methods.

This application note demonstrates the ability of the HydroInert source to allow the use of H₂ carrier gas, while retaining critical functional groups, such as nitro groups and halogens. Retention of mass spectral fidelity is a breakthrough for the use of H₂ carrier gas with GC/MS systems, especially for environmental analyses such as EPA method 8270. Also, a method for EPA 8270 has been developed that retains similar sensitivity to a He carrier gas analysis, which allows for most compounds to be calibrated between 0.1 to 100 µg/mL with fewer than 20% of compounds requiring linear curve fits.

Experimental

A set of stock standards containing 119 target compounds and surrogates was selected to provide a representative mixture of acids, bases, and neutral compounds, as well as comprising various compound classes, from nitrophenols to PAHs. The nine stock standards of target analytes were at concentrations of 2,000 µg/mL; part numbers for these stock standards are as follows: SVM-160, SVM-121, SVM-122, SVM-123, SVM-124, SVM-125, SVM-126-1, SVM-127, and US-211. Pyridine was diluted from a pure standard to 1,000 µg/mL as a working standard. The surrogate standard (part number ISM-332) contained six compounds at 2,000 µg/mL, indicated in Table 1. An internal standard mixture of six deuterated PAHs (part number ISM-560) was used for recovery and calibration. The stock standards were combined and diluted in dichloromethane to make a working standard at 200 µg/mL. The working standard was then diluted to form the following nominal concentrations for the targets and surrogates for calibration standards: 0.1, 0.2, 0.5, 0.8, 1, 2, 5, 10, 20, 35, 50, 75, and 100 µg/mL. Internal standards were added to each calibration standard at a concentration level of 40 µg/mL. Table 1 lists the compounds that were used in the study. The compound numbers in Table 1 were assigned based on the retention order of the targets and surrogates, with the internal standards listed at the end of the table out of the retention order.

The tuning standard (part number GCM-150), containing a mixture of benzidine, pentachlorophenol, 4,4’- dichlorodiphenyltrichloroethane (4,4’-DDT), and decafluorotriphenylphosphine (DFTPP), was diluted to 25 µg/mL and used to obtain the MS calibration and tuning settings.

A composite mixture of soils extracted with dichloromethane was prepared for EPA method 8270 analysis. The mixture was a representative matrix residue that is typically encountered in the lab and was procured from Pace Analytical (Mt. Juliet, TN).
<table>
<thead>
<tr>
<th>No.</th>
<th>Compound</th>
<th>No.</th>
<th>Compound</th>
<th>No.</th>
<th>Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>N-Nitrosodimethylamine</td>
<td>43</td>
<td>4-Chloro-3-methylphenol</td>
<td>85</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>2</td>
<td>Pyridine</td>
<td>44</td>
<td>2-Methylnapthalene</td>
<td>86</td>
<td>Pentachloronitrobenzene</td>
</tr>
<tr>
<td>3</td>
<td>2-picoline</td>
<td>45</td>
<td>Hexachlorocyclopentadiene</td>
<td>87</td>
<td>Propyzamide</td>
</tr>
<tr>
<td>4</td>
<td>N-Nitroso-N-methylethylamine</td>
<td>46</td>
<td>1,2,4,5-Tetrachlorobenzene</td>
<td>88</td>
<td>Dinoeb</td>
</tr>
<tr>
<td>5</td>
<td>Methyl methanesulfonate</td>
<td>47</td>
<td>2,4,6-Trichlorophenol</td>
<td>89</td>
<td>Disulfoton</td>
</tr>
<tr>
<td>6</td>
<td>2-Fluorophenol</td>
<td>48</td>
<td>2,4,5-Trichlorophenol</td>
<td>90</td>
<td>Phenanthrene</td>
</tr>
<tr>
<td>7</td>
<td>N-Nitrosodiethyamine</td>
<td>49</td>
<td>2-Fluorobiphenyl (surrogate)</td>
<td>91</td>
<td>Anthracene</td>
</tr>
<tr>
<td>8</td>
<td>Ethyl methanesulfonate</td>
<td>50</td>
<td>2-Chloronaphthalene</td>
<td>92</td>
<td>Methyl parathion</td>
</tr>
<tr>
<td>9</td>
<td>Phenol-d$_6$ (surrogate)</td>
<td>51</td>
<td>1-Chloronaphthalene</td>
<td>93</td>
<td>Dibutyl phthalate</td>
</tr>
<tr>
<td>10</td>
<td>Phenol</td>
<td>52</td>
<td>2-Nitroaniline</td>
<td>94</td>
<td>Parathion</td>
</tr>
<tr>
<td>11</td>
<td>Aniline</td>
<td>53</td>
<td>Dimethyl phthalate</td>
<td>95</td>
<td>4-Nitroquinoline-1-oxide</td>
</tr>
<tr>
<td>12</td>
<td>Bis(2-chloroethyl) ether</td>
<td>54</td>
<td>2,6-Dinitrotoluene</td>
<td>96</td>
<td>Fluoranthen</td>
</tr>
<tr>
<td>13</td>
<td>2-Chlorophenol</td>
<td>55</td>
<td>Acenaphthyline</td>
<td>97</td>
<td>Benzidine</td>
</tr>
<tr>
<td>14</td>
<td>1,3-Dichlorobenzene</td>
<td>56</td>
<td>m-Nitroaniline</td>
<td>98</td>
<td>Pyrene</td>
</tr>
<tr>
<td>15</td>
<td>1,4-Dichlorobenzene</td>
<td>57</td>
<td>Acenaphthene</td>
<td>99</td>
<td>Aramite</td>
</tr>
<tr>
<td>16</td>
<td>Benzyl alcohol</td>
<td>58</td>
<td>2,4-Dinitrophenol</td>
<td>100</td>
<td>p-ter-Terphenyl-d$_6$ (surrogate)</td>
</tr>
<tr>
<td>17</td>
<td>1,2-Dichlorobenzene</td>
<td>59</td>
<td>4-Nitrophenol</td>
<td>101</td>
<td>Aramite II</td>
</tr>
<tr>
<td>18</td>
<td>2-Methylphenol (o-cresol)</td>
<td>60</td>
<td>Pentachlorobenzene</td>
<td>102</td>
<td>p-(Dimethylamino)azobenzene</td>
</tr>
<tr>
<td>19</td>
<td>Bis(2-chloro-1-methyl) ether</td>
<td>61</td>
<td>2,4-Dinitrotoluene</td>
<td>103</td>
<td>Chlorobenzilate</td>
</tr>
<tr>
<td>20</td>
<td>1-Nitrosopyrrolidine</td>
<td>62</td>
<td>Dibenzofuran</td>
<td>104</td>
<td>3,3’-Dimethylenzidine</td>
</tr>
<tr>
<td>21</td>
<td>o-Toluidine</td>
<td>63</td>
<td>1-Naphthalenamine</td>
<td>105</td>
<td>Benzy1 butyl phosphate</td>
</tr>
<tr>
<td>22</td>
<td>N-Nitrosodi-n-propylamine</td>
<td>64</td>
<td>2,3,4,6-Tetrachlorobenzene</td>
<td>106</td>
<td>3,3’-Dichlorobenzidine</td>
</tr>
<tr>
<td>23</td>
<td>Acetophenone</td>
<td>65</td>
<td>2-Naphthalenamine</td>
<td>107</td>
<td>Benz[a]anthracene</td>
</tr>
<tr>
<td>24</td>
<td>4-Nitrosomorpholine</td>
<td>66</td>
<td>Diethyl phthalate</td>
<td>108</td>
<td>Chrysene</td>
</tr>
<tr>
<td>25</td>
<td>o-Toluidine</td>
<td>67</td>
<td>Thionazin</td>
<td>109</td>
<td>Bis(2-ethylhexyl) phthalate</td>
</tr>
<tr>
<td>26</td>
<td>Hexachloroethane</td>
<td>68</td>
<td>Fluorene</td>
<td>110</td>
<td>Di-n-octyl phthalate</td>
</tr>
<tr>
<td>27</td>
<td>Nitrobenzene-d$_6$ (surrogate)</td>
<td>69</td>
<td>4-Chlorophenyl phenyl ether</td>
<td>111</td>
<td>7,12-Dimethylenz[a]anthracene</td>
</tr>
<tr>
<td>28</td>
<td>Nitrobenzene</td>
<td>70</td>
<td>5-Nitro-o-toluidine</td>
<td>112</td>
<td>Benzo[b]fluoranthen</td>
</tr>
<tr>
<td>29</td>
<td>N-Nitrosopiperidine</td>
<td>71</td>
<td>4-Nitroaniline</td>
<td>113</td>
<td>Benzo[k]fluoranthen</td>
</tr>
<tr>
<td>30</td>
<td>Isophorone</td>
<td>72</td>
<td>2-Methyl, 4,6-dinitrophenol</td>
<td>114</td>
<td>Benzo[a]pyrene</td>
</tr>
<tr>
<td>31</td>
<td>2-Nitrophenol</td>
<td>73</td>
<td>Diphenylamine</td>
<td>115</td>
<td>3-Methylcholanthen</td>
</tr>
<tr>
<td>32</td>
<td>2,4-Dimethylphenol</td>
<td>74</td>
<td>Azobenzene</td>
<td>116</td>
<td>Dibenz[a]acridine</td>
</tr>
<tr>
<td>33</td>
<td>Benzoic acid</td>
<td>75</td>
<td>2,4,6-Tribromophenol</td>
<td>117</td>
<td>Indeno(1,2,3-cd)pyrene</td>
</tr>
<tr>
<td>34</td>
<td>Bis(2-chloroethoxy)methane</td>
<td>76</td>
<td>Sulforip</td>
<td>118</td>
<td>Dibenz[a]anthracene</td>
</tr>
<tr>
<td>35</td>
<td>2,4-Dichlorophenol</td>
<td>77</td>
<td>Diolate I</td>
<td>119</td>
<td>Benzo[gh]pyrene</td>
</tr>
<tr>
<td>36</td>
<td>1,2,4-Trichlorobenzene</td>
<td>78</td>
<td>Diolate II</td>
<td>120</td>
<td>1,4-Dichlorobenzene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>37</td>
<td>Naphthalene</td>
<td>79</td>
<td>Phorlate</td>
<td>121</td>
<td>Naphthalene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>38</td>
<td>a,a-Dimethylphenothalamine</td>
<td>80</td>
<td>Phenacetin</td>
<td>122</td>
<td>Acenaphthalene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>39</td>
<td>p-Chloroaniline</td>
<td>81</td>
<td>4-Bromophenyl phenyl ether</td>
<td>123</td>
<td>Phenanthrene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>40</td>
<td>2,6-Dichlorophenol</td>
<td>82</td>
<td>Hexachlorobenzene</td>
<td>124</td>
<td>Chrysene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>41</td>
<td>Hexachlorobutadiene</td>
<td>83</td>
<td>Dimethoate</td>
<td>125</td>
<td>Perylene-d$_6$ (internal standard)</td>
</tr>
<tr>
<td>42</td>
<td>N-nitrosodibutylamine</td>
<td>84</td>
<td>4-Aminobiphenyl</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Instrumental methods
The Agilent 8890 GC system was configured with an Agilent J&W DB-5ms Ultra Inert column (part number 121-5523UI) interfaced with an Agilent 5977B Inert Plus MS system with an Agilent HydroInert source. Table 2 summarizes the GC/MS instrumentation and consumables used in this study. The GC and MSD method parameters (Table 3) have been optimized to provide a 12-minute method, while retaining the required resolution for isomer pairs and following the EPA method 8270 guidelines for method parameters, such as scan range and scan rate.

Instrumentation

Table 2. GC and MSD instrumentation and consumables.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC</td>
<td>Agilent 8890 GC system</td>
</tr>
<tr>
<td>MS</td>
<td>Agilent 5977B Inert Plus GC/MSD</td>
</tr>
<tr>
<td>Source</td>
<td>Agilent HydroInert source with 9 mm HydroInert extraction lens</td>
</tr>
<tr>
<td>Syringe</td>
<td>Agilent Blue Line autosampler syringe, 10 µL, PTFE-tip plunger (part number G4513-80203)</td>
</tr>
<tr>
<td>Column</td>
<td>Agilent DB-5ms Ultra Inert, 20 m × 0.18 mm, 0.36 µm (part number 121-5523UI)</td>
</tr>
<tr>
<td>Inlet Liner</td>
<td>Agilent Ultra Inert inlet liner, split, low pressure drop, glass wool (part number 5190-2295)</td>
</tr>
</tbody>
</table>

Instrument conditions

Table 3. GC and MSD instrument conditions.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Injection Volume</td>
<td>1 µL</td>
</tr>
<tr>
<td>Inlet</td>
<td>230 °C</td>
</tr>
<tr>
<td>Column Temperature Program</td>
<td>40 °C (0 min hold) 30 °C/min to 320 °C (hold 2 min)</td>
</tr>
<tr>
<td>Carrier Gas and Flow Rate</td>
<td>H₂, 1.2 mL/min constant flow</td>
</tr>
<tr>
<td>Transfer Line Temperature</td>
<td>320 °C</td>
</tr>
<tr>
<td>Ion Source Temperature</td>
<td>300 °C</td>
</tr>
<tr>
<td>Quadrupole Temperature</td>
<td>150 °C</td>
</tr>
<tr>
<td>Scan</td>
<td>35 to 500 m/z</td>
</tr>
<tr>
<td>Tune</td>
<td>etune.u</td>
</tr>
<tr>
<td>Gain Factor</td>
<td>0.5</td>
</tr>
<tr>
<td>Threshold</td>
<td>0</td>
</tr>
<tr>
<td>A/D Samples</td>
<td>4</td>
</tr>
</tbody>
</table>

Method development
Switching carrier gas from He to H₂ introduced several challenges for EPA method 8270 analyses with a GC/MS single quadrupole instrument. Balance between sensitivity changes, inlet pressure and flow rates, and column capacity and dimensions must be managed to attain the required calibration range of 0.1 to 100 µg/mL for most compounds. For example, if the typical EPA method 8270 analysis with He carrier gas used a 30 m × 0.25 mm, 0.25 µm DB-5ms Ultra Inert column was changed to use a 20 m × 0.18 mm, 0.18 µm DB-5ms Ultra Inert column for H₂ carrier gas, this 20 m column would have ~33% of the 30 m column capacity, requiring changes to the injection parameters to avoid column overload. However, when a 20:1 split injection was used, limitations in sensitivity were observed with issues of reaching below 0.5 µg/mL injected concentration (25 ng/mL on column); using etune.u did not solve the issue. Another investigated method used the 30 m × 0.25 mm, 0.25 µm DB-5ms Ultra Inert column with a pulsed splitless injection and 1.5 mL/min flow rate. This method could reach the 0.1 µg/mL lower-end concentration for most compounds but had issue with severely fronting peaks above ~75 µg/mL, indicating overload, which also caused an increase in linear fits. A pulsed split injection with 10:1 split was tested for the 30 m column method with an atune.u tune, but most compounds were not detected at 0.1 µg/mL. For the column referenced in this work (20 m × 0.18 mm, 0.36 µm DB-5ms Ultra Inert), various injection parameters and both atune and etune algorithms were tested. The final method parameters listed in Table 3 provided the best balance between column capacity, sensitivity, and ability to produce calibration results in the 0.1 to 100 µg/mL range. While atune would be preferred, the lowest concentration tended to end at 0.2 µg/mL for most of the compounds.
Results and discussion

Mass spectral fidelity

A major concern with H₂ carrier gas is changes in the mass spectra of nitro compounds and heavily halogenated compounds. In the presence of H₂, high temperature, and metal surfaces, nitro functional groups are hydrogenated to amines, while heavily chlorinated compounds are dechlorinated; all these factors are present in the mass spectrometer. The following is an example of the benefits of the HydroInert source with nitrobenzene. In an experiment with an extractor source with a 3 mm extraction lens, H₂ was used as the carrier gas, where nitrobenzene was one of the compounds in the mixture (part number SVM-122-1). Hydrogenation of nitrobenzene (molecular weight (MW) 123 m/z) will form aniline (MW 93 m/z). When reviewing the mass spectrum under the TIC peak for the extractor source and H₂ carrier gas, the mass spectrum in Figure 1A was observed. There is a large abundance of 93 m/z and low 123 m/z, indicating conversion of nitrobenzene to aniline in the source; this is confirmed to occur in the source because the mass spectrum is observed at the retention time of nitrobenzene, which is well separated from aniline. Comparatively, the same mixture containing nitrobenzene was tested on a HydroInert source (with a 9 mm extraction lens), where we observe the expected distribution of 123 and 93 m/z in the mass spectrum (Figure 1B), indicating that the nitrobenzene is retained in the source and not converted to aniline. This comparison can also be reviewed in the extracted ion chromatograms (EICs) shown in Figure 2A (for the extractor source conversion) and 2B (for HydroInert source retention of nitrobenzene), where there is an improved 123/93 ratio using the HydroInert source, while the extractor source EIC overlay shows significant conversion to 93 m/z and significant tailing.

Figure 1. Mass spectra for peak eluting at nitrobenzene retention time with H₂ carrier gas in (A) extractor source with 3 mm extraction lens showing hydrogenation to aniline with the abundant 93 m/z ion and (B) Agilent HydroInert source, showing an improved mass spectrum that correlates to nitrobenzene.
A critical component of EPA method 8270 is the tune criteria associated with the ion ratios of DFTPP. This method used the etune algorithm for the factor of 10 increase in signal to balance the split injection. For the GC/MS single quadrupole system, the DFTPP ion ratio criteria from Table 3 of EPA methods 8270E and 8270D were used to test the HydroInert source with H\textsubscript{2} carrier gas.

There is always concern for inlet and column cleanliness for EPA method 8270 to work, no matter the carrier gas; DDT, pentachlorophenol, and benzidine are used to track inlet breakdown and column health. Increased DDT breakdown indicates a need for inlet maintenance, while increasing tailing factors of benzidine and pentachlorophenol inform the user to trim or change the column. With the introduction of H\textsubscript{2} carrier gas, users may be worried about increased reactions of active compounds, such as DDT, in the inlet; the recommendation is to lower the inlet temperature to 230 to 250 °C or use a temperature-programmable inlet, such as the multimode inlet to protect the active compounds, while still being able to increase the temperature to 320 °C and drive out the PAHs. This study used the most common inlet existing in laboratories, the split/splitless inlet, and ran the inlet at 230 °C.

Reviewing the results of the GC/MS tuning mixture for DDT breakdown and compound tailing factors, the DDT (%) breakdown was 0.2%, the pentachlorophenol tailing factor was 1.2, and the benzidine tailing factor was 1.3. All values are within the EPA method 8270 criteria of <20% DDT breakdown and tailing factors <2.0.

Calibration criteria
The initial calibration consisted of 13 levels across the concentration range of 0.1 to 100 µg/mL for this 12-minute method. Figure 3 is a TIC of the target analytes, surrogates, and internal standards.

---

**Table 4.** DFTPP ions, abundance criteria from EPA method 8270D and 8270E\textsuperscript{1,2}, measured relative abundance, and pass/fail of the relative abundance.

<table>
<thead>
<tr>
<th>Target Mass (m/z)</th>
<th>Ion Abundance Criteria</th>
<th>Measured Relative Abundance</th>
<th>Pass/Fail</th>
</tr>
</thead>
<tbody>
<tr>
<td>51</td>
<td>*10 to 80% of 198 m/z</td>
<td>38.5%</td>
<td>Pass</td>
</tr>
<tr>
<td>68</td>
<td>&gt;2% of 69 m/z</td>
<td>1.0%</td>
<td>Pass</td>
</tr>
<tr>
<td>69</td>
<td>Present</td>
<td>36.5%</td>
<td>Pass</td>
</tr>
<tr>
<td>70</td>
<td>&lt;2% of 69 m/z</td>
<td>0.4%</td>
<td>Pass</td>
</tr>
<tr>
<td>127</td>
<td>*10 to 80% of 198 m/z</td>
<td>54.4%</td>
<td>Pass</td>
</tr>
<tr>
<td>197</td>
<td>&lt;2% of 198 m/z</td>
<td>0.0%</td>
<td>Pass</td>
</tr>
<tr>
<td>198</td>
<td>Base peak or present</td>
<td>51.6%</td>
<td>Pass</td>
</tr>
<tr>
<td>199</td>
<td>5 to 9% of 198 m/z</td>
<td>5.0%</td>
<td>Pass</td>
</tr>
<tr>
<td>275</td>
<td>10 to 60% of base peak</td>
<td>30.4%</td>
<td>Pass</td>
</tr>
<tr>
<td>365</td>
<td>&gt;1% of base peak</td>
<td>4.9%</td>
<td>Pass</td>
</tr>
<tr>
<td>441</td>
<td>&lt;150% of 443 m/z present, *but &lt;24% of 442</td>
<td>83.1%, *15.7%</td>
<td>Pass</td>
</tr>
<tr>
<td>442</td>
<td>Base peak or present</td>
<td>100% (base peak)</td>
<td>Pass</td>
</tr>
<tr>
<td>443</td>
<td>15 to 24% of 442 m/z</td>
<td>18.9%</td>
<td>Pass</td>
</tr>
</tbody>
</table>

* Denotes 8270D requirement difference from EPA method 8270E requirement.
Critical pair resolution

With the shorter method time and a different column, critical pair resolution above 50% was verified for phenanthrene and anthracene (EIC 178 \(m/z\)), benz[a]anthracene and chrysene (EIC 228 \(m/z\)), and benzo(b)fluoranthene and benzo(k)fluoranthene (EIC 252 \(m/z\)). All three isomer pairs are shown in Figure 4 at a midlevel concentration of 5 µg/mL; phenanthrene and anthracene (Figure 4A) have baseline resolution, benz[a]anthracene and chrysene (Figure 4B) are nearly baseline-resolved, and benzo(b)fluoranthene and benzo(k)fluoranthene (Figure 4C) are over 50% resolved, satisfying the EPA method 8270 criteria.

Figure 3. TIC of the 20 µg/mL calibration standard containing 119 target analytes and surrogates and six internal standards using \(H_2\) carrier gas and the Agilent HydroInert source.

Figure 4. Midlevel standard (5 µg/mL) EICs for critical isomer pairs: (A) phenanthrene and anthracene (EIC 178 \(m/z\)); (B) benz[a]anthracene and chrysene (EIC 228 \(m/z\)); (C) benzo(b)fluoranthene and benzo(k)fluoranthene (EIC 252 \(m/z\)).
Response factor comparison between hydrogen and helium carrier gases

When moving an analysis from He to H$_2$ carrier gas, there is always concern about maintenance of response factors (RFs) and sensitivity for single quadrupole systems. Table 5 lists the RFs from EPA method 8270E guidance criteria (Table 4); RFs from a GC/MS analysis with He carrier gas when using a splitless injection, then a pulsed split injection, and RFs for GC/MS analysis with the HydroInert source and H$_2$ carrier gas. Since the H$_2$ method uses a split injection, the pulsed split injection with He provides a good comparison, while the splitless He data is the traditional analysis. The RFs from EPA method 8270E (Table 4) are guidance criteria and not requirements to pass the method, but ideally the RFs should be like these guidance values. For the He (splitless injection) GC/MS analysis, two compounds have RFs below the guidance criteria: hexachloroethane and N-nitroso-di-n-propylamine; these compounds' RFs are also low for the H$_2$ HydroInert results. For the H$_2$ HydroInert GC/MS analysis, five additional compounds have RFs below the guidance criteria, where four are within 0.1 points. For example, the guidance RF criteria for bis(2-chloroethyl)ether is 0.7 and the H$_2$ HydroInert GC/MS RF was 0.6. For the pulsed split He GC/MS results, all reported RFs match or are higher than the guidance from the EPA, but this data set did not report RFs for the seven indicated compounds in Table 5. In total, only seven compounds of the 72 listed in Table 5 had RFs lower than the EPA guidance for the H$_2$ HydroInert GC/MS results; five of these were within 0.1 points of the guidance RF value, and the other two RF values were within 0.3 or fewer points of the guidance.

<table>
<thead>
<tr>
<th>Compound</th>
<th>From EPA 8270E</th>
<th>He GC/MS</th>
<th>He GC/MS, Pulsed Split</th>
<th>H$_2$ HydroInert GC/MS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acenaphthene</td>
<td>0.9</td>
<td>1.3</td>
<td>1.1</td>
<td>1.1</td>
</tr>
<tr>
<td>Acenaphthylene</td>
<td>0.9</td>
<td>1.9</td>
<td>2.0</td>
<td>1.4</td>
</tr>
<tr>
<td>Acetophenone</td>
<td>0.01</td>
<td>1.2</td>
<td>–</td>
<td>0.4</td>
</tr>
<tr>
<td>Anthracene</td>
<td>0.7</td>
<td>1.1</td>
<td>1.1</td>
<td>1.0</td>
</tr>
<tr>
<td>Benzo(a)anthracene</td>
<td>0.8</td>
<td>1.4</td>
<td>1.3</td>
<td>1.5</td>
</tr>
<tr>
<td>Benzo(a)pyrene</td>
<td>0.7</td>
<td>1.2</td>
<td>1.0</td>
<td>0.9</td>
</tr>
<tr>
<td>Benzo(b)fluoranthene</td>
<td>0.7</td>
<td>1.4</td>
<td>1.0</td>
<td>1.2</td>
</tr>
<tr>
<td>Benzo(g,h,i)perylene</td>
<td>0.5</td>
<td>1.1</td>
<td>1.1</td>
<td>1.0</td>
</tr>
<tr>
<td>Benzo(k)fluoranthene</td>
<td>0.7</td>
<td>1.2</td>
<td>1.1</td>
<td>1.2</td>
</tr>
<tr>
<td>Bis(2-chloroethoxy)methane</td>
<td>0.3</td>
<td>0.4</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>Bis(2-chloroethyl)ether</td>
<td>0.7</td>
<td>0.8</td>
<td>1.1</td>
<td>0.6</td>
</tr>
<tr>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>0.01</td>
<td>0.8</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>4-Bromophenyl-phenyl ether</td>
<td>0.1</td>
<td>0.3</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>Butyl benzyl phthalate</td>
<td>0.01</td>
<td>0.6</td>
<td>0.5</td>
<td>0.3</td>
</tr>
<tr>
<td>4-Chloroaniline</td>
<td>0.01</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>4-Chloro-3-methylphenol</td>
<td>0.2</td>
<td>0.3</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>2-Chloronaphthalene</td>
<td>0.8</td>
<td>2.4</td>
<td>1.2</td>
<td>1.0</td>
</tr>
<tr>
<td>2-Chlorophenol</td>
<td>0.8</td>
<td>0.8</td>
<td>1.2</td>
<td>0.7</td>
</tr>
<tr>
<td>4-Chlorophenyl-phenyl ether</td>
<td>0.4</td>
<td>0.7</td>
<td>0.6</td>
<td>0.5</td>
</tr>
<tr>
<td>Chrysene</td>
<td>0.7</td>
<td>1.2</td>
<td>1.2</td>
<td>1.1</td>
</tr>
<tr>
<td>Dibenzo(a,h)anthracene</td>
<td>0.4</td>
<td>1.1</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Dibenzofuran</td>
<td>0.8</td>
<td>1.7</td>
<td>1.7</td>
<td>1.5</td>
</tr>
<tr>
<td>Di-n-butyl phthalate</td>
<td>0.01</td>
<td>1.3</td>
<td>1.2</td>
<td>0.8</td>
</tr>
<tr>
<td>3,3’-Dichlorobenzidine</td>
<td>0.01</td>
<td>0.5</td>
<td>–</td>
<td>0.4</td>
</tr>
<tr>
<td>2,4-Dichlorophenol</td>
<td>0.2</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>Diethyl phthalate</td>
<td>0.01</td>
<td>1.4</td>
<td>1.3</td>
<td>1.0</td>
</tr>
<tr>
<td>Dimethyl phthalate</td>
<td>0.01</td>
<td>1.4</td>
<td>1.3</td>
<td>1.0</td>
</tr>
<tr>
<td>2,4-Dimethylphenol</td>
<td>0.2</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>4,6-Dinitro-2-methylphenol</td>
<td>0.01</td>
<td>0.2</td>
<td>–</td>
<td>0.1</td>
</tr>
<tr>
<td>2,4-Dinitrophenol</td>
<td>0.01</td>
<td>0.2</td>
<td>–</td>
<td>0.1</td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>0.2</td>
<td>0.4</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>2,6-Dinitrotoluene</td>
<td>0.2</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>0.01</td>
<td>1.3</td>
<td>1.4</td>
<td>0.8</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>0.6</td>
<td>1.2</td>
<td>1.2</td>
<td>1.2</td>
</tr>
<tr>
<td>Fluorene</td>
<td>0.9</td>
<td>1.3</td>
<td>1.3</td>
<td>1.2</td>
</tr>
<tr>
<td>Hexachlorobenzene</td>
<td>0.1</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>0.01</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>Hexachlorocyclopentadiene</td>
<td>0.05</td>
<td>0.3</td>
<td>0.4</td>
<td>0.1</td>
</tr>
<tr>
<td>Hexachloroethane</td>
<td>0.3</td>
<td>0.2</td>
<td>0.5</td>
<td>0.1</td>
</tr>
<tr>
<td>Indeno(1,2,3-cd)pyrene</td>
<td>0.5</td>
<td>1.2</td>
<td>1.0</td>
<td>1.2</td>
</tr>
<tr>
<td>Isophorone</td>
<td>0.4</td>
<td>0.6</td>
<td>0.5</td>
<td>0.4</td>
</tr>
</tbody>
</table>
### Compound Response Factors

<table>
<thead>
<tr>
<th>Compound</th>
<th>From EPA 8270E</th>
<th>He GC/MS</th>
<th>He GC/MS, Pulsed Split</th>
<th>H₂ HydroInert GC/MS</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-Methylnaphthalene</td>
<td>0.4</td>
<td>0.7</td>
<td>0.7</td>
<td>0.7</td>
</tr>
<tr>
<td>2-Methylphenol</td>
<td>0.7</td>
<td>0.7</td>
<td>1.0</td>
<td>0.6</td>
</tr>
<tr>
<td>4-Methylphenol</td>
<td>0.6</td>
<td>1.0</td>
<td>1.1</td>
<td>0.3</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>0.7</td>
<td>1.1</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>2-Nitroaniline</td>
<td>0.01</td>
<td>0.4</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>3-Nitroaniline</td>
<td>0.01</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>4-Nitroaniline</td>
<td>0.01</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>Nitrobenzene</td>
<td>0.2</td>
<td>0.3</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>2-Nitrophenol</td>
<td>0.1</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>4-Nitrophenol</td>
<td>0.01</td>
<td>0.2</td>
<td>–</td>
<td>0.1</td>
</tr>
<tr>
<td>N-Nitroso-di-n-propylamine</td>
<td>0.5</td>
<td>0.4</td>
<td>0.7</td>
<td>0.4</td>
</tr>
<tr>
<td>N-Nitrosodiphenylamine</td>
<td>0.01</td>
<td>2.1</td>
<td>0.6</td>
<td>0.9</td>
</tr>
<tr>
<td>2,2'-Oxybis-(1-chloropropane)</td>
<td>0.01</td>
<td>0.5</td>
<td>1.1</td>
<td>0.5</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>0.05</td>
<td>0.2</td>
<td>–</td>
<td>0.1</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>0.7</td>
<td>1.2</td>
<td>1.1</td>
<td>1.1</td>
</tr>
<tr>
<td>Phenol</td>
<td>0.8</td>
<td>0.9</td>
<td>1.4</td>
<td>0.7</td>
</tr>
<tr>
<td>Pyrene</td>
<td>0.6</td>
<td>1.3</td>
<td>1.3</td>
<td>1.2</td>
</tr>
<tr>
<td>1,2,4,5-Tetrachlorobenzene</td>
<td>0.01</td>
<td>0.4</td>
<td>–</td>
<td>0.3</td>
</tr>
<tr>
<td>2,3,4,6-Tetrachlorophenol</td>
<td>0.01</td>
<td>0.4</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>2,4,5-Trichlorophenol</td>
<td>0.2</td>
<td>0.3</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>2,4,6-Trichlorophenol</td>
<td>0.2</td>
<td>0.3</td>
<td>0.4</td>
<td>0.2</td>
</tr>
</tbody>
</table>

**Calibration results**

A multipoint calibration was performed with the maximum of 13 concentration levels and the relative RF was determined for each compound and calibration level. The mean RF was calculated to build the calibration curve of each compound along with the relative standard deviation (RSD). The average RF %RSD must be <20%, which is the preferred passing criteria; if not achievable with at least six calibration levels, an R² value >0.990 is required for a linear curve fit, or a quadratic fit may be used. Accuracy for the lowest data point must be within 30% of estimated concentration with a minimum of six points for the curve fit. Results for the initial calibration using H₂ carrier gas and the HydroInert source can be found in Table 6.

Of 119 compounds, 14 compounds required linear fits and one quadratic fit was required. Table 6 summarizes the calibration results for the 119 target compounds and surrogates with average RF %RSD values, and the lowest and highest concentration level if the values are different from the full calibration range (0.1 to 100 µg/mL). Over 87% of the compounds pass the calibration criteria with an average RF %RSD below 20%. An increase in the number of compounds requiring linear fits is predictable since H₂ is more reactive than He and the inlet is set to a lower temperature to avoid formation of hydrochloric acid in the presence of higher temperatures and water in the inlet. Use of a multimode inlet may result in improved heavy phthalate and PAH results.

Sensitivity loss with H₂ carrier gas and existing mass spectrometer systems has been well reported. Due to this concern, particular attention was paid to the calibration range and verifying that most compounds were able to achieve the same calibration range as previous He analyses. On the topic of sensitivity, 96 compounds were analyzed in a previous application for EPA method 8270 with He carrier gas on GC/MS. Comparing these compounds with the same set using the HydroInert source and H₂ carrier gas (also GC/MS), 15 compounds have a narrower calibration range, where six compounds are only narrower by one concentration level starting at 200 ng/mL instead of 100 ng/mL, and four compounds start at 500 ng/mL. For benzoic acid, the HydroInert source with H₂ carrier gas has the same calibration range of 0.8 to 100 µg/mL, as observed with He carrier gas on a GC/MS; 2,4-dinitrophenol passed calibration criteria with average RF for the range of 0.5 to 100 µg/mL with H₂ and the HydroInert source, while helium-collected data required a linear fit for the same calibration range. Pentachlorophenol also had matched calibration ranges between the He and H₂ results of 0.5 to 100 µg/mL, but the H₂ data required a linear fit. On the positive side, some compounds had wider calibration ranges with H₂ and the HydroInert source, such as 4-nitrophenol and 2-methyl-4,6-dinitrophenol, which each included an extra calibration level of 100 and 200 ng/mL, respectively. Also, these two compounds did not require linear curve fits, but passed calibration criteria with average RF %RSD values of 18.7% for 4-nitrophenol and 19.7% for 2-methyl-4,6 dinitrophenol. In total, 24 compounds out of 119 had narrower calibration ranges than the default of 0.1 to 100 µg/mL. The use of H₂ carrier gas with the HydroInert source retains the sensitivity range for over 84% of the previously tested 96 SVOCs.
Table 6. Initial calibration results for 119 target compounds and surrogates for H$_2$ carrier gas and the Agilent HydroInert source for EPA method 8270.

<table>
<thead>
<tr>
<th>Name</th>
<th>Retention Time (min)</th>
<th>Average RF</th>
<th>Average RF %RSD</th>
<th>Curve Fit R$^2$</th>
<th>Curve Fit</th>
<th>Low Standard (µg/mL)</th>
<th>High Standard (µg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-Nitrosodimethylamine</td>
<td>1.339</td>
<td>0.273</td>
<td>7.41</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pyridine</td>
<td>1.372</td>
<td>0.459</td>
<td>15.39</td>
<td></td>
<td></td>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td>2-Picoline</td>
<td>1.705</td>
<td>0.561</td>
<td>5.89</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>N-Nitroso-N-methylethylamine</td>
<td>1.741</td>
<td>0.232</td>
<td>7.23</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Methyl methanesulfonate</td>
<td>1.890</td>
<td>0.256</td>
<td>15.04</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Fluorophenol</td>
<td>1.983</td>
<td>0.568</td>
<td>5.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>N-Nitroso-N-diethylamine</td>
<td>2.120</td>
<td>0.258</td>
<td>7.13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ethyl methanesulfonate</td>
<td>2.286</td>
<td>0.374</td>
<td>13.02</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenol-d$_6$</td>
<td>2.532</td>
<td>0.667</td>
<td>4.93</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenol</td>
<td>2.541</td>
<td>0.664</td>
<td>6.32</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aniline</td>
<td>2.583</td>
<td>0.968</td>
<td>7.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bis(2-chloroethyl) ether</td>
<td>2.617</td>
<td>0.616</td>
<td>10.72</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Chlorophenol</td>
<td>2.665</td>
<td>0.661</td>
<td>8.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>2.774</td>
<td>0.773</td>
<td>6.96</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>2.825</td>
<td>0.804</td>
<td>7.53</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzyl alcohol</td>
<td>2.892</td>
<td>0.442</td>
<td>12.90</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>2.931</td>
<td>0.756</td>
<td>7.53</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Methylphenol (o-cresol)</td>
<td>2.965</td>
<td>0.559</td>
<td>9.73</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bis(2-chloro-1-methylethyl) ether</td>
<td>2.998</td>
<td>0.545</td>
<td>11.21</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Nitrosopyrrolidine</td>
<td>3.068</td>
<td>0.260</td>
<td>6.02</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p-Cresol</td>
<td>3.074</td>
<td>0.333</td>
<td>7.00</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>N-Nitrosodi-n-propylamine</td>
<td>3.089</td>
<td>0.370</td>
<td>12.94</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetophenone</td>
<td>3.092</td>
<td>0.445</td>
<td>6.48</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Nitrosomorpholine</td>
<td>3.095</td>
<td>0.107</td>
<td>8.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>o-Toluidine</td>
<td>3.116</td>
<td>0.487</td>
<td>8.39</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hexachloroethane</td>
<td>3.180</td>
<td>0.112</td>
<td>8.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrobenzene-d$_6$</td>
<td>3.201</td>
<td>0.097</td>
<td>10.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrobenzene</td>
<td>3.216</td>
<td>0.197</td>
<td>6.59</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrosopiperidine</td>
<td>3.325</td>
<td>0.132</td>
<td>8.87</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Isophorone</td>
<td>3.395</td>
<td>0.433</td>
<td>7.86</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Nitrophenol</td>
<td>3.455</td>
<td>0.112</td>
<td>11.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4-Dimethylphenol</td>
<td>3.480</td>
<td>0.295</td>
<td>6.34</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzoic acid</td>
<td>3.519</td>
<td>0.117</td>
<td>0.9946</td>
<td>Linear</td>
<td>0.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bis(2-chloroethoxy)methane</td>
<td>3.558</td>
<td>0.345</td>
<td>8.69</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4-Dichlorophenol</td>
<td>3.637</td>
<td>0.243</td>
<td>13.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>3.710</td>
<td>0.356</td>
<td>10.34</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Naphthalene</td>
<td>3.773</td>
<td>0.978</td>
<td>8.27</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a,a-Dimethylphenethylamine</td>
<td>3.782</td>
<td>0.360</td>
<td>0.9976</td>
<td>Linear</td>
<td>0.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Chloroaniline</td>
<td>3.807</td>
<td>0.401</td>
<td>8.01</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,6-Dichlorophenol</td>
<td>3.816</td>
<td>0.232</td>
<td>16.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>3.873</td>
<td>0.177</td>
<td>19.36</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Name</td>
<td>Retention Time (min)</td>
<td>Average RF</td>
<td>Average RF %RSD</td>
<td>Curve Fit R²</td>
<td>Curve Fit</td>
<td>Low Standard (µg/mL)</td>
<td>High Standard (µg/mL)</td>
</tr>
<tr>
<td>----------------------------------</td>
<td>----------------------</td>
<td>------------</td>
<td>-----------------</td>
<td>--------------</td>
<td>-----------</td>
<td>----------------------</td>
<td>-----------------------</td>
</tr>
<tr>
<td>N-Nitrosobutylamine</td>
<td>4.079</td>
<td>0.172</td>
<td>9.34</td>
<td></td>
<td></td>
<td></td>
<td>0.2</td>
</tr>
<tr>
<td>4-Chloro-3-methylphenol</td>
<td>4.185</td>
<td>0.204</td>
<td>10.56</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>4.321</td>
<td>0.656</td>
<td>6.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hexachlorocyclopentadiene</td>
<td>4.455</td>
<td>0.136</td>
<td>0.9928</td>
<td>Linear</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,2,4,5-Tetrachlorobenzene</td>
<td>4.458</td>
<td>0.308</td>
<td>19.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4,6-Trichlorophenol</td>
<td>4.545</td>
<td>0.241</td>
<td>13.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4,5-Trichlorophenol</td>
<td>4.570</td>
<td>0.288</td>
<td>13.13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Fluorobiphenyl</td>
<td>4.618</td>
<td>0.613</td>
<td>9.30</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Chloronaphthalene</td>
<td>4.715</td>
<td>1.018</td>
<td>9.32</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Chloronaphthalene</td>
<td>4.733</td>
<td>1.003</td>
<td>9.15</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Nitroaniline</td>
<td>4.791</td>
<td>0.226</td>
<td>14.72</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dimethyl phthalate</td>
<td>4.948</td>
<td>1.005</td>
<td>10.34</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,6-Dinitrotoluene</td>
<td>4.994</td>
<td>0.153</td>
<td>17.84</td>
<td>0.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acenaphthylene</td>
<td>5.051</td>
<td>1.362</td>
<td>9.04</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>m-Nitroaniline</td>
<td>5.124</td>
<td>0.178</td>
<td>10.30</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acenaphthene</td>
<td>5.196</td>
<td>1.083</td>
<td>9.75</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4-Dinitrophenol</td>
<td>5.212</td>
<td>0.074</td>
<td>15.34</td>
<td>0.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Nitrophenol</td>
<td>5.260</td>
<td>0.143</td>
<td>18.74</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pentachlorobenzene</td>
<td>5.305</td>
<td>0.428</td>
<td>14.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>5.321</td>
<td>0.200</td>
<td>16.37</td>
<td>75</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dibenzofuran</td>
<td>5.339</td>
<td>1.486</td>
<td>9.57</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Naphthylamine</td>
<td>5.396</td>
<td>0.655</td>
<td>19.57</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,3,4,6-Tetrachlorophenol</td>
<td>5.436</td>
<td>0.177</td>
<td>0.9912</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Naphthylamine</td>
<td>5.463</td>
<td>0.908</td>
<td>8.77</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Diethyl Phthalate</td>
<td>5.536</td>
<td>0.978</td>
<td>12.37</td>
<td>0.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thionazin</td>
<td>5.599</td>
<td>0.142</td>
<td>16.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fluorene</td>
<td>5.620</td>
<td>1.242</td>
<td>9.88</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5-Nitro-o-toluidine</td>
<td>5.623</td>
<td>0.209</td>
<td>19.75</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Chlorophenyl phenyl ether</td>
<td>5.623</td>
<td>0.530</td>
<td>15.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Nitroaniline</td>
<td>5.626</td>
<td>0.206</td>
<td>0.9943</td>
<td>Linear</td>
<td>0.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2-Methyl, 4,6-dinitrophenol</td>
<td>5.654</td>
<td>0.098</td>
<td>19.68</td>
<td>0.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>5.717</td>
<td>0.943</td>
<td>9.95</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Azobenzene</td>
<td>5.754</td>
<td>0.397</td>
<td>5.84</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4,6-Tribromophenol</td>
<td>5.814</td>
<td>0.083</td>
<td>19.91</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sulfopep</td>
<td>5.863</td>
<td>0.082</td>
<td>0.9976</td>
<td>Quadratic</td>
<td>0.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Diellate I</td>
<td>5.963</td>
<td>0.144</td>
<td>7.38</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phorate</td>
<td>5.969</td>
<td>0.210</td>
<td>11.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phenacetin</td>
<td>5.972</td>
<td>0.224</td>
<td>12.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Bromophenyl phenyl ether</td>
<td>6.026</td>
<td>0.197</td>
<td>8.23</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Diellate II</td>
<td>6.038</td>
<td>0.050</td>
<td>10.31</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hexachlorobenzene</td>
<td>6.072</td>
<td>0.245</td>
<td>16.95</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dimethoate</td>
<td>6.099</td>
<td>0.141</td>
<td>16.58</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Aminobiphenyl</td>
<td>6.235</td>
<td>0.611</td>
<td>10.94</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Name</td>
<td>Retention Time (min)</td>
<td>Average RF</td>
<td>Average RF %RSD</td>
<td>Curve Fit R²</td>
<td>Curve Fit</td>
<td>Low Standard (µg/mL)</td>
<td>High Standard (µg/mL)</td>
</tr>
<tr>
<td>-------------------------------------</td>
<td>----------------------</td>
<td>------------</td>
<td>-----------------</td>
<td>--------------</td>
<td>-----------</td>
<td>----------------------</td>
<td>----------------------</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>6.235</td>
<td>0.101</td>
<td></td>
<td>0.9911</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Pentachloronitrobenzene</td>
<td>6.247</td>
<td>0.054</td>
<td>19.27</td>
<td></td>
<td></td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Propyzamide</td>
<td>6.293</td>
<td>0.204</td>
<td>14.45</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dinoseb</td>
<td>6.390</td>
<td>0.089</td>
<td>19.44</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Disulfoton</td>
<td>6.402</td>
<td>0.317</td>
<td></td>
<td>0.9666</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>6.411</td>
<td>1.091</td>
<td>14.31</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Anthracene</td>
<td>6.453</td>
<td>1.009</td>
<td>11.90</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Methyl parathion</td>
<td>6.708</td>
<td>0.124</td>
<td>10.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dibutyl phthalate</td>
<td>6.889</td>
<td>0.840</td>
<td>16.44</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Parathion</td>
<td>7.032</td>
<td>0.089</td>
<td>12.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Nitroquinoline-1-oxide</td>
<td>7.044</td>
<td>0.064</td>
<td>19.82</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fluoranthenes</td>
<td>7.395</td>
<td>1.188</td>
<td>8.54</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzidine</td>
<td>7.504</td>
<td>0.544</td>
<td>9.47</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pyrene</td>
<td>7.580</td>
<td>1.207</td>
<td>8.59</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aramite</td>
<td>7.710</td>
<td>0.044</td>
<td>18.03</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p-Terphenyl-d₁₄</td>
<td>7.716</td>
<td>0.422</td>
<td>14.16</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aramite II</td>
<td>7.770</td>
<td>0.044</td>
<td>12.41</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>p-(Dimethylamino)azobenzene</td>
<td>7.834</td>
<td>0.195</td>
<td></td>
<td>0.9919</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Chlorobenzilate</td>
<td>7.876</td>
<td>0.294</td>
<td>10.53</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3,3’-Dimethylbenzidine</td>
<td>8.107</td>
<td>0.466</td>
<td>17.39</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzyl butyl phthalate</td>
<td>8.128</td>
<td>0.343</td>
<td></td>
<td>0.9926</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>3,3’-Dichlorobenzidine</td>
<td>8.549</td>
<td>0.364</td>
<td></td>
<td>0.9939</td>
<td>Linear</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Benz[a]anthracene</td>
<td>8.570</td>
<td>1.443</td>
<td></td>
<td>0.9985</td>
<td>Linear</td>
<td>0.2</td>
<td></td>
</tr>
<tr>
<td>Chrysene</td>
<td>8.600</td>
<td>1.047</td>
<td>11.58</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bis(2-ethylhexyl) phthalate</td>
<td>8.612</td>
<td>0.502</td>
<td>17.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>9.118</td>
<td>0.832</td>
<td>16.61</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7,12-Dimethylbenz[a]anthracene</td>
<td>9.397</td>
<td>0.376</td>
<td></td>
<td>0.9947</td>
<td>Linear</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Benzo[b]fluoranthenes</td>
<td>9.400</td>
<td>1.198</td>
<td>17.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzo[k]fluoranthenes</td>
<td>9.421</td>
<td>1.170</td>
<td>16.60</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzo[a]pyrene</td>
<td>9.657</td>
<td>0.874</td>
<td>17.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3-Methylcholanthrene</td>
<td>9.954</td>
<td>0.328</td>
<td></td>
<td>0.9905</td>
<td>Linear</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Dibenz[a,j]acridine</td>
<td>10.523</td>
<td>0.594</td>
<td></td>
<td>0.9908</td>
<td>Linear</td>
<td>0.8</td>
<td></td>
</tr>
<tr>
<td>Indeno(1,2,3-cd)pyrene</td>
<td>10.720</td>
<td>1.210</td>
<td>19.76</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dibenz[a,h]anthracene</td>
<td>10.738</td>
<td>1.016</td>
<td>19.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Benzo[ghi]perylene</td>
<td>11.020</td>
<td>1.024</td>
<td>17.29</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
As an example of full calibration range retention, Figure 5 compares the linear range for nitrobenzene in He carrier gas (Figure 5A), and in H₂ carrier gas with the HydroInert source (Figure 5B). The average RF %RSDs are remarkably similar between the results for He carrier gas and H₂ carrier gas with the HydroInert source, at 6.33% RSD for He carrier gas, and 6.59% RSD for H₂ carrier gas and the HydroInert source. The qualifiers and raw spectrum for nitrobenzene in this data set can be reviewed to verify consistent mass spectra and ion fragment ratios for the HydroInert source with H₂ carrier gas.

Figure 6 shows (A) the nitrobenzene base peak EIC, (B) an overlay of the base peak and qualifier EICs, and (C) the raw mass spectrum, at calibration level 8 (10 µg/mL). In Figure 6B, the qualifier EICs are scaled to match height, but the ratios between the qualifier ion and base peak are indicated in the upper left of the figure and the accuracy of the ratio to the quantitative method reference ratios. The reference ratio of 93 to 77 m/z for this quantitative method is 31; Figure 6B ratio of 93/77 was 35.1, which is within 20% of the expected ratio, and significant conversion of nitrobenzene to aniline was not observed. The retention of nitrobenzene and avoidance of hydrogenation is also shown in the raw spectrum of Figure 6C, where 93 m/z is not taller than 123 nor 77 m/z.

---

**Figure 5.** Nitrobenzene linear range (0.1 to 100 µg/mL) collected on a GC/MS system in (A) He and in (B) H₂ carrier gas with the Agilent HydroInert source.
The large EPA method 8270 mixture of compounds was also diluted to a concentration of 15 µg/mL to act as a calibration verification standard, since 15 µg/mL was not a calibration point. To test the repeatability of the HydroInert source in GC/MS with H₂ carrier gas, the standard was sandwich injected with 1 µL of a composite soil matrix to simulate a spiked matrix sample. This injection was repeated nine times. Table 7 contains the following data for each compound: average calculated concentration of the nine replicates of 15 µg/mL calibration verification in soil matrix and the %RSD for the nine replicate injections in soil matrix. Looking at the average calculated concentration of the 15 µg/mL sample in matrix, only two compounds are identified outside of the ±20% range for a calibration verification, which are both reported as lower concentrations: 5-nitro-o-toluidine and dibutyl phthalate. The two compounds are within 25% of the 15 µg/mL spike value, and the matrix may be causing a small amount of signal suppression. The %RSD for the replicate injections in soil matrix are all below 7% RSD, indicating that the method is robust and consistent.

<table>
<thead>
<tr>
<th>Name</th>
<th>Average Calculated Concentration in Matrix of 15 µg/mL Spike</th>
<th>%RSD of Nine Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-Nitrosodimethylamine</td>
<td>15.6</td>
<td>2.21%</td>
</tr>
<tr>
<td>Pyridine</td>
<td>17.6</td>
<td>3.16%</td>
</tr>
<tr>
<td>2-Picoline</td>
<td>14.9</td>
<td>1.35%</td>
</tr>
<tr>
<td>N-Nitroso-N-methylethylamine</td>
<td>15.8</td>
<td>1.26%</td>
</tr>
<tr>
<td>Methyl methanesulfonate</td>
<td>15.0</td>
<td>2.05%</td>
</tr>
<tr>
<td>2-Fluorophenol</td>
<td>15.9</td>
<td>1.82%</td>
</tr>
<tr>
<td>N-Nitroso-N-diethylamine</td>
<td>15.6</td>
<td>2.53%</td>
</tr>
<tr>
<td>Ethyl methanesulfonate</td>
<td>15.0</td>
<td>2.14%</td>
</tr>
<tr>
<td>Phenol-d₆</td>
<td>15.6</td>
<td>1.91%</td>
</tr>
<tr>
<td>Phenol</td>
<td>15.1</td>
<td>1.00%</td>
</tr>
<tr>
<td>Aniline</td>
<td>15.7</td>
<td>1.62%</td>
</tr>
<tr>
<td>Bis(2-chloroethyl) ether</td>
<td>15.0</td>
<td>1.49%</td>
</tr>
<tr>
<td>2-Chlorophenol</td>
<td>15.1</td>
<td>1.54%</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>15.0</td>
<td>1.11%</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>14.4</td>
<td>1.31%</td>
</tr>
<tr>
<td>Benzyl alcohol</td>
<td>15.2</td>
<td>2.39%</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>15.3</td>
<td>1.86%</td>
</tr>
<tr>
<td>2-Methylphenol (o-cresol)</td>
<td>15.6</td>
<td>1.43%</td>
</tr>
<tr>
<td>Bis(2-chloro-1-methylethyl) ether</td>
<td>14.4</td>
<td>1.91%</td>
</tr>
<tr>
<td>1-Nitrosopyrrolidine</td>
<td>14.9</td>
<td>2.73%</td>
</tr>
<tr>
<td>p-Cresol</td>
<td>14.2</td>
<td>1.08%</td>
</tr>
<tr>
<td>N-Nitrosodi-n-propylamine</td>
<td>14.6</td>
<td>2.71%</td>
</tr>
<tr>
<td>Acetophenone</td>
<td>14.7</td>
<td>2.35%</td>
</tr>
<tr>
<td>Name</td>
<td>Average Calculated Concentration in Matrix of 15 µg/mL Spike</td>
<td>%RSD of Nine Replicates</td>
</tr>
<tr>
<td>-------------------------------------------</td>
<td>-------------------------------------------------------------</td>
<td>-------------------------</td>
</tr>
<tr>
<td>4-Nitrosomorpholine</td>
<td>14.4</td>
<td>2.40%</td>
</tr>
<tr>
<td>o-Toluidine</td>
<td>14.4</td>
<td>1.26%</td>
</tr>
<tr>
<td>Hexachloroethane</td>
<td>15.0</td>
<td>4.80%</td>
</tr>
<tr>
<td>Nitrobenzene-d₈</td>
<td>15.0</td>
<td>1.53%</td>
</tr>
<tr>
<td>Nitrobenzene</td>
<td>14.8</td>
<td>1.87%</td>
</tr>
<tr>
<td>Nitrosopiperidine</td>
<td>14.5</td>
<td>2.32%</td>
</tr>
<tr>
<td>Isophorone</td>
<td>14.7</td>
<td>2.52%</td>
</tr>
<tr>
<td>2-Nitrophenol</td>
<td>15.4</td>
<td>3.43%</td>
</tr>
<tr>
<td>2,4-Dimethylphenol</td>
<td>14.3</td>
<td>1.79%</td>
</tr>
<tr>
<td>Benzoic acid</td>
<td>14.3</td>
<td>6.81%</td>
</tr>
<tr>
<td>Bis(2-chloroethoxy)methane</td>
<td>14.8</td>
<td>1.73%</td>
</tr>
<tr>
<td>2,4-Dichlorophenol</td>
<td>14.9</td>
<td>1.64%</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>15.0</td>
<td>1.31%</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>14.4</td>
<td>1.50%</td>
</tr>
<tr>
<td>a,a-Dimethylenaphthalamine</td>
<td>14.0</td>
<td>2.25%</td>
</tr>
<tr>
<td>4-Chloroaniline</td>
<td>15.5</td>
<td>1.80%</td>
</tr>
<tr>
<td>2,6-Dichlorophenol</td>
<td>17.9</td>
<td>1.34%</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>13.5</td>
<td>3.66%</td>
</tr>
<tr>
<td>N-Nitrosobutylamine</td>
<td>14.2</td>
<td>2.45%</td>
</tr>
<tr>
<td>4-Chloro-3-methylphenol</td>
<td>15.1</td>
<td>2.29%</td>
</tr>
<tr>
<td>2-Methylphenalene</td>
<td>14.7</td>
<td>1.59%</td>
</tr>
<tr>
<td>Hexachlorocyclopentadiene</td>
<td>12.6</td>
<td>3.44%</td>
</tr>
<tr>
<td>1,2,4,5-Tetrachlorobenzene</td>
<td>14.9</td>
<td>2.77%</td>
</tr>
<tr>
<td>2,4,6-Trichlorophenol</td>
<td>15.3</td>
<td>1.92%</td>
</tr>
<tr>
<td>2,4,5-Trichlorophenol</td>
<td>15.3</td>
<td>1.91%</td>
</tr>
<tr>
<td>2-Fluorobiphenyl</td>
<td>15.5</td>
<td>1.47%</td>
</tr>
<tr>
<td>1-Chloronaphthalene</td>
<td>14.9</td>
<td>1.65%</td>
</tr>
<tr>
<td>2-Chloronaphthalene</td>
<td>15.3</td>
<td>1.64%</td>
</tr>
<tr>
<td>2-Nitroaniline</td>
<td>15.4</td>
<td>1.75%</td>
</tr>
<tr>
<td>Dimethyl phthalate</td>
<td>15.8</td>
<td>1.42%</td>
</tr>
<tr>
<td>2,6-Dinitrotoluene</td>
<td>13.1</td>
<td>3.81%</td>
</tr>
<tr>
<td>Acenaphthyline</td>
<td>15.0</td>
<td>1.03%</td>
</tr>
<tr>
<td>m-Nitroaniline</td>
<td>12.4</td>
<td>2.93%</td>
</tr>
<tr>
<td>Acenaphthene</td>
<td>14.5</td>
<td>1.52%</td>
</tr>
<tr>
<td>2,4-Dinitrophenol</td>
<td>12.3</td>
<td>5.97%</td>
</tr>
<tr>
<td>4-Nitrophenol</td>
<td>12.8</td>
<td>2.57%</td>
</tr>
<tr>
<td>Pentachlorobenzene</td>
<td>16.2</td>
<td>1.84%</td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>15.6</td>
<td>2.45%</td>
</tr>
<tr>
<td>Dibenzo furan</td>
<td>14.9</td>
<td>1.23%</td>
</tr>
<tr>
<td>1-Naphthylamine</td>
<td>14.1</td>
<td>1.28%</td>
</tr>
<tr>
<td>2,3,4,6-Tetrachlorophenol</td>
<td>12.7</td>
<td>3.86%</td>
</tr>
<tr>
<td>2-Naphthylamine</td>
<td>14.7</td>
<td>1.26%</td>
</tr>
<tr>
<td>Diethy1 phthalate</td>
<td>14.4</td>
<td>2.21%</td>
</tr>
<tr>
<td>Thionazin</td>
<td>14.0</td>
<td>2.99%</td>
</tr>
<tr>
<td>Fluorene</td>
<td>14.2</td>
<td>1.72%</td>
</tr>
<tr>
<td>4-Chlorophenyl phenyl ether</td>
<td>14.4</td>
<td>2.41%</td>
</tr>
<tr>
<td>5-Nitro-o-toluidine</td>
<td>11.4</td>
<td>4.16%</td>
</tr>
<tr>
<td>4-Nitroaniline</td>
<td>14.9</td>
<td>3.37%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Name</th>
<th>Average Calculated Concentration in Matrix of 15 µg/mL Spike</th>
<th>%RSD of Nine Replicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-Methyl, 4,6-dinitrophenol</td>
<td>13.6</td>
<td>2.93%</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>15.2</td>
<td>0.66%</td>
</tr>
<tr>
<td>Azobenzene</td>
<td>14.8</td>
<td>2.76%</td>
</tr>
<tr>
<td>2,4,6-Tribromophenol</td>
<td>15.5</td>
<td>3.74%</td>
</tr>
<tr>
<td>Sulfotep</td>
<td>13.1</td>
<td>4.28%</td>
</tr>
<tr>
<td>Diallate I</td>
<td>15.6</td>
<td>3.38%</td>
</tr>
<tr>
<td>Phorate</td>
<td>14.9</td>
<td>2.14%</td>
</tr>
<tr>
<td>Phencacetin</td>
<td>16.1</td>
<td>2.66%</td>
</tr>
<tr>
<td>4-Bromophenyl phenol ether</td>
<td>14.8</td>
<td>2.08%</td>
</tr>
<tr>
<td>Diallate II</td>
<td>14.9</td>
<td>3.70%</td>
</tr>
<tr>
<td>Hexachlorobenzene</td>
<td>16.9</td>
<td>2.73%</td>
</tr>
<tr>
<td>Dimethoate</td>
<td>12.7</td>
<td>2.42%</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>13.4</td>
<td>4.84%</td>
</tr>
<tr>
<td>4-Aminobiphenyl</td>
<td>16.0</td>
<td>2.40%</td>
</tr>
<tr>
<td>Pentachloronitrobenzene</td>
<td>16.7</td>
<td>6.40%</td>
</tr>
<tr>
<td>Propyzamide</td>
<td>15.2</td>
<td>2.86%</td>
</tr>
<tr>
<td>Dinoeb</td>
<td>13.0</td>
<td>3.24%</td>
</tr>
<tr>
<td>Disulfoton</td>
<td>14.2</td>
<td>4.39%</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>14.5</td>
<td>0.88%</td>
</tr>
<tr>
<td>Anthracene</td>
<td>15.0</td>
<td>2.01%</td>
</tr>
<tr>
<td>Methyl parathion</td>
<td>15.5</td>
<td>3.70%</td>
</tr>
<tr>
<td>Dibutyl phthalate</td>
<td>11.5</td>
<td>3.70%</td>
</tr>
<tr>
<td>Parathion</td>
<td>15.7</td>
<td>2.21%</td>
</tr>
<tr>
<td>4-Nitroquinoline-1-oxide</td>
<td>16.9</td>
<td>2.04%</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>15.0</td>
<td>0.95%</td>
</tr>
<tr>
<td>Benzidine</td>
<td>14.0</td>
<td>2.76%</td>
</tr>
<tr>
<td>Aramite</td>
<td>13.9</td>
<td>3.71%</td>
</tr>
<tr>
<td>Aramite II</td>
<td>13.3</td>
<td>3.59%</td>
</tr>
<tr>
<td>Pyrene</td>
<td>14.8</td>
<td>1.62%</td>
</tr>
<tr>
<td>p-Terphenyl-d₈</td>
<td>15.3</td>
<td>1.98%</td>
</tr>
<tr>
<td>p-(Dimethylamino)azobenzene</td>
<td>14.0</td>
<td>2.05%</td>
</tr>
<tr>
<td>Chlorbenzilate</td>
<td>14.9</td>
<td>1.92%</td>
</tr>
<tr>
<td>3,3'-Dimethylbenzidine</td>
<td>14.6</td>
<td>2.11%</td>
</tr>
<tr>
<td>Benzyl butyl phthalate</td>
<td>13.8</td>
<td>2.51%</td>
</tr>
<tr>
<td>3,3'-Dichlorobenzidine</td>
<td>15.8</td>
<td>1.90%</td>
</tr>
<tr>
<td>Benz[a]anthracene</td>
<td>13.7</td>
<td>0.98%</td>
</tr>
<tr>
<td>Chrysene</td>
<td>14.5</td>
<td>1.31%</td>
</tr>
<tr>
<td>Bis(2-ethylhexyi) phthalate</td>
<td>15.2</td>
<td>1.89%</td>
</tr>
<tr>
<td>Di-n-octyl phthalate</td>
<td>14.3</td>
<td>1.30%</td>
</tr>
<tr>
<td>7,12-Dimethylbenz[a]anthracene</td>
<td>12.2</td>
<td>1.40%</td>
</tr>
<tr>
<td>Benzo[b]fluoranthene</td>
<td>14.7</td>
<td>1.50%</td>
</tr>
<tr>
<td>Benzo[k]fluoranthene</td>
<td>15.4</td>
<td>2.94%</td>
</tr>
<tr>
<td>Benzo[a]pyrene</td>
<td>15.4</td>
<td>2.07%</td>
</tr>
<tr>
<td>3-Methylcholanthrene</td>
<td>14.6</td>
<td>2.77%</td>
</tr>
<tr>
<td>Diben[z,a]acidine</td>
<td>13.0</td>
<td>1.58%</td>
</tr>
<tr>
<td>Indeno(1,2,3-cd)pyrene</td>
<td>15.8</td>
<td>1.44%</td>
</tr>
<tr>
<td>Diben[z,a]anthracene</td>
<td>15.5</td>
<td>2.18%</td>
</tr>
<tr>
<td>Benzo[ghi]perylene</td>
<td>15.5</td>
<td>1.56%</td>
</tr>
</tbody>
</table>
Conclusion

A method for testing SVOCs using H₂ carrier gas and the Agilent HydroInert source, which prevents hydrogenation and dechlorination of target analytes, has been developed for the Agilent 5977B Inert Plus GC/MSD. Method criteria for EPA method 8270D/E are met for the GC/MS tuning mixture, DFTPP tuning criteria, and initial calibration over the normal working range of 0.1 to 100 µg/mL in a single 12-minute run, with 15 compounds of the 119 tested compounds requiring curve fits. Retention of mass spectral fidelity is a breakthrough for the use of H₂ carrier gas with GC/MS systems, especially for environmental analyses, such as EPA method 8270.

References


2. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); Method 8270E, United Stated Environmental Protection Agency, Revision 4, June 2018.
