Microwave Digestion of Sediment (BCCS - 1 CRM) (Leach)

Procedure
Weigh 0.5 g of the sample into the digestion vessel. Add 10 mL of HNO3. Gently swirl the mixture and wait approximately 15 minutes before closing the vessel.

Notes
This method may not provide a total digest of all samples. Hydrofluoric acid will be required to provide complete digestion of some sample matrices.

The addition of Conc. HCl (0-4 mL) is appropriate for the stabilization of Ag, Ba and Sb, and high concentrations of Fe and Al in solution. The amount of HCl will vary depending on the matrix and the concentration of the analytes. The addition of HCl may, however, limit the techniques or increase the difficulties of analysis.

Recommended Equipment
MARS 6
MARS 6 iWave

Recommended Vessels
EasyPrep
EasyPrep Plus
MARSXpress
MARSXpress Plus

Reagents
HNO₃

Max Sample Weight
0.5 g

Sample Type
Organic

Control Type
Ramp to Temperature

Method Type
One Touch

Heating Program

<table>
<thead>
<tr>
<th>Stage</th>
<th>Temp (°C)</th>
<th>*Ramp (mm:ss)</th>
<th>Hold (mm:ss)</th>
<th>Pressure (psi)</th>
<th>* Power (W)</th>
<th>Stirring</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>180</td>
<td>15:00</td>
<td>15:00</td>
<td>800</td>
<td>900-1050</td>
<td>Off</td>
</tr>
</tbody>
</table>

* Ramp times and power may vary depending on the type and number of vessels.

Results
Sample was clear, colorless, and particle free upon dilution to 50 mL.

General Precaution
a) This procedure is a reference point for sample digestion using a CEM system and may need to be modified or changed to obtain the required results on your sample.
b) If using a vessel other than the recommended choice, adjust sample size and pressure limit to values appropriate for the vessel chosen.
c) The control / reference vessel must contain the largest and most reactive sample.
d) Manual venting of CEM vessels should be performed when wearing hand/eye/body protection and when the vessel contents are at or below room temperature to avoid the potential for chemical burns. Always point the vent hole away from the operator.
e) If programming as One Touch, the ramp time and power will be automatically determined based on the number and type of vessels detected.