Introduction
Microwave sample preparation provides an efficient and clean sample preparation for multi-element analytical techniques such as ICP-OES and ICP-MS. As Microwave assisted digestion has evolved, so have the methodologies. EPA method 3052 is designed for the “total” analysis in a variety of matrices including soil, sediments, sludge, oils, biological and botanical materials. This method is the most versatile and has been well proven. It allows variations in reagents and methodology, making it ideal for a variety of matrices and elements. The variability of this method also requires a basic understanding of digestion chemistry.

Benefits of Microwave Assisted Digestion
• Cleanliness of preparation environment
• Reproducible digestion,
• Improved QA/QC
• Reduces skill level as a factor
• Greatly reduces preparation time
The following table suggests reagents and their ratios using method 3052.

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Reference Material</th>
<th>Reagent and Volume (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soil</td>
<td>NIST SRM 2711 Montana Soil</td>
<td>HNO₃ 9  HF 3  HCl 2  H₂O₂ 1</td>
</tr>
<tr>
<td>Sediment</td>
<td>NIST SRM 2704 Buffalo River Sediment</td>
<td>HNO₃ 9  HF 3  HCl 2  H₂O₂ 1</td>
</tr>
<tr>
<td>Biological</td>
<td>NIST SRM 1577a Bovine liver</td>
<td>HNO₃ 9  HF 0  HCl 1  H₂O₂ 2</td>
</tr>
<tr>
<td>Botanical</td>
<td>NIST SRM 1547 Peach leaves</td>
<td>HNO₃ 9  HF 0.5  HCl 0.5  H₂O₂ 1</td>
</tr>
<tr>
<td>Botanical</td>
<td>NIST SRM 1567a Wheat flour</td>
<td>HNO₃ 9  HF 0  HCl 0.5  H₂O₂ 2</td>
</tr>
<tr>
<td>Waste Oil</td>
<td>NIST SRM 1054a Wear-metals in lubricating oil</td>
<td>HNO₃ 9  HF 0.5  HCl 0.5  H₂O₂ 2</td>
</tr>
</tbody>
</table>

The use of HCl in the digestion should be used anytime that Ag or Sb are analytes of interest. Studies have shown very poor recoveries without the use of HCl.

Peroxide may be used in all digestions, however be aware of the increased reactivity with organic materials.

**Method Procedure**

A 0.25 to 1.0 g sample is weighed out in the reaction vessel. 9 mL of nitric acid are then added to each vessel. Then depending on the matrix the proper amount of hydrofluoric and/or hydrochloric acids are then added. Finally the hydrogen peroxide is added. The vessel is allowed to react for approximately one minute prior to sealing the vessels.

Vessels should then be placed in the rotor and placed in the microwave. The vessels should then be heated to at least 180 °C over 5.5 minutes and then held at 180 °C for at least 9.5 minutes. (The heating profile may be modified for reactive matrices). An example of the Multiwave program is shown below.

### Analytical Results (ppb)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Expected Range</th>
<th>Spirulina-A</th>
<th>Spirulina-B</th>
<th>Spirulina Avg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>As</td>
<td>150 to 300</td>
<td>269</td>
<td>274</td>
<td>272</td>
</tr>
<tr>
<td>Cd</td>
<td>10 to 75</td>
<td>16</td>
<td>15</td>
<td>15.5</td>
</tr>
<tr>
<td>Hg</td>
<td>10 to 75</td>
<td>16</td>
<td>14</td>
<td>15</td>
</tr>
<tr>
<td>Pb</td>
<td>50 to 150</td>
<td>77</td>
<td>74</td>
<td>75.5</td>
</tr>
</tbody>
</table>
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CVAA, graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma atomic emission spectrometry (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS) and other analytical elemental analysis techniques where applicable.

- Aluminum • Cadmium • Iron • Molybdenum • Sodium • Antimony • Calcium

- Lead • Nickel • Strontium • Arsenic • Chromium • Magnesium • Potassium

- Thallium • Boron • Cobalt • Manganese • Selenium • Vanadium • Barium

- Copper • Mercury • Silver • Zinc • Beryllium

Other elements and matrices may be analyzed by this method if performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest.

Conclusion

The Multiwave 3000 is designed for both high productivity and excellent digestion performance without compromising safety. Up to 16 PTFE-TFM vessels per run cope with a wide selection of samples, which require reaction conditions of up to 40 bar and 240 °C. An immersing pressure/temperature sensor in one reference vessel provides accurate reaction control. Tool-free and easy vessel handling allows for safe and convenient routine work.

The Multiwave 3000 is one of the most versatile sample preparation platforms available. It can easily perform the EPA method 3052 digestion procedure, bringing to the lab all of the benefits associated with microwave assisted closed vessel digestion. Any laboratory doing trace metals analysis would instantly benefit from the application of this digestion procedure. Samples would see less contamination, digestions would become more reproducible, and QC would be much tighter.

References


Results

The goal of this method is total sample decomposition and with judicious choice of acid combinations this is achievable for most matrices. Selection of reagents which give the highest recoveries for the target analytes is considered the optimum method condition.

Digests and alternative procedures produced by the method are suitable for analysis by flame atomic absorption spectrometry (FLAA), cold vapor atomic absorption spectrometry

Example of digestion and analytical results. Sample was a high organic matrix with volatile elements.