

Standard Operating Procedure  
3051a Microwave Assisted Acid Digestion of Soil  
Soil Environmental Chemistry Program, The Ohio State University  
Version 12

## **1.0 SCOPE**

- 1.1 This method is a microwave-assisted extraction using aqua regia and HNO<sub>3</sub>. This method is more aggressive in dissolving the sample matrix than methods using conventional heating with nitric acid (HNO<sub>3</sub>), or alternatively, nitric acid and hydrochloric acid (HCl), according to EPA Methods 200.2 and 3050. However, because Method 3051a does not accomplish total decomposition of the sample, the extracted analyte concentrations may not reflect the total content in samples where the analytes are occluded in recalcitrant mineral phases. This method is applicable to the microwave-assisted acid extraction/dissolution† of sediments, sludges, and soils, for the following elements: Aluminum (Al)\*, Antimony (Sb)\*, Arsenic (As), Barium (Ba)\*, Beryllium (Be)\*, Boron (B), Cadmium (Cd), Calcium (Ca), Chromium (Cr)\*, Cobalt (Co), Copper (Cu), Iron (Fe)\*, Lead (Pb), Magnesium (Mg)\*, Manganese (Mn), Molybdenum (Mo), Nickel (Ni), Potassium (K), Selenium (Se), Silver (Ag)\*, Sodium (Na), Strontium (Sr), Thallium (Tl), Vanadium (V)\*, Zinc (Zn). \*Indicates elements which typically require the addition of HCl to achieve equivalent results with EPA Method 3050, as noted in reference 3. This method is intended to provide a rapid multi-element acid extraction or dissolution prior to analysis. Many types of samples will be dissolved by this method. A few refractory sample matrix compounds, such as quartz, silicates, titanium dioxide, alumina, and other oxides may not be dissolved and in some cases may sequester target analyte elements. These bound elements are considered non-mobile in the environment and are excluded from most aqueous transport mechanisms of pollution.

## **2.0 DEFINITIONS**

- 2.1 Laboratory Control Sample: The laboratory control used for the microwave digestion is a standard reference material (SRM) or certified reference material (CRM) that goes through the same extraction/preparation procedure as the samples. The analyte composition of the laboratory control sample is certified by acid dissolution method 3051a, 3050, or equivalent.
- 2.2 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.
- 2.3 Interference Check Standards: To verify interelement and background correction factors for the ICP, an Interference Check Samples (ICS) shall be analyzed with each microwave batch. The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists

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of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP

- 2.4 Duplicate Samples: A duplicate test involves splitting a sample into two sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.
- 2.5 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.
- 2.6 ICP-AES: Inductively Coupled Plasma-Atomic Emission Spectrometry.
- 2.7 ICP-HG-AES: ICP-AES with sample introduction using automated hydride generation
- 2.8 ICP-MS: Inductively Coupled Plasma-Mass Spectrometry.

### **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 manufacturer's specifications every six months.
- 3.2 Trace metal grade nitric acid.
- 3.3 Trace metal grade hydrochloric acid.
- 3.4  $\geq 18$  M $\Omega$  deionized water (DI).
- 3.5 15ml Falcon tubes
- 3.6 Spex CeriPrep Spike Sample Standard 1 (Cat# SPIKE-1-500)

### **4.0 PROCEDURE**

- 4.1 Weigh 0.5g of well-mixed samples in duplicate to the nearest 0.01 g into an acid washed Teflon vessel (4.1a) equipped with a controlled pressure relief mechanism.
- 4.2 Vessels should go through acid bath and DI rinse followed by 3x rinse with 3% acid from squirt bottle, then 3x rinse with reagent DI from squirt bottle.  
Note: Store washed vessels inverted in plastic racks.

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- 4.3 Record mass of sample on analysis sheet.
- 4.4 Add 1.0 mL of spiking solutions to the spike sample. Check pipette accuracy and record results in Appendix prior to spiking the sample.
- 4.5 Add  $3.0 \pm 0.1$  mL concentrated trace metal grade hydrochloric acid and  $9.0 \pm 0.1$  mL concentrated trace metal grade nitric acid with pipettes checked for accuracy (Section 9.0, Appendix) to each vessel in a fume hood.
  - 4.5.1 Pipette acids from disposable plastic dixie/solo cups.
  - 4.5.2 Any remaining acid should be collected into glass bottle for ICP torch cleaning.
  - 4.5.3 Seal the vessel according to manufacturer's specifications.
  - 4.5.4 Record the mass of each sample+vessel+acids.
- 4.6 Properly place the vessel in the microwave system according to the manufacturer's recommended specifications.
- 4.7 Enable appropriate 3051a method in the MARS unit software according to number of samples.
- 4.8 Once the digests have cooled to less than 75°C, remove from the microwave, remove one vessel at a time and:
  - 4.8.1 Record the mass on sample worksheet.
  - 4.8.2 The mass must be within 1.0 g of the pre-digest mass.
- 4.9 Remove cap, tare on vessel and add 38 g  $\geq 18$  MΩ DI water.
- 4.10 Return cap and invert several times.
- 4.11 Allow sediment to settle and pour off approximately 12 ml into labeled falcon tubes.
- 4.12 Pour off approximately 10ml of ICSA and 10ml of ICSB into labeled falcon tubes.
  - 4.12.1 Make sure ICSA and ICSB are on the analysis sheet (one set/analysis sheet).

## **5.0 QUALITY CONTROL**

- 5.1 Laboratory Control Sample (LCS): The laboratory control sample must fall within  $\pm 20\%$  of the known value or within the 95% prediction interval of the certified 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  $\pm 20\%$ . One sample duplicate must be run with every microwave batch.

$$RPD = 100 \times \frac{(S - D)}{S + D}$$

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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  $\geq 10$  times the preparation blank concentration. A preparation blank must be performed with each batch of microwave digests.
- 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 digests.
- 5.5 Interference Check Standard: The analytical results for those target analytes with MDLs  $< 10$  ug/L shall fall within  $+ 2x$  MDL of the analyte's true value (the true value shall be zero unless otherwise stated) in the ICS Solution A (ICSA). For example, if the analysis result(s) for Arsenic (MDL = 10 ug/L, ICSA true value = 0 ug/L) in the ICSA analysis during the run is  $+ 19$  ug/L, then the analytical result for Arsenic falls within the  $+ 2x$  MDL window for Arsenic in the ICSA. Results for the ICP analyses of Solution AB during the analytical runs shall fall within the control limit of  $+20\%$  of the true value for the analytes included in the Interference Check Samples.
- 5.6 INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes (mg/L)		Interferents (mg/L)
ICS B		ICS A & ICS B
Se 0.05	Tl 0.1	Al 500
As 0.1	Zn 1.0	Ca 500
Ba 0.5		Fe 200
Be 0.5		Mg 500
Cd 1.0		
Co 0.5		
Cr 0.5		
Cu 0.5		
Mn 0.5		
Ni 1.0		
Pb 0.05		

## **6.0 REPORTING**

- 6.1 Worksheets: Fill in appendix for pipettes used during the course of this SOP.

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## **7.0 CORRECTIVE ACTION**

Pass/ Fail	Flag	Measurement	QA/QC Check <sup>1</sup>	Frequency	Acceptance Criteria	Corrective Action
	i	3051a Method	LCS	1/batch	±20% or w/in 95% PI	Check microwave function and re-digest batch.
	ii	Sample prep	Blank	1/batch	Below MDL or samples >10x	Check ICP for carryover and dish washing procedures re-digest batch.
	iii	Reproducibility	Duplicate	1/batch	RPD ±20%	Check microwave function and re-digest batch.
	iv	3051a Method/ Matrix affects	Pre-Digest Spike	1/batch	±25%	Check microwave function and ICP for signs of matrix affects. Re-digest batch if ICP is acceptable.
	v	Interferences	ICS	1/batch	See 5.5	Determine how to correct the problem with the ICP and re- analyze samples by ICP.

## **8.0 REFERENCES**

- 8.1 Brobst, R. 1995. Biosolids management handbook. U.S. Environmental Protection Agency, Denver, CO.  
<https://www.epa.gov/sites/production/files/documents/handbook1.pdf>.
- 8.2 USEPA. 2007. Method 3051a. Microwave assisted acid digestion of sediments, sludges, soils, and oils. *In* SW-846. U.S. Environmental Protection Agency, Washington, DC.
- 8.3 USEPA. 2007. Method 6010C. Inductively coupled plasma-atomic emission spectrometry. *In* SW-846. U.S. Environmental Protection Agency, Washington, DC.
- 8.4 US Geological Survey. National Geochemical Survey database. US Department of Interior, <http://mrdata.usgs.gov/geochemistry/ngs.html>.

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**9.0 APPENDIX**

Pipette Calibration Verification

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

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## 10.0 INTERPRETATION

10.1 Soil blends and soil blend components should be screened for elemental toxicity according to the USEPA part 503 table 3 limits (Table 1). US Geological Survey background soil data from Ohio (Table 1) should also be used to assess whether soil blend elemental content falls within typical soil ranges.

Table 1. Background soil ranges for the state of Ohio from the US Geological Survey database (USGS), and USEPA Part 503 limits (Brobst, 1995).

Element	Min	Max	Mean	Median	95th	Part 503 Table 3
mg kg <sup>-1</sup>						
Ag	<1	<1	.	.	.	
Al	2.87	7.75	5.05	5.00	7.23	
As	4.30	26.6	9.97	9.70	16.9	41
Ba	242	565	438	450	529.5	
Be	0.800	2.80	1.54	1.50	2.45	
Bi	0.110	0.410	0.215	0.210	0.345	
Ca	0.0800	4.29	0.582	0.440	1.625	
Cd	<0.1	0.900	.	0.300	0.8	39
Ce	30.4	101	62.3	60.1	83.85	
Co	3.30	32.4	11.6	10.7	20.55	
Cr	16.0	66.0	38.4	37.0	58	
Cs	<5	10.0	.	5.00	8	
Cu	7.50	55.1	20.4	19.1	37.15	1500
Fe	1.17	4.29	2.48	2.46	3.55	
Ga	5.89	16.8	10.0	9.61	15.15	
Hg	0.0200	0.190	0.0561	0.0500	0.13	17
In	0.0300	0.0800	0.0462	0.0400	0.07	
K	1.03	2.59	1.67	1.68	2.36	
La	14.4	51.4	31.2	30.1	43.4	
Li	14.0	66.0	30.2	28.0	51.5	
Mg	0.160	1.94	0.482	0.420	0.97	
Mn	155	2710	822	684	2200	
Mo	0.690	12.7	2.94	2.25	7.115	
Na	0.210	1.06	0.556	0.530	0.9	
Nb	6.30	14.0	10.7	10.8	13.75	
Ni	7.80	39.3	21.2	20.2	37.1	420
P	310	3840	873	770	1545	
Pb	16.6	148	33.8	29.8	50.75	300
Rb	40.3	126	76.9	76.5	107	

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S	0.0200	0.0900	0.0458	0.0500	0.075	
Sb	0.400	1.74	0.781	0.720	1.255	
Sc	3.90	14.0	7.61	7.30	11.9	
Se	0.300	0.900	0.578	0.600	0.85	100
Sn	1.10	11.0	2.27	1.90	5	
Sr	42.0	193	97.3	89.7	167.5	
Te	<0.1	0.300	.	0.100	.	
Th	4.60	16.6	10.6	10.3	14.2	
Ti	0.210	0.420	0.327	0.330	0.41	
Tl	0.300	1.50	0.743	0.700	1.05	
U	1.70	9.00	4.14	3.90	6.2	
V	31.0	120	65.6	66.0	96.5	
W	0.600	2.40	1.24	1.20	1.8	
Y	10.3	30.9	16.9	15.8	26.8	
Zn	33.0	423	91.9	85.0	158	2800

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