1.0 SCOPE

1.1 This method is a microwave assisted digestion, using potassium dichromate (K₂Cr₂O₇), to determine the organic carbon content of a soil sample. As organic carbon in soil gets oxidized by the digestion procedure, Cr(VI) gets reduced to Cr(III), which adsorbs light at 600nm. There are negligible interferences from carbonates, Fe(II) and readily reducible Mn.

2.0 DEFINITIONS

2.1 Laboratory Control Sample: The laboratory control sample is an intralaboratory developed sample whose true organic carbon value is approximated by the average of repeated measures.

2.2 Duplicate Samples: A duplicate test involves splitting a sample to sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.

2.3 Preparation Blank: The Preparation Blank is a sample that contains only the reagents used in the extraction procedure. The preparation blanks is processed through the same preparation procedures as the samples and therefore gives an indication of any contamination picked up during the sample preparation process.

2.4 Pre-digestion Spike: A duplicate sample is spiked prior to digestion in order to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

3.0 EQUIPMENT AND SUPPLIES

3.1 MARS 1600 watt microwave (CEM corporation, Mathews, NC). Note: The microwave power output test, power calibration, and temperature probe calibration should be performed according to manufacturers specifications every six months.

3.2 49.64 g Na₂Cr₂O₇/L. All weighing mixing and transferring should take place in designated fume hood in 409. Refer to chemical hygiene plan – SOP for chromic acid for additional precautions and procedures.

3.3 Trace metal H₂SO₄

3.4 ≥18 MΩ deionized water (DI).

3.5 4.754g sucrose/L (1ml = 2mg C)
Organic Carbon Determination by Reduction of Dichromate
Soil Environmental Chemistry Program, The Ohio State University
Version 8

3.6 0.475g sucrose/L (1mL = 0.2mg C)
3.7 15ml Falcon tubes
3.8 Spectrophotometer

4.0 PROCEDURE

4.1 Weigh 0.5g of well-mixed samples in duplicate to the nearest 0.001 g into an acid washed Teflon vessel equipped with a controlled pressure relief mechanism.

4.2 Prepare standard curve as follows:
4.2.1 Record pipette accuracy.

<table>
<thead>
<tr>
<th>Standard curve (mg C/50 mL)</th>
<th>blk</th>
<th>0.5</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>15</th>
<th>20</th>
<th>30</th>
</tr>
</thead>
<tbody>
<tr>
<td>mL sucrose stock (2mg C/mL)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>2.5</td>
<td>5.0</td>
<td>7.5</td>
<td>10.0</td>
<td>15.00</td>
</tr>
<tr>
<td>mL sucrose stock (0.2mg C/mL)</td>
<td>0</td>
<td>2.500</td>
<td>5.000</td>
<td>0.000</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>mL (g) DI post digest</td>
<td>20</td>
<td>17.50</td>
<td>15.00</td>
<td>17.50</td>
<td>15.00</td>
<td>12.50</td>
<td>10.00</td>
<td>5.00</td>
</tr>
</tbody>
</table>

4.3 Weigh 0.5g Prepare pre-digestion spike by spiking a designated sample duplicate with 5mL of 2mg C/mL sucrose stock.

4.4 Add 10ml ± 0.1 mL of the K$_2$Cr$_2$O$_7$ solution.
4.4.1 4.3a Record pipette accuracy.

4.5 CAREFULLY add 20ml ± 1.0 mL of H$_2$SO$_4$.
4.5.1 Record pipette accuracy.

****Add H$_2$SO$_4$ very slowly. The reaction occurring has the potential to be very violent and hazardous.***

4.6 Seal the vessel according to manufacturer’s specifications.

4.7 Record the mass of each sample+vessel+reagents.

4.8 Properly place the vessel in the microwave system according to the manufacturer’s recommended specifications.

4.9 Enable appropriate organic carbon method in the MARS unit software according to number of samples. 4.5a Samples are heated at 135°C for 30min.
4.10 Once the digests have cooled, remove from the microwave, remove one vessel at a time and:
   4.10.1 Record the mass. The mass must be within 1.0 g of the pre-digest mass.
   4.10.2 Remove cap, tare on vessel and add 20.0 g ≥18 MΩ DI water to samples.
   4.10.3 Add 15.0 g ≥18 MΩ DI water to pre-digestion spike sample
   4.10.4 Add ≥18 MΩ DI water to standards according to 4.2.
   4.10.5 Return cap and invert several times.
   4.10.6 Allow sediment to settle and pour of approximately 12 ml into labeled falcon tubes.

5.0 QUALITY CONTROL

5.1 Laboratory Control Sample (LCS): The laboratory control sample must fall within ± 20% of the known value. The laboratory control sample must be run with each batch of microwave digestions.

5.2 Sample Duplicates: The relative percent difference (RPD) must be no more than ±20%. One sample duplicate must be run with every microwave batch.

\[
\text{RPD} = 100 \times \frac{(S - D)}{\text{Avg. (S,D)}}
\]

5.3 Preparation Blank: If any analyte concentration is above the detection limit in the preparation blank, the lowest concentration of the analyte reported in associated samples must be ≥ 10 times the preparation blank concentration. A preparation blank must be performed with each batch of microwave digestes.

5.4 Pre-digestion Spike: Spike recoveries must fall within the limits of 75-125%. At least one spike analyses (matrix spikes) shall be performed on each batch of digestes.

6.0 REPORTING

6.1 Worksheets and appendices.
7.0 CORRECTIVE ACTION

<table>
<thead>
<tr>
<th>Pass/Fail</th>
<th>Flag</th>
<th>Measurement</th>
<th>QA/QC Check</th>
<th>Frequency</th>
<th>Acceptance Criteria</th>
<th>Corrective Action</th>
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</thead>
<tbody>
<tr>
<td>i</td>
<td>Dichromate Method</td>
<td>LCS</td>
<td>1/batch</td>
<td>±20% or w/in 95% PI</td>
<td>Check microwave function and re-digest batch.</td>
<td></td>
</tr>
<tr>
<td>ii</td>
<td>Sample prep</td>
<td>Blank</td>
<td>1/batch</td>
<td>Below MDL or samples &gt;10x</td>
<td>Check ICP for carryover and dish washing procedures re-digest batch.</td>
<td></td>
</tr>
<tr>
<td>iii</td>
<td>Reproducibility</td>
<td>Duplicate</td>
<td>1/batch</td>
<td>RPD ±20%</td>
<td>Check microwave function and re-digest batch.</td>
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<tr>
<td>iv</td>
<td>Dichromate Method/Matrix affects</td>
<td>Pre-Digest Spike</td>
<td>1/batch</td>
<td>±25%</td>
<td>Check microwave function and ICP for signs of matrix affects. Re-digest batch if ICP is acceptable.</td>
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8.0 REFERENCES


## 9.0 APPENDIX

Pipette Calibration Verification

<table>
<thead>
<tr>
<th>Volume</th>
<th>g DI</th>
<th>g DI</th>
<th>g DI</th>
<th>g DI</th>
<th>g DI</th>
<th>date</th>
<th>initials</th>
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10.0 INTERPRETATION

10.1 Soil organic matter, also referred to as “humus”, greatly influences the physical and chemical properties of soil. Mineral particles bound together by organic matter contribute to soil structure, lower bulk density, and increased water retention, all of which are beneficial for agriculture (Soane, 1990). Decomposing organic matter is a major source of nutrients for plants, especially phosphorus and nitrogen (Brady and Weil, 1996; Nelson and Sommers, 1996). Well-drained mineral soils typically contain 1-6% organic matter by mass, although it can be as high as 20-30% in some cases (Brady and Weil, 1996). Organic matter is typically estimated from organic carbon, which can be reliably determined in the laboratory. Multiplying organic carbon by 1.72 gives an approximate value of organic matter. However, a number of studies have suggested that factors of 1.9 or 2.0 may be more accurate in calculating organic matter, particularly in peat or other organic soils in which organic matter is greater than 20-30% (Brady and Weil, 1996; Nelson and Sommers, 1996). Mineral soils in Ohio would be expected to contain 1-4% organic carbon.