

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 (HNO₃), 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.