REPORT

Final Report

Spacelab Charcoal Analyses - Phase II

To

Ion Electronics

6767 Madison Pike

Huntsville, AL 35806

January 20, 1995
Final Report
Spacelab Charcoal Analyses - Phase II

to

ION Electronics
Contract No. NAS8-38250-14

January 20, 1995

by

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Introduction

This report describes analytical methods and results obtained from chemical analysis of 31 charcoal samples in five sets. Each set was obtained from a single scrubber used to filter ambient air on board a Spacelab mission. Analysis of the charcoal samples was conducted by thermal desorption followed by gas chromatography/mass spectrometry (GC/MS). All samples were analyzed using identical methods. Two of the mission sample sets (IML, USML) were analyzed previously by Battelle (Contract No. 38250-06) using a similar method, however that method was unable to detect very volatile and/or very polar compounds. The method used for these analyses was able to detect compounds independent of their polarity or volatility.

In addition to the charcoal samples, Battelle was requested and authorized under Contract Modification No. 5 to conduct analyses of three ECLSS water samples received on December 15, 1994 specifically for trimethylamine.

Because of the number of samples and compounds identified, results from the charcoal analyses are provided in Appendix A together with supporting GC/MS total ion chromatograms. Results from Trimethylamine analyses on the three ECLSS water samples are provided in the Results and Discussion section of this report.

Technical Approach

Charcoal Samples

Table 1 identifies the charcoal samples analyzed under this contract. As indicated earlier, charcoal from the IML and USML missions was analyzed previously by Battelle and was maintained in glass jars at room temperature from date of receipt. The charcoal type shown in Table 1 was not indicated on the samples but was presumed by Battelle staff based on the sample numbers, discussions with ION personnel and prior documentation received on
on the sample numbers, discussions with ION personnel and prior documentation received on charcoal samples. Also included in these analyses was a sample of clean activated charcoal provided by Battelle that was stored with the IML and USML samples from the date of receipt. This "method blank" material is not identified in Table 1 but is represented in subsequent results tables and in the data of Appendix A.

Table 1. Identification of Charcoal Samples

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Date Received</th>
<th>Presumed Charcoal Type</th>
<th>Estimated Total Weight, g.</th>
</tr>
</thead>
<tbody>
<tr>
<td>IML-1 #1</td>
<td>3/1/93</td>
<td>2% Platinum</td>
<td>213</td>
</tr>
<tr>
<td>IML-1 #2</td>
<td>3/1/93</td>
<td>Acid Washed</td>
<td>1523</td>
</tr>
<tr>
<td>IML-1 #3</td>
<td>3/1/93</td>
<td>Activated Carbon</td>
<td>285</td>
</tr>
<tr>
<td>IML-1 #4</td>
<td>3/1/93</td>
<td>Activated Carbon</td>
<td>499</td>
</tr>
<tr>
<td>IML-1 #5</td>
<td>3/1/93</td>
<td>Activated Carbon</td>
<td>493</td>
</tr>
<tr>
<td>USML-1 #1</td>
<td>4/15/93</td>
<td>2% Platinum</td>
<td>348</td>
</tr>
<tr>
<td>USML-1 #2</td>
<td>4/15/93</td>
<td>Acid Washed</td>
<td>1767</td>
</tr>
<tr>
<td>USML-1 #3</td>
<td>4/15/93</td>
<td>Activated Carbon</td>
<td>453</td>
</tr>
<tr>
<td>USML-1 #4</td>
<td>4/15/93</td>
<td>Activated Carbon</td>
<td>453</td>
</tr>
<tr>
<td>USML-1 #5</td>
<td>4/15/93</td>
<td>Activated Carbon</td>
<td>453</td>
</tr>
<tr>
<td>SLJ-1 #1</td>
<td>5/5/94</td>
<td>2% Platinum</td>
<td>362</td>
</tr>
<tr>
<td>SLJ-1 #2</td>
<td>5/5/94</td>
<td>Acid Washed</td>
<td>1750</td>
</tr>
<tr>
<td>SLJ-1 #3</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>506</td>
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<td>SLJ-1 #4</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>503</td>
</tr>
<tr>
<td>SLJ-1 #5</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>501</td>
</tr>
<tr>
<td>SLD-2 #1</td>
<td>5/5/94</td>
<td>2% Platinum</td>
<td>396</td>
</tr>
<tr>
<td>SLD-2 #2</td>
<td>5/5/94</td>
<td>Acid Washed</td>
<td>1826</td>
</tr>
<tr>
<td>SLD-2 #3</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>456</td>
</tr>
<tr>
<td>SLD-2 #4</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>472</td>
</tr>
<tr>
<td>SLD-2 #5</td>
<td>5/5/94</td>
<td>Activated Carbon</td>
<td>453</td>
</tr>
<tr>
<td>P1-STS-58 (SLS-02)</td>
<td>5/5/94</td>
<td>2% Platinum</td>
<td>114</td>
</tr>
<tr>
<td>P2-STS-58</td>
<td>5/5/94</td>
<td>2% Platinum</td>
<td>113</td>
</tr>
</tbody>
</table>
The analysis method used for these samples was thermal desorption coupled on line to a gas chromatograph/mass spectrometer system. The specific instrumentation and operating conditions are summarized in Table 2. Unlike previous analyses, a dry purge was not conducted prior to thermal desorption, nor was a Nafton in-line dryer used to remove water vapor desorbed from the charcoal. The impact of water vapor on the analysis was significant but was minimized to the extent possible by reducing the quantity of sample actually analyzed.

To improve quantitative accuracy, standard mixtures were prepared containing known concentrations of selected compounds that were among the most abundant in previous scrubber charcoal analyses including those conducted at Battelle. This standard mixture was analyzed for purposes of calibration by direct splitless injection into the GC/MS system or for purposes of estimating recovery by spiking onto a charcoal sample followed by thermal desorption GC/MS. Initial trials using solvent based spikes onto previously desorbed charcoal samples were not successful because the solvent created excessive peak broadening rendering identification and quantification difficult. To overcome this problem, a pressurized gas standard was prepared whose composition is shown in Table 3. This gas standard was
used both for direct injection to single point calibrate the GC/MS system, and for loading previously desorbed "blank" charcoal samples for purpose of estimating recovery.

Table 2. Instrumentation and Analysis Parameters

<table>
<thead>
<tr>
<th>Charcoal Analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Thermal Desorber:</strong></td>
</tr>
<tr>
<td><strong>Dry Purge:</strong></td>
</tr>
<tr>
<td><strong>Desorb Temperature:</strong></td>
</tr>
<tr>
<td><strong>Desorb Flow:</strong></td>
</tr>
<tr>
<td><strong>Desorb Time:</strong></td>
</tr>
<tr>
<td><strong>GC/MS System:</strong></td>
</tr>
<tr>
<td><strong>GC Column:</strong></td>
</tr>
<tr>
<td><strong>Initial Temperature:</strong></td>
</tr>
<tr>
<td><strong>Temperature Program:</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td><strong>Ionization:</strong></td>
</tr>
<tr>
<td><strong>MS Scan Range:</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Water Analyses (Trimethylamine)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Instrument:</strong></td>
</tr>
<tr>
<td><strong>Sample Introduction:</strong></td>
</tr>
<tr>
<td><strong>Mobile Phase:</strong></td>
</tr>
<tr>
<td><strong>LC/MS Interface:</strong></td>
</tr>
<tr>
<td><strong>Ionization:</strong></td>
</tr>
<tr>
<td><strong>Operating Mode:</strong></td>
</tr>
<tr>
<td><strong>Parent Ion:</strong></td>
</tr>
<tr>
<td><strong>Daughter Ions:</strong></td>
</tr>
<tr>
<td><strong>Dwell Time:</strong></td>
</tr>
<tr>
<td><strong>Collision Energy (E_{Lab}):</strong></td>
</tr>
<tr>
<td><strong>Target Thickness:</strong></td>
</tr>
</tbody>
</table>
Table 3. Composition of Standards Gas Cylinder

<table>
<thead>
<tr>
<th>Compound</th>
<th>Concentration ng/mL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>30</td>
</tr>
<tr>
<td>Isopropanol</td>
<td>30</td>
</tr>
<tr>
<td>Acetone</td>
<td>30</td>
</tr>
<tr>
<td>2-Ethoxyethyl acetate</td>
<td>10</td>
</tr>
<tr>
<td>Benzene</td>
<td>10</td>
</tr>
<tr>
<td>Toluene</td>
<td>10</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>10</td>
</tr>
<tr>
<td>Cyclohexane</td>
<td>10</td>
</tr>
<tr>
<td>Dibromochloromethane</td>
<td>10</td>
</tr>
<tr>
<td>Trichlorofluoromethane (Freon 11)</td>
<td>10</td>
</tr>
<tr>
<td>1,1,2-Trichloro-1,2,2-trifluoro methane (Freon 113)</td>
<td>10</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>10</td>
</tr>
<tr>
<td>Butanol</td>
<td>10</td>
</tr>
</tbody>
</table>

Water Samples

Trimethylamine (TMA) analyses were conducted by direct aqueous flow injection combined with tandem mass spectrometry. No sample preparation was necessary. The instrument and configuration are summarized in Table 2. The tandem mass spectrometer is a PE-Sciex API III configured with a corona discharge atmospheric pressure chemical ionization (APCI) ion source and a proprietary Battelle interface which vaporizes the flow injected carrier (mobile phase) prior to its entry into the APCI ion source. Water vapor serves as the predominant reagent gas resulting in protonation of TMA (m/z 60). Ions of
this mass are isolated by the first mass analyzer and subjected to collisional activated
dissociation (CAD) using argon as a low pressure inert target. Two characteristic daughter
ions (fragment ions) are monitored for TMA (m/z 44, 45). The ratio of the daughter ions
serves as a highly selective qualitative feature while the daughter ion response serves as a
means for quantification.

Results and Discussion

Charcoal Samples

Initial testing was concerned with quantifying our ability to recover the standard
compounds in the cylinder gas standard from the charcoal samples. One sample representing
each type of charcoal was thermally desorbed for 20 min., allowed to cool, then spiked with
100 mL (measured electronically as standard cm³) then analyzed under the conditions
anticipated for sample analysis. A backup trap was attached to the exit of the "sample" to
detect any breakthrough of analytes from the carbon sample. No target analytes were found
in any of the backup traps. Results from these analyses of the spiked charcoal samples are
summarized in Table 4. Percent recovery for each analyte was based upon the total ion
current in the background subtracted mass spectrum for the spiked charcoal sample divided
by an equivalent total ion current value obtained by direct injection into the GC of the
cylinder gas standard. Low recovery was evident in some cases. As an example, ethanol
and isopropanol were poorly recovered from the untreated and platinum charcoal samples.
These results suggest that longer desorption times may be a factor to more efficiently recover
these compounds, however on separate trials with unconditioned samples we observed that
longer desorption times contributed to degradation of chromatographic resolution presumably
as a result of additional water introduced to the GC column.
Table 4. Recovery of Standards Spiked onto Different Charcoal Types

<table>
<thead>
<tr>
<th>Compound</th>
<th>Spike Loading, micrograms</th>
<th>% Recovery SLD2 #5 (Untreated)</th>
<th>% Recovery USML1 #2 (Acid Washed)</th>
<th>% Recovery USML1 #1 (Platinum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>3.0</td>
<td>3.3</td>
<td>19.0</td>
<td>2.4</td>
</tr>
<tr>
<td>Isopropanol</td>
<td>3.0</td>
<td>4.7</td>
<td>18.2</td>
<td>5.9</td>
</tr>
<tr>
<td>Acetone</td>
<td>3.0</td>
<td>12.4</td>
<td>37.8</td>
<td>35.0</td>
</tr>
<tr>
<td>2-Ethoxyethyl acetate</td>
<td>1.0</td>
<td>6.6</td>
<td>67.2</td>
<td>55.8</td>
</tr>
<tr>
<td>Benzene + Cyclohexane</td>
<td>1.0</td>
<td>25.3</td>
<td>54.2</td>
<td>52.5</td>
</tr>
<tr>
<td>Toluene</td>
<td>1.0</td>
<td>31.5</td>
<td>47.6</td>
<td>33.1</td>
</tr>
<tr>
<td>o-Xylene</td>
<td>1.0</td>
<td>27.6</td>
<td>35.9</td>
<td>45.2</td>
</tr>
<tr>
<td>Dibromochloromethane</td>
<td>1.0</td>
<td>2.5</td>
<td>30.7</td>
<td>15.2</td>
</tr>
<tr>
<td>Trichlorofluoromethane (Freon 11)</td>
<td>1.0</td>
<td>16.8</td>
<td>43.6</td>
<td>14.6</td>
</tr>
<tr>
<td>1,1,2-Trichloro-1,2,2-trifluoromethane (Freon 113)</td>
<td>1.0</td>
<td>21.2</td>
<td>53.7</td>
<td>26.1</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>1.0</td>
<td>15.8</td>
<td>51.9</td>
<td>27.6</td>
</tr>
<tr>
<td>Butanol</td>
<td>1.0</td>
<td>33.4</td>
<td>28.1</td>
<td>73.8</td>
</tr>
</tbody>
</table>

1. See text for details.

2. Benzene and Cyclohexane were not chromatographically resolved under the conditions of analysis in either the spiked charcoal samples or the standard from which recovery is based.

Gravimetric results for each of the charcoal samples analyzed are summarized in Table 5. Included in Table 5 are the original weight of sample desorbed, as well as percent weight loss presumably due to water vapor. Except for IML and USML charcoal samples, all
samples within a mission set show comparable water content with a slight positive bias in the acid washed and platinum loaded charcoals. This trend is not observed in the older IML and USML samples which, despite precautions for maintaining a hermetic seal, may have gained or lost water during storage. The two "Blank" samples shown in Table 5 were each aliquots of a single laboratory charcoal sample stored with IML and USML samples. The relatively low water content of this charcoal combined with good agreement in these replicate analyses indicate that the water content of the samples is substantial and that these measurements are reproducible.

Table 5. Weight of Sample Analyzed and Estimated Percent Water.

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Weight Desorbed, g.</th>
<th>Weight Loss, g.</th>
<th>Weight Loss, Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>IML-1 #1</td>
<td>0.1432</td>
<td>0.0254</td>
<td>17.7</td>
</tr>
<tr>
<td>IML-1 #2</td>
<td>0.1063</td>
<td>0.0197</td>
<td>18.5</td>
</tr>
<tr>
<td>IML-1 #3</td>
<td>0.1133</td>
<td>0.0494</td>
<td>41.4</td>
</tr>
<tr>
<td>IML-1 #4</td>
<td>0.1158</td>
<td>0.0097</td>
<td>8.4</td>
</tr>
<tr>
<td>IML-1 #5</td>
<td>0.1611</td>
<td>0.0954</td>
<td>59.2</td>
</tr>
<tr>
<td>USML-1 #1</td>
<td>0.1377</td>
<td>0.0253</td>
<td>18.4</td>
</tr>
<tr>
<td>USML-1 #2</td>
<td>0.2263</td>
<td>0.0374</td>
<td>16.5</td>
</tr>
<tr>
<td>USML-1 #3</td>
<td>0.1655</td>
<td>0.0214</td>
<td>12.9</td>
</tr>
<tr>
<td>USML-1 #4</td>
<td>0.1769</td>
<td>0.0154</td>
<td>8.7</td>
</tr>
<tr>
<td>USML-1 #5</td>
<td>0.1142</td>
<td>0.0109</td>
<td>9.5</td>
</tr>
<tr>
<td>SLJ-1 #1</td>
<td>0.1219</td>
<td>0.0236</td>
<td>19.4</td>
</tr>
<tr>
<td>SLJ-1 #2</td>
<td>0.1823</td>
<td>0.0359</td>
<td>19.7</td>
</tr>
<tr>
<td>SLJ-1 #3</td>
<td>0.1357</td>
<td>0.0152</td>
<td>11.2</td>
</tr>
<tr>
<td>SLJ-1 #4</td>
<td>0.1263</td>
<td>0.0134</td>
<td>10.6</td>
</tr>
<tr>
<td>SLJ-1 #5</td>
<td>0.1171</td>
<td>0.0132</td>
<td>11.3</td>
</tr>
<tr>
<td>SLD-2 #1</td>
<td>0.1076</td>
<td>0.0259</td>
<td>24.1</td>
</tr>
<tr>
<td>Sample Identification</td>
<td>Weight Desorbed, g.</td>
<td>Weight Loss, g.</td>
<td>Weight Loss, Percent¹</td>
</tr>
<tr>
<td>-----------------------</td>
<td>---------------------</td>
<td>----------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>SLD-2 #2</td>
<td>0.1095</td>
<td>0.0244</td>
<td>22.3</td>
</tr>
<tr>
<td>SLD-2 #3</td>
<td>0.1003</td>
<td>0.0208</td>
<td>20.7</td>
</tr>
<tr>
<td>SLD-2 #4</td>
<td>0.1242</td>
<td>0.0282</td>
<td>22.7</td>
</tr>
<tr>
<td>SLD-2 #5</td>
<td>0.1154</td>
<td>0.0246</td>
<td>21.3</td>
</tr>
<tr>
<td>P1-STS-58 (SLS-02)</td>
<td>0.1194</td>
<td>0.0313</td>
<td>26.2</td>
</tr>
<tr>
<td>P2-STS-58</td>
<td>0.1426</td>
<td>0.0356</td>
<td>25.0</td>
</tr>
<tr>
<td>P3-STS-58</td>
<td>0.1694</td>
<td>0.0414</td>
<td>24.4</td>
</tr>
<tr>
<td>AW1-STS-58</td>
<td>0.1772</td>
<td>0.0402</td>
<td>22.7</td>
</tr>
<tr>
<td>AW2-STS-58</td>
<td>0.1509</td>
<td>0.0355</td>
<td>23.5</td>
</tr>
<tr>
<td>AW3-STS-58</td>
<td>0.1280</td>
<td>0.0277</td>
<td>21.6</td>
</tr>
<tr>
<td>ACT1-STS-58</td>
<td>0.1666</td>
<td>0.0375</td>
<td>22.5</td>
</tr>
<tr>
<td>ACT2-STS-58</td>
<td>0.1816</td>
<td>0.0421</td>
<td>23.2</td>
</tr>
<tr>
<td>ACT3-STS-58</td>
<td>0.1262</td>
<td>0.0303</td>
<td>24.0</td>
</tr>
<tr>
<td>ACT4-STS-58</td>
<td>0.1112</td>
<td>0.0245</td>
<td>22.0</td>
</tr>
<tr>
<td>ACT5-STS-58</td>
<td>0.1037</td>
<td>0.0228</td>
<td>22.0</td>
</tr>
<tr>
<td>BLANK #1</td>
<td>0.1175</td>
<td>0.0100</td>
<td>9.4</td>
</tr>
<tr>
<td>BLANK #2</td>
<td>0.1058</td>
<td>0.0096</td>
<td>9.1</td>
</tr>
</tbody>
</table>

¹ Measured weight loss is presumed to be almost entirely from adsorbed water.

Chromatographic and tabular results are provided in Appendix A for one of the Blank charcoal analyses and the three spiked charcoal samples described above. Note that the data in Appendix A is organized so that tabular and chromatographic data for the same sample are on facing pages. Following these data are tabular and chromatographic results from one analysis of each unspiked sample. All of the chromatographic data in Appendix A were
acquired under the same thermal desorption and chromatographic conditions. Additional analyses, for example split injections from the charcoal to the chromatographic column, were carried out in some cases for diagnostic purposes to better interpret (qualitatively) chromatographic or mass spectral results. Sample analysis results provided in Appendix A are represented as the identified compound, an RIC value corresponding to the total ion current response, the measured concentration for that compound, and in those cases where the identified compound was one of those in our gas standard, a corrected concentration. The corrected concentration was calculated as the measured concentration divided by the fractional recovery of that compound from the same type of charcoal.

Sample #5 contained the most compounds and highest concentrations in both the IML and USML sample sets. These results are consistent with their presumed most forward location in the scrubber. A decrease is observed in the methylene chloride concentration with decreasing sample number in both sample sets, consistent with expectation. The results obtained from these analyses identify some of the same compounds but at approximately 10 fold lower concentration. Some of the compounds identified, for example acetone in USML #5 were not present in subsequent samples. Many of the compounds identified from re-analyses of the IML and USML sample sets were previously identified in these samples, but now at significantly lower concentration. This difference may represent loss of analyte because of sample age or may be the result of excess water co-introduced into the chromatographic system.

Results from the SLD and SLJ sample sets show similar trends in that Sample #5 was in each case the most heavily loaded with organic compounds. The lowest concentrations were observed in the #1 samples however a declining trend in concentration from the first to the last charcoal sample in the collection sequence is not clear. Some of the compounds, for example methylene chloride in the SLD samples, show an erratic distribution that does not appear to be correlated to their position in the scrubber. Methylene chloride may be an artifact of sample recovery or storage prior to delivery at Battelle. This compound is not present in Battelle’s method blank charcoal that was stored with the samples. The high
concentration of isopropanol in SLJ-1 #5 may be a contamination artifact because it would be expected to be present in succeeding samples of that set. The STS-58 samples revealed high concentrations of organic compounds in the activated charcoal (AC5 through AC1) with an apparent maximum concentration in AC3. As expected, lesser concentrations were observed in the acid washed charcoal samples (AW3 through AW1) and even lower concentrations in the Platinum loaded charcoals (P3 through P1).

Water Samples

Figure 1 provides the actual signal response obtained for each of the reaction monitoring channels (60/44 and 60/45) resulting from flow injection of a water blank, a 75 ng/mL standard, a 149 ng/mL standard and two injections of a 372 ng/mL standard. Both channels are recorded nearly simultaneously by alternating acquisition of the two specific daughter ions of TMA. The key elements of the TMA signature are the presence of the two daughter ions and the near equivalence of their relative abundance. The ion transition at the m/z 44 daughter ion (60/44) was used for quantification. The resulting TMA calibration curve is shown in Table 2. Results obtained from analysis of the three ECLSS water samples are summarized in Table 6. Our limit of detection for this compound in water is 75 ng/mL, and is supported by a calibration standard whose results are distinguishable from zero. Included in Table 6 are preliminary results from electrospray ionization trials that provided unconfirmed evidence of sodium cation and iodide anion in selected samples. One of the samples contained TMA at the limit of detection while another was approximately three times this concentration. The third sample provided a suggestive response but was below that of our lowest standard and is therefore not detected. Graphical results from analyses of these samples are shown in Figures 3 through 5. In each case, three injections were made corresponding to a water blank, the ECLSS sample diluted by a factor of 5, and finally the undiluted ECLSS sample. Injection of the diluted sample was a precaution to avoid potentially overloading the ion source. In all but the non-detect case, WRT-ST9-0108-0-CETA-127-T-MFS, a clear response is observed for TMA that is greater than the water
blank or the diluted sample, and with a daughter ion abundance ratio consistent with that from the calibration standards.

Table 6. Trimethylamine Analysis Results for Water Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Trimethylamine ng/mL (ppb)</th>
<th>Sodium Cation</th>
<th>Iodide Anion</th>
</tr>
</thead>
<tbody>
<tr>
<td>WRT-ST9-0107-1-CETA-127-T-MFS</td>
<td>75</td>
<td>+</td>
<td>nd</td>
</tr>
<tr>
<td>WRT-ST9-0106-0-CETA-127-T-MFS</td>
<td>240</td>
<td>++++</td>
<td>X</td>
</tr>
<tr>
<td>WRT-ST9-0108-0-CETA-127-T-MFS</td>
<td>nd</td>
<td>++++</td>
<td>XX</td>
</tr>
</tbody>
</table>

1. Samples were approximately 125 mL each, received on December 15, 1994

nd = not detected.

+ denotes incidental non-quantified observation of sodium cation, number of symbols approximately proportional to response.

X denotes incidental non-quantified observation of iodide anion, number of symbols approximately proportional to response.

In addition to flow injection MS/MS analyses for TMA, positive and negative ion electrospray mass spectra were acquired for each water sample. These results are shown in Figures 6 through 8 which correspond to the background corrected mass spectra from flow injection. An aliquot of each sample was diluted with an equal volume of methanol to reduce surface tension and improve the stability of the electrospray process. Each figure contains the positive ion and negative ion mass spectra for one sample. The spectra contain evidence of coincident corona discharge APCI ionization in addition to electrospray ionization resulting in several mass peaks due to protonated water and methanol clusters and other unidentified species. Because these samples were described as having unexpected conductivity, we have highlighted the presence of sodium cation at m/z 23 in the positive ion
spectra and iodide cation at m/z 127 in the negative ion spectra for two of the three water samples.

Conclusions and Recommendations

The results obtained from the charcoal analyses represent a range of compounds and in some cases unusually high concentrations of polar compounds such as ethanol and acetone. The use of GC/MS to analyze a range of compounds is widely utilized for environmental analyses, however this approach becomes complicated for charcoal analyses because of the unusually high water loading. The older IML and USML charcoal samples did not show consistent water loading nor did these samples reveal the same concentration of non-polar compounds as determined under the previous contract. The remaining three sample sets showed a variety of polar and non-polar organic compounds.

Analysis of the water samples for TMA was accomplished using flow injection APCI tandem mass spectrometry. A detection limit of 75 ng/mL was obtained which allowed detection of TMA in two of the three samples provided by ION Electronics. In addition to TMA results, positive and negative ion electrospray mass spectra revealed qualitative evidence for low concentrations of sodium cation (m/z 23) and, unexpectedly, iodide anion (m/z 127) in two of the three samples. If absolute quantitative information for sodium and/or iodide is desired, we recommend atomic emission (Na) and ion chromatography (I) as the analysis methods of choice. These analyses can be conducted at Battelle using remaining quantities of these samples.
Figure 1. Flow injection response at the two daughter ions of trimethylamine as a function of aqueous standard concentration.
Figure 2. Calibration curve for Trimethylamine from m/z 60/44 response.
Figure 3. Trimethylamine for diluted and undiluted WRT-ST9-0107-1-CETA-127-T-MFS.
Figure 5. Trimethylamine ion response to diluted and undiluted WRT-ST9-0108-0-CETA-127-T-MFS.
Figure 6. Positive ion (a) and negative ion (b) electrospray mass spectra from WRT-ST9-0107-1-CETA-127-T-MFS.
Appendix A

Results from Charcoal Analyses
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Known Conc (µg/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ethanol</td>
<td>35178</td>
<td>0.030</td>
</tr>
<tr>
<td>2</td>
<td>Trichlorofluoromethane</td>
<td>4160</td>
<td>0.010</td>
</tr>
<tr>
<td>3</td>
<td>Acetone</td>
<td>28438</td>
<td>0.030</td>
</tr>
<tr>
<td>4</td>
<td>IPA</td>
<td>4674</td>
<td>0.030</td>
</tr>
<tr>
<td>5</td>
<td>1,1,2-Trichloro-1,2,2-trifluoroethane</td>
<td>8992</td>
<td>0.010</td>
</tr>
<tr>
<td>6</td>
<td>1,1,1-Trichloroethane</td>
<td>2256</td>
<td>0.010</td>
</tr>
<tr>
<td>7</td>
<td>Butanol</td>
<td>22976</td>
<td>0.010</td>
</tr>
<tr>
<td>8</td>
<td>Benzene/Cyclohexane</td>
<td>64256</td>
<td>0.020</td>
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<tr>
<td>9</td>
<td>Toluene</td>
<td>55360</td>
<td>0.010</td>
</tr>
<tr>
<td>10</td>
<td>Dibromochloromethane</td>
<td>19488</td>
<td>0.010</td>
</tr>
<tr>
<td>11</td>
<td>2-Ethoxyethylacetate</td>
<td>21920</td>
<td>0.010</td>
</tr>
<tr>
<td>12</td>
<td>O-Xylene</td>
<td>46592</td>
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</table>

Gas Standard 198706 (1 mL injected)
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>μg in 100 mL</th>
<th>Measured Amount (μg)</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>Ethanol</td>
<td>18880</td>
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<tr>
<td>2</td>
<td>Trichlorofluoromethane</td>
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<tr>
<td>3</td>
<td>Acetone</td>
<td>441344</td>
<td>3.000</td>
<td>1.051</td>
</tr>
<tr>
<td>4</td>
<td>IPA</td>
<td>85248</td>
<td>3.000</td>
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<td>1,1,1-Trichloroethane</td>
<td>31049</td>
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<td>0.738</td>
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<td>0.152</td>
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<td>2-Ethoxyethylacetate</td>
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</tr>
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<td>RIC Value</td>
<td>µg in 100 mL</td>
<td>Measured Amount (µg)</td>
</tr>
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<td>Peak No.</td>
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<td>RIC Value</td>
<td>μg in 100 mL</td>
<td>Measured Amount (μg)</td>
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<td>-------------------------------------</td>
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<td>12</td>
<td>O-Xylene</td>
<td>832512</td>
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<td>Peak No.</td>
<td>Identification</td>
<td>RIC Value</td>
<td>Conc (µg/g)</td>
<td>Conc (µg/g)</td>
</tr>
<tr>
<td>---------</td>
<td>---------------------------</td>
<td>-----------</td>
<td>-------------</td>
<td>-------------</td>
</tr>
<tr>
<td>1</td>
<td>Bromotrifluoromethane</td>
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<td>0.621</td>
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</tr>
<tr>
<td>2</td>
<td>Methylene chloride</td>
<td>3672</td>
<td>0.064</td>
<td>b</td>
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</tbody>
</table>

*a Quantitation based on an actual standard compound.

*b Quantitation based on comparison to average of three halogenated standard compounds.

*c Quantitation based on comparison to average of four non-halogenated standard compounds.*
<table>
<thead>
<tr>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>Bromotrifluoromethane</td>
<td>22720</td>
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<td>Acetic acid</td>
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<tr>
<td>8</td>
<td>Toluene</td>
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</tr>
<tr>
<td>9</td>
<td>Caprolactam</td>
<td>12960</td>
<td>0.042</td>
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</table>

a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>14640</td>
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<td>BP 45, 61(50)</td>
<td>200192</td>
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<td>BP 31, 45(85), 61(50)</td>
<td>815104</td>
<td>4.13</td>
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</tr>
<tr>
<td>5</td>
<td>Xylene or Ethylbenzene</td>
<td>2616</td>
<td>0.013</td>
<td>c</td>
</tr>
<tr>
<td>6</td>
<td>Phenol</td>
<td>3028</td>
<td>0.015</td>
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<tr>
<td>7</td>
<td>Limonene</td>
<td>3508</td>
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<tr>
<td>8</td>
<td>Benzoic acid, methyl ester</td>
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<td>0.022</td>
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<td>9</td>
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<tr>
<td>10</td>
<td>Caprolactam</td>
<td>8992</td>
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</table>

a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
### IML-1 #4 Compounds Identified

<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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</thead>
<tbody>
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</tr>
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<td>2-Propenal</td>
<td>15456</td>
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<td>12.129</td>
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</tr>
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<td>5</td>
<td>Benzene + Cyclohexane</td>
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- **a** Quantitation based on an actual standard compound.
- **b** Quantitation based on comparison to average of three halogenated standard compounds.
- **c** Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (μg/g)</th>
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a  Quantitation based on an actual standard compound.
b  Quantitation based on comparison to average of three halogenated standard compounds.
c  Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g) Meas.</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g) Meas.</th>
<th>Conc (µg/g) Corr.</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
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<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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c Quantitation based on comparison to average of four non-halogenated standard compounds.
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<tr>
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<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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<th>RIC Value</th>
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<th>Conc (μg/g)</th>
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<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g) Measure</th>
<th>Conc (µg/g) Corr.</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of three non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of three non-halogenated standard compounds.
<table>
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a Quantitation based on an actual standard compound.
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c Quantitation based on comparison to average of three non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
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<th>Conc (µg/g) Meas.</th>
<th>Conc (µg/g) Corr.</th>
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<table>
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<table>
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<th>Peak No.</th>
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<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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<th>Conc (µg/g) Corr.</th>
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a Quantitation based on an actual standard compound.

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<thead>
<tr>
<th>Peak No.</th>
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<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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<td>Bromotrifluoromethane</td>
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<td>0.094</td>
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<td>0.007 a</td>
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<td>Xylene or Ethylbenzene</td>
<td>3356</td>
<td>0.007</td>
<td>c</td>
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</table>

- **a** Quantitation based on an actual standard compound.
- **b** Quantitation based on comparison to average of three halogenated standard compounds.
- **c** Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
<th>Identification</th>
<th>RIC Value</th>
<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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<tbody>
<tr>
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<td>Methylene chloride</td>
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<td>Benzaidehyde</td>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
<thead>
<tr>
<th>Peak No.</th>
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<th>RIC Value</th>
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<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
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* a Quantitation based on an actual standard compound.
* b Quantitation based on comparison to average of three halogenated standard compounds.
* c Quantitation based on comparison to average of four non-halogenated standard compounds.
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a. Quantitation based on an actual standard compound.
b. Quantitation based on comparison to average of three halogenated standard compounds.
c. Quantitation based on comparison to average of four non-halogenated standard compounds.
<table>
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<table>
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<tr>
<th>Peak No.</th>
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<th>Conc (µg/g)</th>
<th>Conc (µg/g)</th>
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a Quantitation based on an actual standard compound.
b Quantitation based on comparison to average of three halogenated standard compounds.
c Quantitation based on comparison to average of four non-halogenated standard compounds.
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a Quantitation based on an actual standard compound.

b Quantitation based on comparison to average of three halogenated standard compounds.

c Quantitation based on comparison to average of four non-halogenated standard compounds.
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*a* Quantitation based on an actual standard compound.

*b* Quantitation based on comparison to average of three halogenated standard compounds.

*c* Quantitation based on comparison to average of four non-halogenated standard compounds.
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*a* Quantitation based on an actual standard compound.

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