Microwave Assisted Acid Digestion of Sediments, Soils, and Tissues


1. Scope and Application

Matrices: This method is applicable to the microwave-assisted acid extraction/dissolution of sediments, soils and tissue samples.

Definitions: Refer to Alpha Analytical Quality Manual.

This microwave extraction method is designed to mimic extraction using conventional heating with nitric acid (HNO₃), or alternatively, nitric acid and hydrochloric acid (HCl), according to EPA Methods 200.2, 3010, and 3050. Since these methods are not intended to accomplish total decomposition of the sample, the extracted analyte concentrations may not reflect the total content in the sample. This method is applicable to the microwave-assisted acid extraction/dissolution of sediments, soils and tissue samples for the elements listed below.

This method is provided as an alternative to EPA Method 3050. This method provides options for improving the performance for certain analytes, such as antimony, iron, aluminum, and silver by the addition of hydrochloric acid, when necessary. It is intended to provide a rapid multi-element acid extraction or dissolution prior to analysis so that decisions can be made about materials and site cleanup levels, the need for TCLP testing of a waste (see EPA Method 1311, Section 1.2, for further details), and whether a BDAT process is providing acceptable performance. Digests produced by the method are suitable for analysis by flame atomic absorption spectrophotometry (FLAA), graphite furnace atomic absorption spectrophotometry (GFAA), inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS). However, the addition of HCl may limit the methods of detection, or increase the difficulties of detection with some techniques.

Due to the rapid advances in microwave technology, consult your manufacturer's recommended instructions for guidance on their microwave digestion system.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.
2. Summary of Method

For soils, sediments and tissues, a representative sample of 0.5-1.5 g is extracted and/or dissolved in 10mL concentrated nitric acid for 10 minutes using microwave heating with a suitable laboratory microwave unit. The sample and acid(s) are placed in a fluorocarbon polymer (PFA or TFM) or quartz microwave vessel or vessel liner. The vessel is sealed and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed by the appropriate determinative method.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to the appropriate analytical SOP for reporting limit information.

4. Interferences

4.1 Very reactive samples or volatile materials may create high pressures due to the evolution of gaseous digestion products. This may cause venting of the vessels with potential loss of sample and/or analytes. The complete decomposition of either carbonates, or carbon based samples, may produce enough pressure to vent the vessel if the soil sample size is greater than 0.25 g (depending on the pressure capability of the vessel). Variations of the method to accommodate very reactive materials are specifically addressed in Section 10.3.1.3.

4.2 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.

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5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The microwave unit cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation. Never use an Advanced Composite Vessel without a composite sleeve. Prior to use, all vessel components must be dry and free of particulate matter. Drops of liquid or particles will absorb microwave energy that can lead to localized charring which can lead to vessel damage or failure. Never install more than one rupture membrane in an Advanced Composite Vessel.

**CAUTION:** There are many safety and operational recommendations specific to the model and manufacturer of the microwave equipment. The analyst is advised to consult the equipment manual (see 1.3), the equipment manufacturer, and other appropriate literature for proper and safe operation of the microwave equipment and vessels.

5.2 The method requires essentially microwave transparent and reagent resistant materials such as fluorocarbon polymers (examples are PFA or TFM) or quartz to contain acids and samples. For higher pressure capabilities the vessel may be contained within layers of different microwave transparent materials for strength, durability, and safety. The internal volume of the vessel should be at least 45mL, and the vessel must be capable of withstanding pressures of 200psi, and capable of controlled pressure relief. These specifications are to provide an appropriate, safe, and durable reaction vessel of which there are many adequate designs by many suppliers. CEM provides 100mL vessels made of Teflon PFA, caps made of Ultem polyetherimide (reinforced), and sleeves of composite glass, Teflon PFA and Ultem.

5.3 **CAUTION:** The reagent combination (9mL nitric acid to 3mL hydrochloric acid) results in greater pressures than those resulting from the use of only nitric acid. Pressures of approximately 12atm have been reached during the heating of the acid mixture alone (no sample in the vessel). Pressures reached during the actual decomposition of a sediment sample (with low organic content) have more than doubled when using the 9mL nitric and 3mL hydrochloric acid mixture. These higher pressures may necessitate the use of vessels having higher pressure capabilities. Matrices having large organic content, such as oils, can produce approximately 25atm of pressure inside the vessel (as described in EPA Method 3052).

5.4 **CAUTION:** Another safety concern relates to the use of sealed containers without pressure relief devices. Temperature is the important variable controlling the reaction. Pressure is needed to attain elevated temperatures, but must be safely contained. Some digestion vessels constructed from certain fluorocarbons may crack, burst, or explode in the unit under certain pressures. Only fluorocarbon (such as PFA or TFM and others) or quartz containers with pressure relief mechanisms or containers with fluorocarbon or quartz liners and pressure relief mechanisms are considered acceptable.

5.5 **CAUTION:** Laboratories should NOT use domestic (kitchen) type microwave ovens for this method because of significant safety issues. When acids such as nitric and hydrochloric are used to effect sample digestion in microwave units in open vessel(s), or sealed vessel(s),
there is the potential for any released acid vapors to corrode the safety devices that prevent the microwave magnetron from shutting off when the door is opened. This can result in operator exposure to microwave energy. Use of a system with isolated and corrosion resistant safety devices prevents this from occurring.

5.6 Users are therefore advised NOT to use domestic (kitchen) type microwave ovens or sealed containers which are not equipped with controlled pressure relief mechanisms for microwave acid digestions by this method. Use of laboratory-grade microwave equipment is required to prevent safety hazards.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection
All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of SW-846. Refer to that chapter, as updated, for guidance.

All sample containers must be pre-cleaned by the vendor. Plastic and glass containers are both suitable. For further information, see Chapter Three of SW-846.

6.2 Sample Preservation
No preservatives are used.

6.3 Sample Shipping
No special shipping requirements. Typical shipping procedures may be found in the Alpha’s Sample Receipt and Log-In SOP (01-01).

6.4 Sample Handling
Samples should be refrigerated upon receipt (4˚C ± 2) or frozen at -20˚C ± 10˚C.

7. Equipment and Supplies

7.1 Microwave apparatus: Milestone Ethos E 1000 W Microwave Oven with maximum operating temperature of 200˚C and maximum operating pressure of 200 PSIG/13.8 Bar.

7.1.1 The temperature performance requirements necessitate the microwave decomposition system to sense the temperature to be within ± 2.5°C and automatically adjust the microwave field output power within 2 seconds of sensing. Temperature sensors should be accurate to ± 2.5°C (including the final reaction temperature of the digestion procedure). Temperature feedback control provides the primary performance mechanism for the method. Due to the variability in sample matrix types and microwave digestion equipment (i.e., different vessel types and microwave oven designs), temperature feedback control is preferred for reproducible microwave heating.

7.1.2 Alternatively, for a specific vessel type, specific set of reagent(s), and sample type, a calibration control mechanism can be developed. Through calibration of the microwave power for a specific number and type of vessels, vessel load, and heat loss characteristics of a specific vessel series, the reaction temperature profile described in Sec. 11.3.5 can be reproduced. The calibration settings are specific for the number and type of vessels and microwave system being used, in addition to the specific reagent combination being used. Therefore, no specific calibration settings are provided in this method. These settings may be developed by using temperature monitoring equipment for each specific set of microwave equipment and vessel type. In this circumstance, the microwave
A rotating turntable is employed to ensure the homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3 rpm. Other types of equipment that are used to assist in achieving uniformity of the microwave field may also be appropriate.

7.1.4 Milestone Fiber Optic Temperature Probe Part # FO00110

7.2 Mechanical Pipette: Class A or appropriate

7.3 Volumetric Graduated Cylinder: Class A or appropriate, 50 or 100mL capacity or equivalent.

7.4 Filter paper, qualitative or equivalent

7.5 Filter funnel, glass, polypropylene, or other appropriate material

7.6 Balance: Top loading balance capable of reading two decimal places

7.7 Spatulas

7.8 Digestion Tubes: Pre-cleaned, graduated, disposable tubes, 50 mL volume (Environmental Express part # SC475 or equivalent) with Filter Assembly (Environmental Express Part # SC0408 or equivalent): The 50mL volume of each Lot of tubes is verified and documented in a logbook (Form No.: 105-02).

8. Reagents and Standards

All acids should be sub-boiling distilled where possible to minimize the blank levels due to metallic contamination. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without decreasing the accuracy of the determination. If the purity of a reagent is questionable, the reagent should be analyzed to determine the level of impurities. The reagent blank must be less than the RL in order to be used. The expiration date of all reagents and standards is six months unless otherwise noted. Reagents and standards are stored at room temperature.

8.1 Concentrated nitric acid (HNO₃). Prior to use, the acid is analyzed to determine levels of impurity. If the method blank is less than the RL, the acid can be used. Results of this analysis are kept in a logbook.

8.2 Concentrated hydrochloric acid (HCl). Prior to use, the acid is analyzed to determine levels of impurity. If the method blank is less than the RL, the acid can be used. Results of this analysis are kept in a logbook.

8.3 Reagent Grade Deionized Water (DI): Reagent water shall be interference free. All references to water in the method refer to reagent grade DI water unless otherwise specified.
8.4 Spiking solutions:

8.4.1 Laboratory Control Sample (LCS) and High Matrix Spiking (MS) solution for 3051: Spike 0.5mL of S1 into the LCS and MS samples. The final concentration in the LCS and MS samples are Be and Cd at 0.5mg/L; Sb, Ba, B, Cr, Cu, Co, Mn, Zn, Ni, Pb, V, Ti, As, Se and Mo at 1.0mg/L; Al, Ca, Fe, Mg, K, and Na at 5.0mg/L

8.4.1.1 S1 Solution: Prepare by adding 5mL of concentrated HNO₃ to a 100mL volumetric flask using a 5mL glass pipette. Then add 10mL of ICUS-624 (Ultra Scientific) solution containing Be and Cd at 500mg/L, for a concentration of 50mg/L, and Sb, Ba, B, Cr, Cu, Co, Mn, and Mo at 1000mg/L, for a concentration of 100mg/L. Next add 10mL of ICUS-625 (Ultra Scientific) solution containing Zn, Ni, Pb and V at 1000mg/L for a concentration of 100mg/L, and Al, Ca, Fe, Mg, K, and Na at 5000mg/L, for a concentration of 500mg/L. Next add 10mL each of Ti, As and Se (SPEX) at 1,000mg/L for a concentration of 100mg/L. This solution is brought to volume with DI water

8.4.2 Laboratory Control Sample (LCS) and Low Matrix Spiking (MS) solution for 3051: Spike 0.80mL of S3 into the LCS sample and MS sample.

8.4.2.1 S3 Solution: Prepare by adding 5mL of concentrated HNO₃ to a 100mL volumetric flask using a 5mL glass pipette. Then add 2.5mL of IQC-026 (Ultra Scientific) Elements solution containing Ag, Al, As, Ba, Be, B, Ca, Cd, Cr, Cu, Co, Fe, Pb, Mg, Mn, Mo, Ni, Sb, Se, Ti, Ti, V, and Zn at 100mg/L. Si is present at 50 mg/L and K is present at 1000 mg/L for a concentration of 2.5mg/L for all elements except Si (1.25 mg/L) and K (25 mg/L). This solution is brought to volume with DI water.

8.4.2.2 Hg solution: Prepare by diluting the 1000 mg/L stock standard 1:50 in 2% HCl. Add 0.1 mL of stock to 100 ml volumetric flask and bring to volume to make a 1.0 mg/L solution. Add 1.25mL of 1.0mg/L Hg to LCS and matrix spike low standards

Note: The mercury spike concentration may be reduced if lower reporting limits are required. The spike volume used for plant tissue is 0.025 mL.

8.5 Solid Matrix Sample: Catalog # 540 from ERA

9 Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A blank must be processed with each analytical batch or every 20 samples whichever is more frequent. A blank is brought through the whole sample preparation and analysis process. All measured analyte concentrations in the preparation blank must be below the reporting limit. Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. The batch should be re-prepared for any analyte whose concentration exceeds the reporting limit. A blank whose analyte concentrations exceed the
reporting limit may be deemed acceptable by the Metals Supervisor if analyte concentration in the affected sample are below the reporting limit or greater than ten times the reporting limit.

If required, and a blank solid material of a similar matrix type (Environmental Resource Associates #058 or equivalent) of high enough purity can be obtained to meet required reporting limits, a solid material may be used in the Method Blank preparation to matrix match QC samples and field samples.

9.2 Laboratory Control Sample (LCS)

A LCS is processed with each analytical batch or every 20 samples whichever is more frequent. A LCS is a blank spike brought through the whole sample preparation and analysis process. Nominal recovery limits for an LCS are 80% - 120%. Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. The batch should be re-prepared for any analyte whose recovery is outside of these limits. The Metals Supervisor may deem an LCS whose recovery for any analyte is outside of these limits acceptable if acceptable recoveries from a matrix spike and/or SRM prepared with the batch are within control limits.

If required, a solid LCS (Environmental Resources Associates # 544 or equivalent) may be prepared to matrix match QC samples and field samples.

9.3 Initial Calibration Verification (ICV)

Not Applicable

9.4 Continuing Calibration Verification (CCV)

Not Applicable

9.5 Matrix Spike

Spiked samples (MS) or standard reference materials should be included with each group of samples processed, or every 20 samples whichever is more frequent. A spiked sample should also be included whenever a new sample matrix is being analyzed. An SRM is used when required by the project.

9.6 Laboratory Duplicate

Duplicate samples should be processed on a routine basis. A duplicate sample is a sample brought through the whole sample preparation and analysis process. A duplicate sample must be processed with each analytical batch or every 20 samples whichever is more frequent. A duplicate sample should be prepared for each matrix type (i.e., soil, sludge, etc.).

9.7 Method-specific Quality Control Samples

9.8 Method Sequence

- Method Blank
- Laboratory Control Sample
- Laboratory Duplicate
- Matrix Spike
- Samples 1-20

10 Procedure
10.1 Equipment Set-up

10.1.1 Temperature control of closed vessel microwave instruments provides the main feedback control performance mechanism for the method. Method control requires a temperature sensor in one or more vessels during the entire decomposition. The microwave decomposition system should sense the temperature to within ± 2.5 °C and permit adjustment of the microwave output power within 2 seconds.

10.1.2 All digestion vessels and volumetric ware must be carefully acid washed and rinsed with DI water. For normal cleaning, vessels are rinsed with DI water and soaked in DI water overnight. They are then assembled, 10 ml concentrated HNO₃ is added and digested using the method program. They are then rinsed with DI water and dried in a clean environment. When switching between high concentration samples and low concentration samples, all digestion vessels (fluoropolymer or quartz liners) should be thoroughly cleaned using the procedure above, visually inspected, and checked for contamination by the analysis of a preparation blank. This cleaning procedure should also be used whenever the prior use of the digestion vessels is unknown or cross contamination from prior sample digestions in vessels is suspected.

10.2 Initial Calibration

Not applicable

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Digestion

10.3.1.1 For soil, sediment and tissue samples, weigh a well-mixed sample to the nearest 0.01 g into an appropriate vessel equipped with a controlled pressure relief mechanism. For soils, sediments, and tissues use 0.5-1.5 g. If the sample can not be well mixed and homogenized on an as received basis, then air or oven drying at 60°C or less, crushing, sieving, grinding, and mixing should be performed as necessary to homogenize the sample until the subsampling variance is less than the data quality objectives of the analysis. While proper sample preparation generally produces great reduction in analytical variability, be aware that in certain unusual circumstances there could be loss of volatile metals (e.g., Hg, organometallics) or irreversible chemical changes (e.g., precipitation of insoluble species, change in valence state). See Chapter Three of SW-846 for more details.

10.3.1.2 For soil, sediment and tissue samples, add 10mL ± 0.1mL concentrated nitric acid to the vessel in a fume hood (or fume exhausted enclosure). The addition of concentrated hydrochloric acid to the nitric acid is appropriate for the stabilization of certain analytes, such as Ag, Ba, and Sb and high concentrations of Fe and Al in solution. The addition of hydrochloric acid may, however, limit the detection techniques or increase the difficulties of analysis for some detection systems such as ICP-MS (Method 6020) or GFAA (Method 7000). Spike LCS and MS samples. Samples are predigested for 10-120 minutes under a hood with the vessels loosely capped to allow gases to escape.

**CAUTION:** The addition of hydrochloric acid must be in the form of concentrated hydrochloric acid and not from a premixed combination of acids as a buildup of chlorine gas, as well as other gases, will result from a premixed acid solution. These gases may be violently released upon heating. This is avoided by adding the acid in the described manner.
CAUTION: Toxic nitrogen oxide(s) and chlorine fumes are usually produced during digestion. Therefore, all steps involving open or the opening of microwave vessels must be performed in a properly operating fume ventilation system.

CAUTION: The analyst should wear protective gloves and face protection.

CAUTION: The use of microwave equipment with temperature feedback control is required to control any unfamiliar reactions that may occur during the leaching of samples of unknown composition. The leaching of these samples may require additional vessel requirements such as increased pressure capabilities.

10.3.1.3 The analyst should be aware of the potential for a vigorous reaction, especially with samples containing volatile or easily oxidized organic species. When digesting a matrix of this type, initially use no more than 0.100 g of sample. If a vigorous reaction occurs upon the addition of reagent(s), allow the sample to predigest in the uncapped digestion vessel until the reaction ceases. Heat may be added in this step for safety considerations (for example, the rapid release of carbon dioxide from carbonates, easily oxidized organic matter, etc.). Once the initial reaction has ceased, the sample may continue through the digestion procedure. However, if no appreciable reaction occurs, a sample mass of 0.250 g for oils, or 0.10-5 g for soil, sediments or tissues may be used.

10.3.1.4 Seal the vessel according to the manufacturer's directions. Properly place the vessel in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure sensors to vessels according to manufacturer's specifications.

10.3.1.5 This method is a performance based method, designed to achieve or approach consistent leaching of the sample through achieving specific reaction conditions. For soil, sediment and tissue samples, the temperature of each sample should rise to 175 ± 5 °C in approximately 5 minutes and remain at 175 ± 5°C for the remainder of the 10 minutes digestion period. When using temperature feedback control, the number of samples that may be simultaneously digested may vary, from one sample up to the maximum number of vessels that can be heated by the magnetron of the microwave unit according to the heating profile specified previously in this section. (The number will depend on the power of the unit, the number of vessels, and the heat loss from the vessels). If vessel rupture is observed in soil sample digestion, the temperature may be reduced to 150 °C.

NOTE: The pressure should peak between 5 and 10 minutes for most soil, sediment and tissue samples. If the pressure exceeds the pressure limits of the vessel, the pressure should be safely and controllably reduced by the pressure relief mechanism of the vessel.

10.3.1.5.1 Calibration control is applicable in reproducing this method provided the power in watts versus time parameters are determined to reproduce the specifications listed in 11.3.5. The calibration settings will be specific to the quantity of reagents, the number of vessels, and the heat loss characteristics of the vessels. If calibration control is being used, any vessels containing acids for analytical blank purposes are counted as sample vessels. When fewer than the recommended number of samples are to be digested, the remaining vessels should be filled with the same acid mixture to achieve the full complement of vessels. This provides an energy balance, since the microwave power absorbed is proportional to the total absorbing mass in the cavity. Irradiate each group of vessels using the predetermined calibration settings. (Different vessel types should not be mixed).
10.3.1.6 At the end of the microwave program, allow the vessels to cool for a minimum of 5 minutes before removing them from the microwave system. Cooling of the vessels may be accelerated by internal or external cooling devices. When the vessels have cooled to near room temperature, determine if the microwave vessels have maintained their seal throughout the digestion. Due to the wide variability of vessel designs, a single procedure is not appropriate. For vessels that are sealed as discrete separate entities, the vessel weight may be taken before and after digestion to evaluate seal integrity. If the weight loss of sample exceeds 10% of the weight of the sample and reagents, then the sample is considered compromised. For vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised vessels. For vessels with resealing pressure relief mechanisms, an auditory or a physical sign that can indicate whether a vessel has vented is appropriate.

Samples must be weighed before and after digestion on a top loading balance capable of reading to 0.01 g. The combined weights of sample plus reagents should lose no more than 10% of the initial mass. If the weight loss exceeds the 10% limit, the sample digest should be considered compromised and re-prepared. Weight loss records will be maintained in the digestion log book along with each sample preparation.

10.3.1.7 Complete the preparation of the sample by carefully uncapping and venting each vessel in a chemical fume hood (or fume exhausted enclosure). Vent the vessels using the procedure recommended by the vessel manufacturer. Quantitatively transfer the sample to a clean vial and bring to 50 mL final volume with DI water. If the digested sample contains particulates that may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be, allowed to settle (10.3.1.7.1), or filtered (10.3.1.7.2).

10.3.1.7.1 Settling: If undissolved material, such as SiO$_2$, TiO$_2$, or other refractory oxides, remains, allow the sample to stand until the supernatant is clear. Allowing a sample to stand overnight will usually accomplish this. If it does not, centrifuge or filter the sample.

10.3.1.7.2 Filtering: Samples may be filtered in the 50 mL sample vial using a filter assembly (See Section 7.8). If necessary, a filtering apparatus may be used after thoroughly cleaning and rinsing with dilute (approximately 10% V/V) nitric acid. Filter the sample through qualitative filter paper into a second acid-cleaned container.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

10.5.1 Schedule annual maintenance with instrument a service company.

10.5.2 The accuracy of the temperature measurement system should be validated prior to use of a new probe and annually. This can be done using oil and an external calibrated temperature measurement system. The solution should be adequately stirred to ensure a homogeneous temperature, and both the microwave temperature sensor and the external temperature sensor placed into the oil. The temperature between the probe and external temperature measurement system should be compared at several different temperatures from room temperature to approximately 100 °C. If the measured temperatures vary by more than 2.5 °C, the microwave temperature measurement system should be calibrated or the probe should be replaced. Consult the microwave manufacturer’s instructions.
10.5.3 Visually inspect digestion vessels for signs of wear.

11 Data Evaluation, Calculations and Reporting

11.1 For soil, sediment and tissue samples, calculate the sample dry-weight fraction as follows:

$$\text{Dry-Wt fraction} = \frac{W_2 - W_3}{W_1 - W_3}$$

Where:

- $W_1 = \text{Wt for sample + vessel, before drying, g}$
- $W_2 = \text{Wt for sample + vessel, after drying, g}$
- $W_3 = \text{Wt for empty, dry vessel, g}$

11.2 Convert the soil, sediment and tissue extract concentrations obtained from the instrument in mg/L to mg/kg dry-weight of sample by

$$\text{Sample concentration} = \frac{(C)(V)(D)}{(W)(S)}$$

Where:

- $C = \text{Concentration in extract (mg/L)}$
- $D = \text{Dilution factor}$
- $S = \text{Solid dry-weight fraction for sample, g/g}$
- $V = \text{Volume of extract, mL x 0.001}$
- $W = \text{Weight of undried sample extracted, g x 0.001}$

12 Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 and the appropriate analytical SOPs outline sample batch QC acceptance criteria. If non-compliant inorganic element results are to be reported, the Metals Section Head and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Section Head performing the secondary analytical review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.
13 Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14 Pollution Prevention and Waste Management

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15 Referenced Documents

- Chemical Hygiene Plan
- SOP/01-01 Sample Receipt & Log-In
- SOP/08-05 MDL/LOD/LOQ Generation
- SOP/08-12 IDC/DOC Generation
- SOP/14-01 Waste Management and Disposal SOP

16 Attachments

None.