METHOD 3051A

# MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

## 1.0 SCOPE AND APPLICATION

1.1 This microwave extraction method is designed to mimic extraction using conventional heating with nitric acid ($HNO_3$), or alternatively, nitric acid and hydrochloric acid (HCl), according to EPA Method 200.2 and Method 3050. Since this method is 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, sludges, soils, and oils for the following elements:

| Element | | CAS Registry No. [a] |
|---|---|---|
| *Aluminum | (Al) | 7429-90-5 |
| *Antimony | (Sb) | 7440-36-0 |
| Arsenic | (As) | 7440-38-2 |
| *Barium | (Ba) | 7440-39-3 |
| *Beryllium | (Be) | 7440-41-7 |
| Boron | (B) | 7440-42-8 |
| Cadmium | (Cd) | 7440-43-9 |
| Calcium | (Ca) | 7440-70-2 |
| *Chromium | (Cr) | 7440-47-3 |
| Cobalt | (Co) | 7440-48-4 |
| Copper | (Cu) | 7440-50-8 |
| *Iron | (Fe) | 7439-89-6 |

| Element | | CAS Registry No. [a] |
|---|---|---|
| Lead | (Pb) | 7439-92-1 |
| *Magnesium | (Mg) | 7439-95-4 |
| Manganese | (Mn) | 7439-96-5 |
| Mercury | (Hg) | 7439-97-6 |
| Molybdenum | (Mo) | 7439-98-7 |
| Nickel | (Ni) | 7440-02-0 |
| Potassium | (K) | 7440-09-7 |
| Selenium | (Se) | 7782-49-2 |
| *Silver | (Ag) | 7440-22-4 |
| Sodium | (Na) | 7440-23-5 |
| Strontium | (Sr) | 7440-24-6 |
| Thallium | (Tl) | 7440-28-0 |
| *Vanadium | (V) | 7440-62-2 |
| Zinc | (Zn) | 7440-66-6 |

[a] Chemical Abstract Service Registry Number

*Indicates elements which typically require the addition of HCl to achieve equivalent results with Method 3050, as noted in Ref. 3.

[‡]Note: For matrices such as certain types of oils, this method may or may not provide total sample dissolution. For other matrices, such as soils and sediments, it should be considered an extraction method. 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 (see Sec. 9.0).

1.2 This method is provided as an alternative to EPA Method 200.2 and 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 Method 1311), 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 quantitation methods, or increase the difficulties of quantitation with some techniques.

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

1.3 Prior to employing this method, analysts are advised to consult the determinative method that may be employed in the overall analysis for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.4 Use of this method is restricted to use by, or under supervision of, properly personnel experienced and trained in the use of microwave digestion systems. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

A representative sample is extracted and/or dissolved in concentrated nitric acid, or alternatively, concentrated nitric acid and concentrated hydrochloric acid 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 for a specified period of time. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed by the appropriate determinative method.

## 3.0 DEFINITIONS

Refer to Chapter One, Chapter Three and the manufacturer's instructions for definitions that may be relevant to this procedure.

## 4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on quality control procedures and to Chapter Three for general guidance on the cleaning of glassware. Also refer to the determinative methods to be used for a discussion of interferences.

4.2 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 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 Sec. 11.3.3.

4.3 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.

4.4 Samples that are highly reactive or contaminated may require dilution before analysis. If samples are diluted, then any dilutions must be accounted for in all subsequent calculations. Highly reactive samples may also require pre-digestion in a hood to minimize the danger of thermal runaway and excessively vigorous reactions.

## 5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 The microwave unit cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation.

CAUTION: There are many safety and operational recommendations specific to the model and manufacturer of the microwave equipment used in individual laboratories. A listing of these specific suggestions is beyond the scope of this method. The analyst is advised to consult the equipment manual, the equipment manufacturer, and other appropriate literature for proper and safe operation of the microwave equipment and vessels. For further details and a review of safety methods during microwave sample preparation, see Ref. 3 and the document of Sec. 13.3.1.

5.3 This method requires 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 45 mL, and the vessel must be capable of withstanding pressures of at least 30 atm (435 psi), 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.

WARNING: The reagent combination (9 mL nitric acid to 3 mL hydrochloric acid) results in greater pressures than those resulting from the use of only nitric acid. As demonstrated in Figures 1 and 2, pressures of approximately 12 atm 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 (SRM 2704, a matrix with low organic content) have more than doubled when using the 9 mL nitric and 3 mL hydrochloric acid mixture. These higher pressures necessitate the use of vessels having higher pressure capabilities (30 atm or 435 psi). Matrices having large organic content, such as oils, can produce approximately 25 atm of pressure inside the vessel (as described in Method 3052).

WARNING: The outer layers of vessels are frequently not as acid or reagent resistant as the liner material. In order to retain the specified performance and safety requirements, these outer layers must not be chemically degraded or physically damaged. Routine examination of the vessel materials is necessary to ensure their safe use.

WARNING: 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 vessels approved by the manufacturer of the microwave system being used are considered acceptable.

WARNING: 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 laboratory-grade microwave equipment system with isolated and corrosion resistant safety devices prevents this from occurring. Use of laboratory-grade microwave equipment is needed to prevent safety hazards. For further details, consult Ref. 3 and the document listed in Sec. 13.3.1.

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.

## 6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

This section does not list common laboratory glassware (e.g., beakers and flasks).

### 6.1 Microwave apparatus requirements

6.1.1 The temperature performance requirements necessitate the microwave decomposition system to sense the temperature to within ± 2.5 EC and automatically adjust the microwave field output power within 2 seconds of sensing. Temperature sensors should be accurate to ± 2 EC (including the final reaction temperature of 175 ± 5 EC). 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. For further details consult Ref. 3.

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. They may be used as previously described in such methods as Methods 3015 and 3052. In this circumstance, the microwave system provides programmable power, which can be programmed to within ± 12 W of the required power. Typical systems provide a nominal 600 W to 1200 W of power. Calibration control provides backward compatibility with older laboratory microwave systems which may not be equipped for temperature monitoring or feedback control and with lower cost microwave systems for some repetitive analyses. Older vessels with lower pressure capabilities may not be compatible (see Refs. 1, 2, and 3 and the documents listed in Secs. 13.3.3 and 13.3.5).

6.1.2 The accuracy of the temperature measurement system should be periodically validated at an elevated temperature. This can be done using a container of silicon oil (a high temperature oil) and an external, calibrated temperature measurement system. The oil should be adequately stirred to ensure a homogeneous temperature, and both the microwave temperature sensor and the external temperature sensor placed into the oil. After heating the oil to a constant temperature of 180 ± 5 EC, the temperature should be measured using both sensors. If the measured temperatures vary by more than 1 to 2 EC, the microwave temperature measurement system should be calibrated. Consult the microwave manufacturer's instructions about the specific temperature sensor calibration procedure.

6.1.3 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.

6.2 Filter paper, qualitative or equivalent.

6.3 Filter funnel, glass, polypropylene, or other appropriate material.

6.4 Analytical balance, of appropriate capacity and resolution meeting data quality objectives.

## 7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 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 lower level of quantitation in order to be used.

7.2.1 Concentrated nitric acid ($HNO_3$) -- The acid should be analyzed to determine levels of impurity. If the method blank is less than the lower level of quantitation, the acid can be used.

7.2.2 Concentrated hydrochloric acid (HCl) -- The acid should be analyzed to determine levels of impurity. If the method blank is less than the lower level of quantitation, the acid can be used.

7.3 Reagent water -- Reagent water must be interference free. All references to water in this method refer to reagent water unless otherwise specified. For further details, consult the document listed in Sec. 13.3.2.

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 See the introductory material to Chapter Three, "Inorganic Analytes."

8.2 All sample containers must be prewashed with acids and water, and metal-free detergents, if necessary, depending on the history of use of the container (Ref. 3). Plastic and glass containers are both suitable. For further information, see Chapter Three.

## 9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection.

9.2 Initial demonstration of proficiency

Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This will include a combination of the sample extraction method and the determinative method (a 6000 or 7000 series method). The laboratory must also repeat the demonstration of proficiency whenever new staff are trained or significant changes in instrumentation are made.

9.2.1 Prepare the reference samples from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate should be made using stock standards prepared independently from those used for calibration.

9.2.2 The procedure for preparation of the reference sample concentrate is dependent upon the method being evaluated. Guidance for reference sample concentrations for certain methods is listed in Sec. 9.2.4. In other cases, the determinative methods may contain guidance on preparing the reference sample concentrate and the reference sample. In the absence of any other guidance, consult Sec. 9.3.3 and prepare the spiking solution accordingly.

The concentration of target analytes in the reference sample may be adjusted to more accurately reflect the concentrations that will be analyzed by the laboratory. If the concentration of an analyte is being evaluated relative to a regulatory limit or action level, see Sec. 9.3.3 for information on selecting an appropriate spiking level.

9.2.3 To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. See the note in Sec. 9.3.1 for important information regarding spiking samples.

9.2.4 Preparation of reference samples for specific determinative methods

The following sections provide guidance on the QC reference sample concentrates for many determinative methods. The concentration of the target analytes in the QC reference sample for the methods listed below may need to be adjusted to more accurately reflect the concentrations of interest in different samples or projects. If the concentration of an analyte is being evaluated relative to a regulatory limit or action level, see Sec. 9.3.3 for information on selecting an appropriate spiking level. In addition, the analyst may vary the concentration of the spiking solution and the volume of solution spiked into the sample. However, because of concerns about the effects of the spiking solution solvent on the sample, the total volume spiked into a sample should generally be held to no more than 1 mL. For any determinative method not listed below, the analyst should consult Sec. 9.3.3 and is free to choose analytes and spiking concentrations appropriate for the intended application. See the note in Sec. 9.3.1 for important information regarding spiking samples.

NOTE: All of the concentrations listed below refer to the concentration of the spiking solution itself, not the concentration of the spiked sample.

9.2.4.1 Method 6010, Inorganic Elements by ICP-AES -- The QC reference sample concentrate should contain each analyte at 1,000 mg/L in reagent water with appropriate type(s) and volume(s) of acid(s). See Method 6010.

9.2.4.2 Method 6020, Inorganic Elements by ICP-MS -- The QC reference sample concentrate should contain each analyte at 1,000 mg/L in reagent water with appropriate type(s) and volume(s) of acid(s). See Method 6020.

9.2.4.2 Method 7000, Inorganic Elements by Flame AAS -- The QC reference sample concentrate should contain each analyte at 1,000 mg/L in reagent water with appropriate type(s) and volume(s) of acid(s). See Method 7000.

9.2.4.3 Method 7010, Inorganic Elements by Graphite Furnace AAS -- The QC reference sample concentrate should contain each analyte at 1,000 mg/L

in reagent water with appropriat type(s) and volume(s) of acid(s). See Method 7010.

9.2.4.4 Method 7061, Arsenic by AA, Gaseous Hydride -- The QC reference sample concentrate should contain arsenic at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. See Method 7061.

9.2.4.5 Method 7062, Antimony and Arsenic by AA, Borohydride Reduction -- The QC reference sample concentrate should contain each analyte at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. See Method 7062.

9.2.4.5 Method 7063, Arsenic by ASV -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are commercially available as spectrophotometric standards. See Method 7063.

9.2.4.6 Method 7470, Mercury in Liquid Waste by Manual Cold-Vapor Technique -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are also commercially available as spectrophotometric standards. See Method 7470.

9.2.4.7 Method 7471, Mercury in Solid or Semisolid Waste by Manual Cold-Vapor Technique -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are also commercially available as spectrophotometric standards. See Method 7471.

9.2.4.8 Method 7472, Mercury by ASV -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are also commercially available as spectrophotometric standards. See Method 7472.

9.2.4.9 Method 7473, Mercury by Thermal, Decomposition, Amalgamation, and AA -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are also commercially available as spectrophotometric standards. See Method 7473.

9.2.4.10 Method 7474, Mercury by Atomic Fluorescence -- The QC reference sample concentrate should contain mercury at 1,000 mg/L in reagent water with appropriate volume of concentrated nitric acid. Stock solutions are also commercially available as spectrophotometric standards. See Method 7474.

9.2.4.11 Method 7741, Selenium by AA, Gaseous Reduction -- The QC reference sample concentrate should contain selenium at 1,000 mg/L in reagent water. See Method 7741.

9.2.4.12 Method 7742, Selenium by AA, Borohydride Reduction -- The QC reference sample concentrate should contain selenium at 1,000 mg/L in reagent water. See Method 7742.

9.2.5 Analyze at least four replicate aliquots of the well-mixed reference samples by the same procedures used to analyze actual samples. This will include a combination of the sample preparation method and the determinative method (a 6000 or 7000 series method). Follow the guidance on data calculation and interpretation presented in the determinative method.

9.3 Sample quality control for preparation and analysis

9.3.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair per analytical batch. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, then laboratories should use a matrix spike and matrix spike duplicate pair. The consideration as to which sample for a given batch is selected for QC analyses should be decided during the project planning process and documented in an approved sampling and analysis plan. The actual sample selected for QC analyses should be representative of the entire matrix composition for a given extraction batch, since data quality assumptions will likely be applied to all batch samples based on compliance to the stated data quality objectives and meeting the recommended precision and accuracy criteria. Therefore, it is inappropriate to combine dissimilar matrices in a single sample preparatory batch and expect to use a single set of QC samples. Sec. 9.3.3 provides guidance on establishing the concentration of the matrix spike compounds in the sample chosen for spiking.

The choice of analytes to be spiked should reflect the analytes of interest for the specific project. Thus, if only a subset of the list of target analytes provided in a determinative method are of interest, then these would be the analytes of interest for the project. In the absence of project-specific analytes of interest, it is suggested that the laboratory periodically change the analytes that are spiked with the goal of obtaining matrix spike data for most, if not all, of the analytes in a given determinative method. If these compounds are not target analytes for a specific project, or if other compounds are known to be of greater concern at a given site, then other matrix spike compounds should be employed.

CAUTION: The utility of the data for the matrix spike compounds depends on the degree to which the spiked compounds mimic the compounds already present in a field sample. Therefore, it is CRITICAL that any compounds added to a sample are added to the sample aliquot PRIOR TO any additional processing steps. It is also CRITICAL that the spiked compounds be in the same chemical form as the target compounds.

9.3.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume: e.g., reagent water for the water matrix or sand or soil for the solid matrix. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

9.3.3 The concentration of the matrix spike sample and/or the LCS should be determined as described in the following sections.

9.3.3.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit or action level, the spike should be at or below the regulatory limit or action level, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher.

9.3.3.2 If historical data are not available, it is suggested that an uncontaminated sample of the same matrix from the site be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries.

9.3.3.3 If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the concentration of the matrix spike should be at the same concentration as the reference sample (Sec. 9.2.4), near the middle of calibration range, or approximately 10 times the quantitation limit in the matrix of interest. It is again suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.

9.3.4 Analyze these QC samples (the LCS and the matrix spikes or the optional matrix duplicates) following the procedures in the determinative method. Calculate and evaluate the QC data as outlined in the determinative method.

9.3.5 Blanks -- The preparation and analysis of method blanks and other blanks are necessary to track potential contamination of samples during the extraction and analysis processes. Refer to Chapter One for specific quality control procedures.

9.4 The laboratory must also have procedures for documenting the effect of the matrix on method performance. Refer to Chapter One and each determinative method for specific guidance on developing method performance data.

9.5 Periodically, the accuracy of the temperature measurement system used to control the microwave equipment should be validated per Sec. 6.1.2.

9.6 (This step is not necessary if using temperature feedback control.) Each day that samples are extracted, the microwave-power calibration should be verified by heating 1 kg of ASTM Type II water (at 22 ± 3 EC) in a covered, microwave-transparent vessel for 2 min at the setting for 490 W and measuring the water temperature after heating per Sec. 10.5. If the power calculated (according to Sec. 12.0) differs from 490 W by more than ± 10 W, the microwave settings should be recalibrated according to Sec. 10.0.

9.7 The choice of an acid or acid mixture for digestion will depend on the analytes of interest and no single acid or acid mixture is universally applicable to all analyte groups. Whatever acid or acid mixture is employed, including those specifically listed in this method, the analyst must demonstrate adequate performance for the analytes of interest, at the levels of interest. At a minimum, such a demonstration will encompass the initial demonstration of proficiency described in Method 3500, using a clean reference matrix. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

## 10.0 CALIBRATION AND STANDARDIZATION

The following sections provide information regarding the calibration of microwave equipment.

NOTE: If the microwave unit uses temperature feedback control to control the performance specifications of the method, then performing the calibration procedure is not necessary.

10.1 Calibration is the normalization and reproduction of a microwave field strength to permit reagent and energy coupling in a predictable and reproducible manner. It balances reagent heating and heat loss from the vessels and is equipment dependent due to the heat retention and loss characteristics of the specific vessel. Available power is evaluated to permit the microwave field output in watts to be transferred from one microwave system to another.

Use of calibration to control this reaction requires balancing output power, coupled energy, and heat loss to reproduce the temperature heating profile given in Sec. 11.3.5. The conditions for each acid mixture and each batch containing the same specified number of vessels must be determined individually. Only identical acid mixtures and vessel models and specified numbers of vessels may be used in a given batch.

10.2 For cavity type microwave equipment, calibration is accomplished by measuring the temperature rise in 1 kg of water exposed to microwave radiation for a fixed period of time. The analyst can relate power in watts to the partial power setting of the system. The calibration format needed for laboratory microwave systems depends on the type of electronic system used by the manufacturer to provide partial microwave power. Few systems have an accurate and precise linear relationship between percent power settings and absorbed power. Where linear circuits have been utilized, the calibration curve can be determined by a three-point calibration method (see Sec. 10.4). Otherwise, the analyst must use the multiple point calibration method (see Sec. 10.3). Assistance in calibration and software guidance of calibration are available in Ref. 3 and the document listed in Sec. 13.3.5.

10.3 The multiple point calibration involves the measurement of absorbed power over a large range of power settings. Typically, for a 600 W unit, the following power settings are measured: 100, 99, 98, 97, 95, 90, 80, 70, 60, 50, and 40% using the procedure described in Sec. 10.5. These data are clustered about the customary working power ranges. Non-linearity has been encountered at the upper end of the calibration. Non-linearity is primarily encountered when using older instrumentation, however, multi-point calibration is recommended for use with all instrumentation when accurate and precise temperature feedback control is not available. If the system's electronics are known to have nonlinear deviations in any region of proportional power control, it will be necessary to make a set of measurements that bracket the power to be used. The final calibration point should be at the partial power setting that will be used in the test. This setting should be checked periodically to evaluate the integrity of the calibration. If a significant change is detected (± 10 W), then the entire calibration should be re-evaluated.

10.4 The three-point calibration involves the measurement of absorbed power at three different power settings. Measure the power at 100% and 50% using the procedure described in Sec. 10.5. From this 2-point line, determine the partial power setting that corresponds to the power, in watts, specified in the procedure to reproduce the heating profile specified in Sec. 11.3.5. Measure the absorbed power at that partial power setting. If the measured absorbed power does not correspond to the specified power within ± 10 W, use the multiple point calibration in Sec. 10.3. This point should also be used to periodically verify the integrity of the calibration.

10.5 Equilibrate a large volume of water to room temperature (22 ± 3 EC). One kg of reagent water is weighed (1,000.0 ± 0.1 g) into a fluorocarbon beaker or a beaker made of some other material that does not significantly absorb microwave energy (glass absorbs microwave energy and is not recommended). The initial temperature of the water should be 22 ± 3 EC measured to ± 0.05 EC. The covered beaker is circulated continuously (in the normal sample path) through the microwave field for 2 min at the desired partial power setting with the system's exhaust fan on maximum (as it will be during normal operation). The beaker is removed and the water vigorously stirred. Use a magnetic stirring bar inserted immediately after microwave irradiation (irradiating with the stir bar in the vessel could cause electrical arcing) and record the maximum temperature within the first 30 seconds to ± 0.05 EC. Use a new sample for each additional measurement. If the water is reused (after making adjustments for any loss of weight due to heating), both the water and the beaker must have returned to 22 ± 3 EC. Three measurements at each power setting should be made.

The absorbed power is determined by the following relationship:

$$P = \frac{(K)(C_p)(m)(\Delta T)}{t}$$

Where:

P = the apparent power absorbed by the sample in watts (W) (joule/sec)

K = the conversion factor for thermochemical calories $sec^{-1}$ to watts (K= 4.184)

$C_p$ = the heat capacity, thermal capacity, or specific heat [cal/(g EC)] of water

m = the mass of the water sample in grams (g)

$\Delta T$ = the final temperature minus the initial temperature (EC)

t = the time in seconds (s)

Using the experimental conditions of 2 minutes (120 sec) and 1 kg (1000 g) of distilled water [heat capacity at 25 EC is 0.9997 cal/(g EC)], the calibration equation simplifies to:

$$P = (\Delta T)(34.86)$$

NOTE: Stable line voltage is necessary for accurate and reproducible calibration and operation. The line voltage should be within manufacturer's specification, and during measurement and operation should not vary by more than ± 2 V (Ref. 3). Electronic components in most microwave units are matched to the system's function and output. When any part of the high voltage circuit, power source, or control components in the system are serviced or replaced, it will be necessary to recheck the system’s

calibration. If the power output has changed significantly (± 10 W), then the entire calibration should be re-evaluated.

## 11.0 PROCEDURE

11.1 Temperature control of closed vessel microwave instruments provides the main feedback control performance mechanism for this 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 EC and permit adjustment of the microwave output power within 2 sec.

11.2 All digestion vessels and volumetric ware must be carefully acid washed and rinsed with reagent water. When switching between high concentration samples and low concentration samples, all digestion vessels (fluoropolymer or quartz liners) should be cleaned by leaching with hot (1:1) hydrochloric acid (greater than 80 EC, but less than boiling) for a minimum of two hours followed by hot (1:1) nitric acid (greater than 80 EC, but less than boiling) for a minimum of two hours. The vessels should then be rinsed with reagent water and dried in a clean environment. 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. Polymeric or glass volumetric ware and storage containers should be cleaned by leaching with more dilute acids (approximately 10% V/V) appropriate for the specific material used and then rinsed with reagent water and dried in a clean environment.

11.3 Sample digestion

11.3.1 Weigh a well-mixed sample to the nearest 0.001 g into an appropriate vessel equipped with a controlled pressure relief mechanism. For soils, sediments, and sludges, use no more than 0.500 g. For oil or oil contaminated soils, initially use no more than 0.250 g. When large samples of oil are necessary, the use of Method 3052, which has sample scale-up options, is recommended. If the sample can not be well mixed and homogenized on an as received basis, then perform air or oven drying at 60 °C or less, crushing, sieving, grinding, and mixing 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).

11.3.2 Add 10 ± 0.1 mL concentrated nitric acid or, alternatively, 9 ± 0.1 mL concentrated nitric acid and 3 ± 0.1 mL concentrated hydrochloric 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. Improvements and optimal recoveries of antimony, iron, and silver from a variety of matrices upon addition of HCl are demonstrated in Sec. 17.0, in Figures 3 through 7 (these data are provided for guidance purposes only). The addition of hydrochloric acid may, however, limit the quantitation techniques or increase the difficulties of analysis for some quantitation systems.

WARNING: 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.

WARNING: 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.

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

CAUTION: The use of microwave equipment with temperature feedback control is needed 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.

11.3.3 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, do not leach this type of sample as described in this method, due to the high potential for unsafe and uncontrollable reactions. Instead, these samples may be predigested in a hood, with the vessel loosely capped to allow gases to escape, eliminating the hazard presented by rapid addition of thermal energy (MW power) to a reactive mixture. After predigestion, the samples may be digested according to the procedures described in this method.

11.3.4 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.

11.3.5 This method is compatible with a performance-based approach, designed to achieve or approach consistent leaching of the sample through achieving specific reaction conditions. The temperature of each sample should rise to 175 ± 5 EC in approximately 5.5 ± 0.25 min and remain at 175 ± 5 EC for 4.5 min, or for the remainder of the 10-min digestion period (see Refs. 2, 3, and 4 and the document listed in Sec. 13.3.4). Figure 2 gives the time versus temperature and pressure profile for a standard sediment sample (these data are presented for guidance purposes only). 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 characteristics of the vessels (Ref. 3).

11.3.5.1 The pressure should peak between 5 and 10 min for most samples (see Refs. 1 and 2 and the document listed in Sec. 13.3.4). 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.

11.3.5.2 Calibration control is applicable in reproducing this method provided the power in watts versus time parameters are determined to reproduce the specifications listed in Sec. 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 (see Ref. 3 and the document listed in Sec. 13.3.3). 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.)

11.3.6 At the end of the microwave program, allow the vessels to cool for a minimum of 5 min 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 1% 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.

11.3.7 Complete the preparation of the sample by venting microwave containers in a fume hood before uncapping, so as to avoid a rush of acid vapor that may still be in the headspace. When sufficiently cool to handle, carefully uncap the vessels, using the procedure recommended by the vessel manufacturer. Quantitatively transfer the sample to an acid-cleaned bottle. If the digested sample contains particulates which may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be centrifuged (Sec. 11.3.7.1), allowed to settle (Sec. 11.3.7.2), or filtered (Sec. 11.3.7.3).

11.3.7.1 Centrifugation -- Centrifugation at 2,000 - 3,000 rpm for 10 min is usually sufficient to clear the supernatant.

11.3.7.2 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.

11.3.7.3 Filtering -- If necessary, the filtering apparatus must be thoroughly cleaned and pre-rinsed with dilute (approximately 10% V/V) nitric acid. Filter the sample through qualitative filter paper into a second acid-cleaned container.

11.3.8 The removal or reduction of the quantity of the nitric and hydrochloric acids prior to analysis may be desirable. The chemistry and volatility of the analytes of interest should be considered and evaluated when using this alternative (Ref. 3). Evaporation to near dryness in a controlled environment with controlled purge gas and neutralizing and collection of exhaust interactions is an alternative where appropriate. This manipulation may be performed in the microwave system, if the system is capable of this function, or external to the microwave system in more common apparatus(s). This option must be tested and validated to determine analyte retention and loss and should be accompanied by equipment validation possibly using the standard addition method and standard reference materials. This alternative may be used to alter either the acid concentration and/or acid composition prior to analysis. (For further information, see Ref. 3 and Method 3052).

NOTE: The final solution typically requires nitric acid to maintain appropriate sample solution acidity and stability of the elements. Commonly, a 2% (v/v) nitric acid concentration is desirable. Waste minimization techniques should be used to capture reagent fumes. This procedure should be tested and validated in the apparatus and on standards before using on unknown samples.

11.3.9 Transfer or decant the sample into volumetric ware and dilute the digest to a known volume. The digest is now ready for analysis for elements of interest using appropriate elemental analysis techniques.

## 12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations -- The concentrations determined are to be reported on the basis of the actual weight of the original sample. All dilutions must be taken into account when computing the final results.

12.2 Prior to using this method, verify that the temperature sensing equipment is properly reading temperature. A procedure for verification is given in Sec. 6.1.2. This will establish the accuracy and precision of the temperature sensing equipment, which should be carried throughout the statistical treatment of the quality assurance data.

12.3 In calibrating the microwave unit (Sec. 10.0), the power absorbed (for each power setting) by 1 kg of reagent water exposed to 120 seconds of microwave energy is determined by the expression

$$\text{Power (in watts)} = (T_1 - T_2)\,(34.86)$$

Where:

$T_1$ = Initial temperature of water (between 21 and 25 EC to nearest 0.1 EC)

$T_2$ = Final temperature of water (to nearest 0.1 EC)

12.4 Plot the power settings against the absorbed power (calculated in Sec. 12.3) to obtain a calibration relationship. Alternatively, use a microwave calibration program to analyze the calibration data (see Ref. 3 and the document listed in Sec. 13.3.5). Interpolate the data to obtain the instrument settings needed to provide the wattage levels specified in Sec. 12.3.

12.5 Calculate the sample dry-weight fraction as follows:

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

Where:

$W_1$ = Wt for sample + vessel, before drying, g

$W_2$ = Wt for sample + vessel, after drying, g

$W_3$ = Wt for empty, dry vessel, g

12.6 Convert the extract concentration 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 = Concentration in extract (mg/L)

D = Dilution factor

S = Solid dry-weight fraction for sample, g/g

V = Volume of extract, mL x 0.001

W = Weight of undried sample extracted, g x 0.001

## 13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The fundamental chemical basis of this method with and without HCl has been compared with Method 3050 in several sources (see 13.3.4 and 13.3.6). Several papers have evaluated the leachability of NIST SRMs with this method (Ref. 1 and Sec. 13.3.5). Evaluations and optimizations of this method have been documented (Refs. 5 and 6), as well as additional leaches performed on more matrices, which may be addressed in future papers. This method has been determined to be appropriate for enhancing recoveries of certain analytes. This data is contained in Sec. 17 of this method. Matrices tested include SRM 2710 (Montana Soil - Highly Elevated Concentrations), SRM 2704 (Buffalo River Sediment), and SRM 1084a (Wear Metals in Oil). Analytes demonstrating better recoveries upon addition of HCl include antimony, iron, and silver. These data are provided for guidance purposes only.

13.3 The following documents may provide additional guidance and insight on this method and technique:

13.3.1 H. M. Kingston and L. B. Jassie, "Safety Guidelines for Microwave Systems in the Analytical Laboratory," in Introduction to Microwave Acid Decomposition: Theory and Practice, Kingston, H.M. and Jassie, L.B., eds., ACS Professional Reference Book Series, American Chemical Society, Washington, DC, 1988.

13.3.2 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water," ASTM, Philadelphia, PA, 1985, D1193-77.

13.3.3 Introduction to Microwave Sample Preparation: Theory and Practice, Kingston, H. M. and Jassie, L. B., Eds., ACS Professional Reference Book Series, American Chemical Society, Washington, DC, 1988.

13.3.4 H. M. Kingston and P. J. Walter, "Comparison of Microwave Versus Conventional Dissolution for Environmental Applications," *Spectroscopy*, Vol. 7 No. 9, 20-27, 1992.

13.3.5 P. J. Walter, Special Publication IR4718: Microwave Calibration Program, 2.0 ed., National Institutes of Standards and Technology, Gaithersburg, MD, 1991.

13.3.6 H. M. Kingston, P. J. Walter, S. J. Chalk, E. M. Lorentzen, D. D. Link, "Environmental Microwave Sample Preparation: Fundamentals, Methods, and Applications," in Microwave Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications, ACS Professional Reference Book Series, American Chemical Society, Washington, DC 1997.

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy,1155 16th Street, NW, Washington, DC 20036, http://www.acs.org.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult The Waste Management Manual for Laboratory Personnel, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street, NW, Washington, DC 20036, (202) 872-4477.

## 16.0 REFERENCES


1. H. M. Kingston, EPA IAG #DWI-393254-01-0 January 1 - March 31, 1988, quarterly report.

2. D. A. Binstock, W. M. Yeager, P. M. Grohse and A. Gaskill, "Validation of a Method for Determining Elements in Solid Waste by Microwave Digestion," Research Triangle Institute Technical Report Draft, RTI Project Number 321U-3579-24, November, 1989, prepared for the Office of Solid Waste, U.S. Environmental Protection Agency, Washington, DC 20460.

3. H. M. Kingston, S. Haswell, Microwave Enhanced Chemistry: Fundamentals, Sample Preparation, and Applications, ACS Professional Reference Book Series, American Chemical Society, Washington, DC 1997.

4. D. A. Binstock, P. M. Grohse, A. Gaskill, C. Sellers, H. M. Kingston, L. B. Jassie, "Development and Validation of a Method for Determining Elements in Solid Waste Using Microwave Digestion," *J. Assoc. Off. Anal. Chem.*, Vol. 74, 360 - 366, 1991.

5. H. M. Kingston, P. J. Walter, E. M., L. Lorentzen, G. P. Lusnak, "The Performance of Leaching Studies on Soil SRM's 2710 and 2711," Final Report to the National Institute of Standards and Technology, Duquesne University, Pittsburgh, PA, April 5, 1994.

6. D. D. Link, H. M. Kingston, P. J. Walter, "Development and Validation of the New EPA Microwave-assisted Leach Method 3051A," *Environmental Science and Technology*, Vol. 32, p. 3628-3632, 1998.

7. D. D. Link, H. M. Kingston, P. J. Walter, "Development and Validation of the EPA Microwave-assisted Methods 3015A and 3051A*:* Validation Studies for Updated Microwave Leach Methods," Proceedings for the Waste Testing and Quality Assurance Symposium, July 1997.

8. H. M. Kingston, P. J. Walter, "Comparison of Microwave verses Conventional Dissolution for Environmental Applications," *Spectroscopy*, Vol. 7 No. 9, 20-27, 1992.


## 17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables and figures referenced by this method.

TABLE 1

COMPARISON OF ANALYTE RECOVERIES FROM SRM 2704 (BUFFALO RIVER SEDIMENT)
USING BOTH DIGEST OPTIONS

| Element | 10 mL $HNO_3$ digest | 9 mL $HNO_3$ + 3 mL HCl digest | Total Analyte Concentration |
|---|---|---|---|
| Cd | 3.40 ± 0.34 | 3.62 ± 0.17 | 3.45 ± 0.22 |
| Cr | 84.7 ± 5.6 | 77.1 ± 12.6 | 135 ± 5 |
| Ni | 45.5 ± 5.9 | 42.2 ± 3.2 | 44.1 ± 3.0 |
| Pb | 163 ± 9 | 161 ± 17 | 161 ± 17 |

Elemental analysis was performed using either FAAS or ICP-MS.
Results reported in µg/g analyte (mean ± 95% confidence limit).
Total concentrations are taken from NIST SRM Certificate of Analysis.
These data are provided for guidance purposes only.
Data taken from Refs. 6 and 7.

TABLE 2

COMPARISON OF ANALYTE RECOVERIES FROM SRM 4355 (PERUVIAN SOIL)
USING BOTH DIGEST OPTIONS

| Element | 10 mL $HNO_3$ digest | 9 mL $HNO_3$ + 3 mL HCl digest | Total Analyte Concentration |
|---|---|---|---|
| Cd | 0.86 ± 0.16 | 0.85 ± 0.17 | (1.50) |
| Cr | 14.6 ± 0.47 | 19.0 ± 0.69 | 28.9 ± 2.8 |
| Ni | 9.9 ± 0.33 | 11.2 ± 0.44 | (13) |
| Pb | 124 ± 5.3 | 130 ± 3.6 | 129 ± 26 |

Elemental analysis was performed using either FAAS or ICP-MS.
Results reported in µg/g analyte (mean ± 95% confidence limit).
Total concentrations are taken from NIST SRM Certificate of Analysis.
Values in parenthesis are reference concentrations.
These data are provided for guidance purposes only.
Data taken from Refs. 6 and 7.

TABLE 3

COMPARISON OF ANALYTE RECOVERIES FROM SRM 1084a (WEAR METALS IN OIL) USING BOTH DIGEST OPTIONS

| Element | 10 mL $HNO_3$ digest | 9 mL $HNO_3$ + 3 mL HCl digest | Total Analyte Concentration |
|---|---|---|---|
| Cu | 91.6 ± 4.0 | 93.0 ± 2.6 | 100.0 ± 1.9 |
| Cr | 91.2 ± 3.3 | 94.3 ± 3.1 | 98.3 ± 0.8 |
| Mg | 93.2 ± 3.6 | 93.5 ± 2.8 | 99.5 ± 1.7 |
| Ni | 91.6 ± 3.9 | 92.9 ± 3.4 | 99.7 ± 1.6 |
| Pb | 104 ± 4.1 | 99.5 ± 5.1 | 101.1 ± 1.3 |

Elemental analysis was performed using either FAAS or ICP-MS.
Results reported in µg/g analyte (mean ± 95% confidence limit).
Total concentrations are taken from NIST SRM Certificate of Analysis.
These data are provided for guidance purposes only.
Data taken from Refs. 6 and 7.

FIGURE 1

PRESSURE PROFILES FOR THE HEATING OF DIFFERENT RATIOS
OF NITRIC ACID TO HYDROCHLORIC ACID

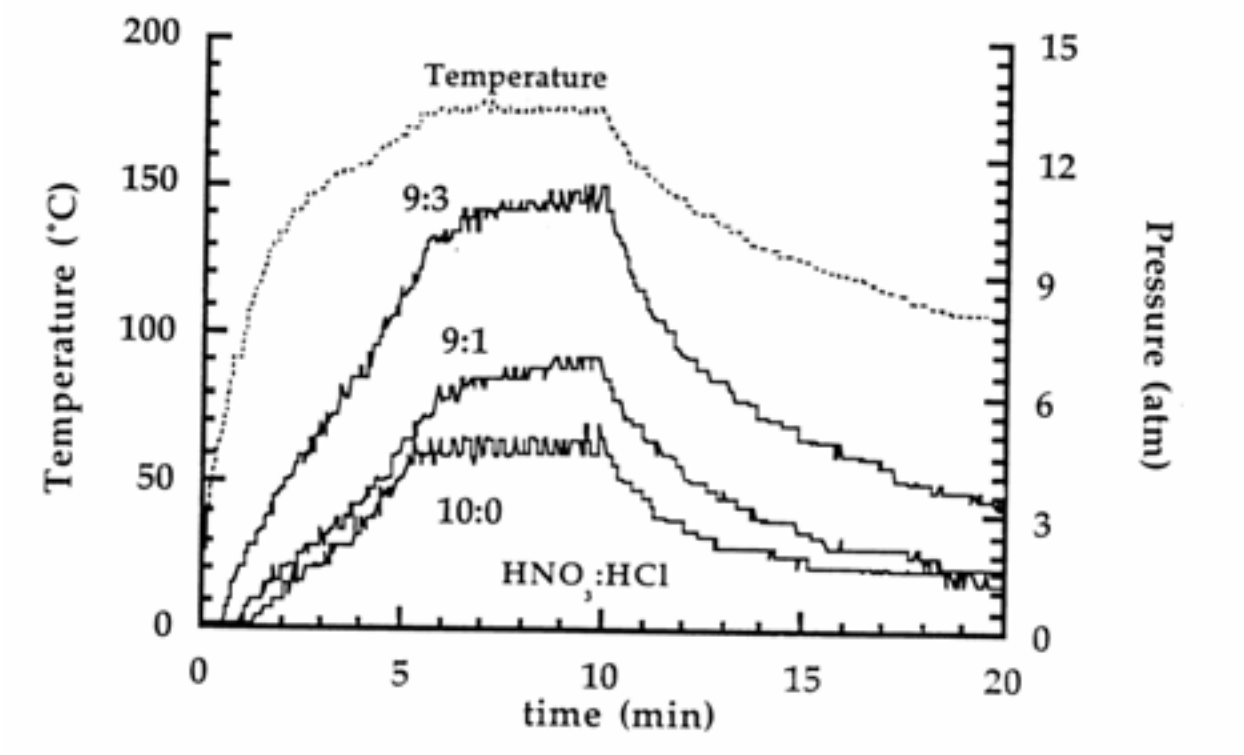



Figure taken from Refs. 6 and 7.

FIGURE 2

TEMPERATURE AND PRESSURE PROFILE
FOR NIST SRM 2704 (BUFFALO RIVER SEDIMENT)
USING DIFFERENT RATIOS OF NITRIC ACID TO HYDROCHLORIC ACID

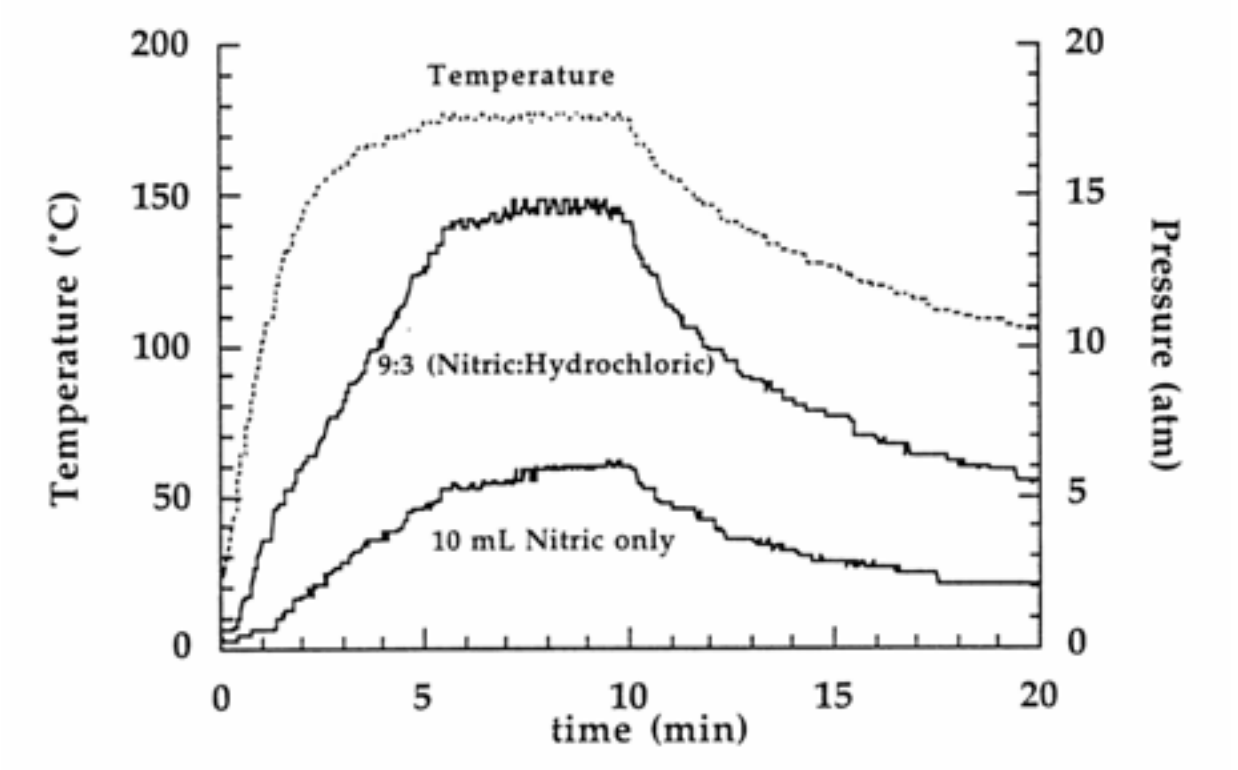



Figure taken from Refs. 6 and 7.

FIGURE 3

PERCENT RECOVERY OF ANTIMONY FROM NIST SRM 2710 (MONTANA SOIL) VERSUS VARIOUS COMBINATIONS OF NITRIC AND HYDROCHLORIC ACIDS (N=6)

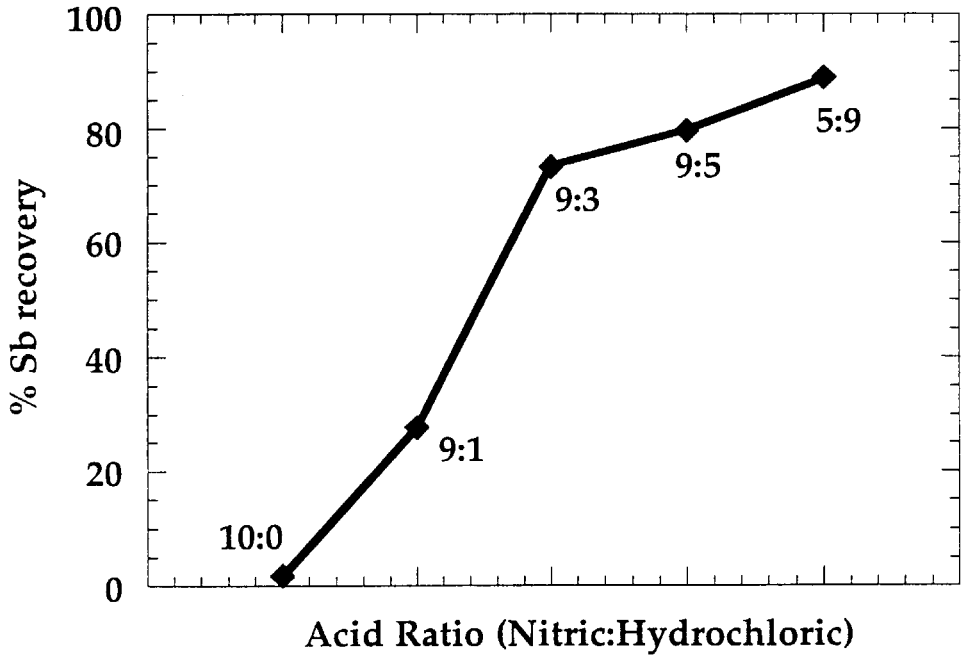



Figure taken from Refs. 6 and 7.

FIGURE 4

PERCENT RECOVERY OF ANTIMONY FROM NIST SRM 2704 (BUFFALO RIVER SEDIMENT) VERSUS VARIOUS COMBINATIONS OF NITRIC AND HYDROCHLORIC ACIDS(N=6)

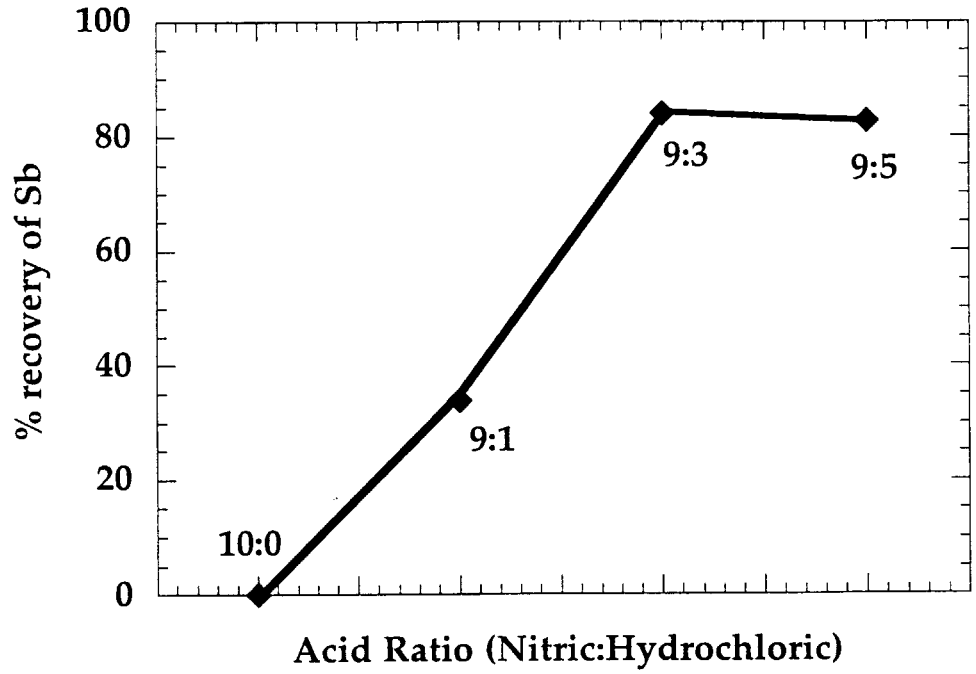



Figure taken from Refs. 6 and 7.

FIGURE 5

PERCENT RECOVERY OF IRON FROM NIST SRM 2704 (BUFFALO RIVER SEDIMENT) VERSUS VARIOUS COMBINATIONS OF NITRIC AND HYDROCHLORIC ACIDS (N=6)

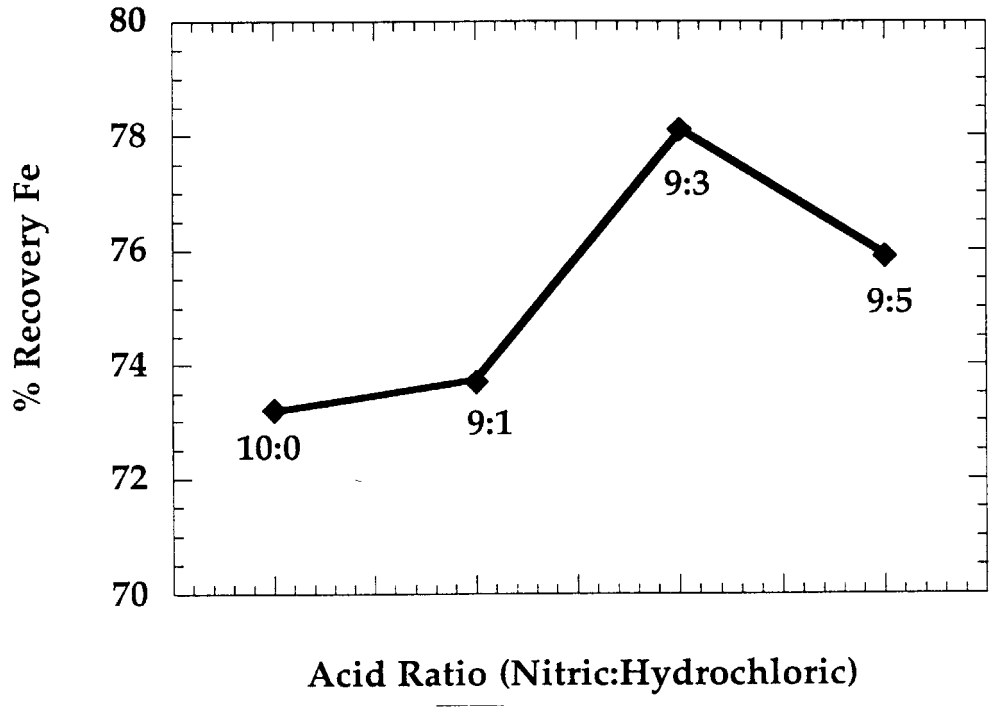



Figure taken from Refs. 6 and 7.

FIGURE 6

PERCENT RECOVERY OF SILVER FROM NIST SRM 2710 (MONTANA SOIL) VERSUS VARIOUS COMBINATIONS OF NITRIC AND HYDROCHLORIC ACIDS (N=6)

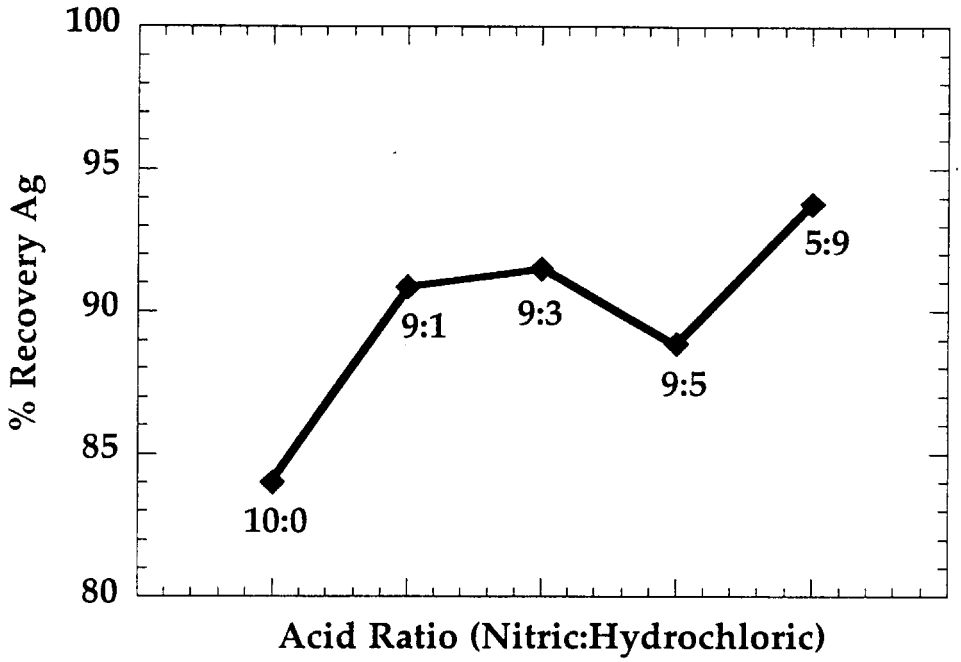



Figure taken from Refs. 6 and 7.

FIGURE 7

PERCENT RECOVERY OF ANTIMONY AND IRON, RESPECTIVELY, FROM SRM 4355 (PERUVIAN SOIL) USING BOTH DIGEST OPTIONS (10 ML $HNO_3$ AND 9 ML $HNO_3$ + 3 ML HCL ) (N=6)

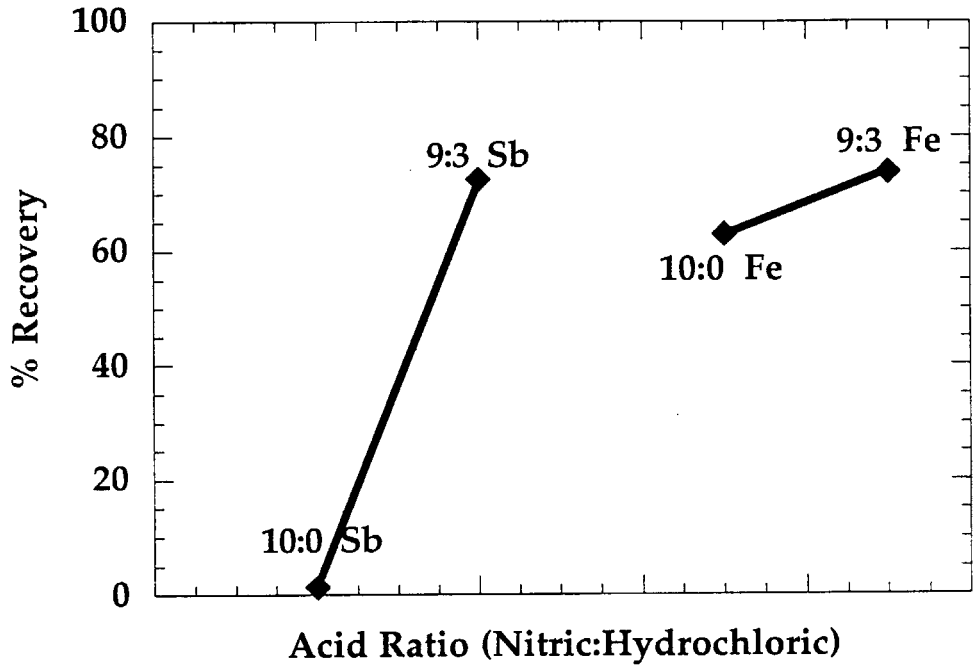
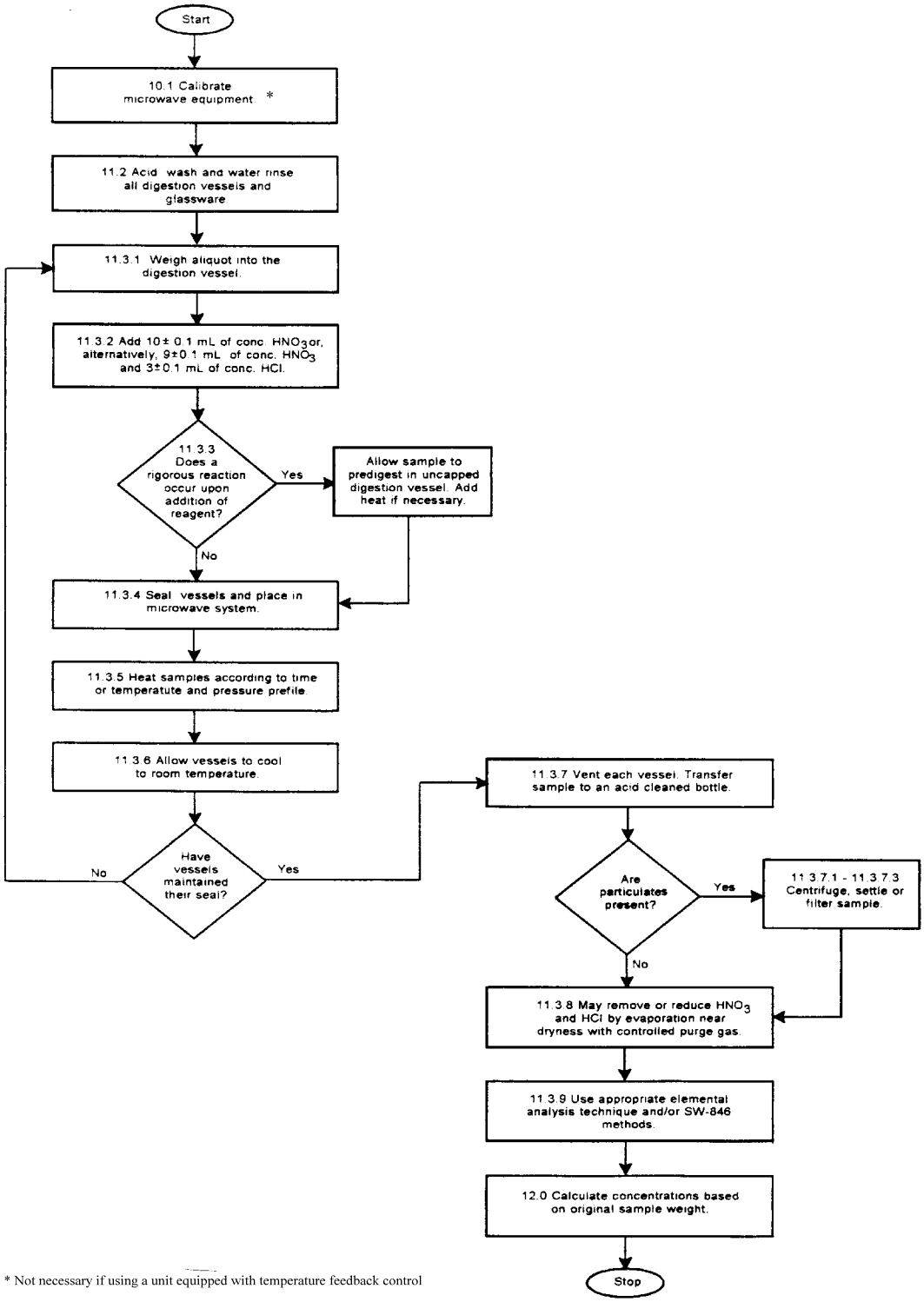



Figure taken from Refs. 6 and 7.

## MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS




* Not necessary if using a unit equipped with temperature feedback control