METHOD 3546

MICROWAVE EXTRACTION

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 method is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. This method was developed and validated on commercially-available solvent extraction systems. Its procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100 - 115 EC and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540), using less solvent and taking significantly less time than the Soxhlet procedure. Other systems and other types of vessels may be used, provided that the analyst demonstrates appropriate performance for the specific application.

1.2 This method is applicable to the extraction of semivolatile organic compounds, organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, phenoxyacid herbicides, substituted phenols, PCBs, and PCDDs/PCDFs, which may then be analyzed by a variety of chromatographic procedures. This method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application (see Method 3500 and Chapter Two).

1.3 This method has been validated for solid matrices containing from 50 to 10,000 µg/kg of semivolatile organic compounds, 250 to 2,500 µg/kg of organophosphorus pesticides, 10 to 5,000 µg/kg of organochlorine pesticides and chlorinated herbicides, 50 to 2,500 µg/kg of substituted phenols, 100 to 5,000 µg/kg of phenoxyacid herbicides, 1 to 5,000 µg/kg of PCBs, and 10 to 6000 ng/kg of PCDDs/PCDFs.

This method may be applicable to samples containing these analytes at higher concentrations and may be employed after adequate performance is demonstrated for the concentrations of interest (see Method 3500). It may also be applicable to classes of analytes, to fuel types, and to petroleum fractions other than those listed in Sec 1.2. However, in order to use this method for additional analytes, fuel types, petroleum fractions, or different concentrations, the analyst must demonstrate that the extraction conditions are appropriate for the analytes of interest. The analyst must also perform the initial demonstration of proficiency described in Sec. 9.2 and Methods 3500 and 8000. When expanding this method to other fuel types or petroleum hydrocarbons, the boiling point range or carbon number range of the material should be carefully defined and the quantitation approach be modified to match such ranges. Analysts are advised to consult authoritative sources, such as the American Petroleum Institute (API), for appropriate definitions of other fuel types or petroleum fractions.

NOTE: Mention of the analyses of other fuel types and petroleum fractions does *not* imply a regulatory requirement for such analyses, using this or any other method.

1.4 This method is only applicable to solid samples with small particle sizes. If practical, soil/sediment samples may be air-dried and ground to a fine powder prior to extraction. Alternatively, if worker safety or the loss of analytes during drying is a concern, soil/sediment samples may be mixed with anhydrous sodium sulfate or pelletized diatomaceous earth. (Drying and grinding samples containing PCDDs/PCDFs is *not* recommended, due to safety concerns.) The total mass of material to be prepared depends on the specifications of the determinative method and the sensitivity needed for the analysis, but an amount of 2 - 20 g of material is usually necessary and can be accommodated by this extraction procedure.

1.5 This method has been validated using a solvent mixture of hexane and acetone (1:1) from matrices such as soil, glass-fibers and sand. This solvent system or other solvent systems may be employed, provided that adequate performance is demonstrated for the analytes of interest (see. Sec. 7.4).

1.6 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) 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.

1.7 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Samples are prepared for extraction by grinding them to a powder and loading them into the extraction vessel.

WARNING: The drying and grinding of samples containing PCDDs/PCDFs is *not* recommended, due to safety concerns. Grinding may also be a concern for other more volatile analytes .

2.2 The appropriate solvent system is added to the vessel and sealed.

2.3 The extraction vessel containing the sample and solvent system is heated to the extraction temperature (see Sec. 11.9) and extracted for 10 min (or as recommended by the instrument manufacturer).

2.4 The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined.

2.5 The extract may be concentrated, if necessary, and, as needed, exchanged into a solvent compatible with the cleanup or determinative procedure to be employed.

3.0 DEFINITIONS

Refer to Chapter One and the manufacturer's instructions for definitions that may be relevant to this analytical 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 Four for general guidance on the cleaning of glassware.

4.2 Refer to Method 3500 for information regarding intereferences

4.3 If necessary, Florisil and/or sulfur cleanup procedures may be employed. In such cases, proceed with Method 3620 and/or Method 3660.

4.4 Samples for PCDD/PCDF analysis should be subjected to the various cleanup procedures described in the determinative methods (Methods 8280 and 8290).

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 use of organic solvents, elevated temperatures, and high pressures in this method present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. The sections to follow describe additional steps that should be taken.

5.3 The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool before opening (the use of a water bath is recommended for this purpose) and always monitor the temperature and pressure by re-connecting the control vessel to the apparatus prior to opening the vessels

5.4 During the heating step, some solvent vapors may escape through the vessel liner/seal cover. Follow the manufacturer's directions regarding the vessel assembly and instrument setup to prevent release of solvent vapors to the laboratory atmosphere.

5.5 The instrument may contain flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. If so equipped, follow the manufacturer's directions regarding replacement of extraction vessel seals when frequent vapor leaks are detected.

5.6 The drying or grinding of samples for PCDDs/PCDFs generally is not recommended due to safety concerns regarding worker exposure to analytes.

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 solvent extraction apparatus

6.1.1 The temperature performance specifications necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.5 EC and automatically adjusting the microwave field output power within 2 sec of sensing. Temperature sensors should be accurate to ± 2 EC. Temperature feedback control provides the primary performance mechanism for this method.

6.1.2 Microwave extraction vessels are needed. Vessels are available that can accommodate 1-g to 20-g samples. Vessels should be transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure specifications (minimum conditions of 200 EC and 200 psi) necessary to perform this procedure. Follow the manufacturer’s instructions regarding cleaning, handling, and sealing the vessels.

6.2 Apparatus for determining percent dry weight

6.2.1 Drying oven

6.2.2 Desiccator

6.2.3 Crucibles -- porcelain or disposable aluminum

6.3 Apparatus for grinding -- capable of reducing particle size to < 1 mm.

6.4 Analytical balance -- capable to weighing to 0.01 g.

6.5 Apparatus for separating sample from solvent extract

6.5.1 Glass funnels

6.5.2 Filter paper

6.5.3 Pasteur pipettes

6.6 Vials for collection of extracts -- 40-mL or 60-mL, or other appropriate volume, pre-cleaned, open-top screw cap equipped with polytetrafluoroethylene (PTFE)-lined silicone septum..

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. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water as defined in Chapter One.

7.3 Drying agents

7.3.1 Sodium sulfate (granular anhydrous), Na_2SO_4

7.3.2 Pelletized diatomaceous earth

7.3.3 The drying agents should be purified by heating at 400 EC for 4 hrs in a shallow tray, or by extraction with methylene chloride. If extraction with methylene chloride is employed, then a reagent blank should be prepared to demonstrate that the drying agent is free of interferences.

7.4 Extraction solvents

This method has been validated using a 1:1 mixture of hexane and acetone from matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy.

Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentrations of interest. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system 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.

Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water on the surface of the solid particles. The water-immiscible solvent extracts organic compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems.

All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 See the introductory material to Chapter Four, "Organic Analytes," Method 3500, and the specific determinative methods to be employed.

8.2 Solid samples to be extracted by this procedure should be collected and stored as any other solid samples containing semivolatile organics.

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 matrix. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. See Method 8000 for information on how to accomplish a demonstration of proficiency.

9.3 Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. For this method, this can be accomplished through the analysis of a solid matrix method blank (e.g., clean sand). As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination.

9.4 Any method blanks, matrix spike samples, or replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.5 Standard quality assurance practices should be used with this method as included in appropriate systematic planning documents and laboratory SOPs. All instrument operating conditions should be recorded.

9.6 Also refer to Method 3500 for extraction and sample preparation QC procedures and the determinative methods to be used for determinative QC procedures.

9.7 When listed in the appropriate determinative method, surrogate standards should be added to all samples prior to extraction. For PCDDs/PCDFs, the labeled internal standards listed in the determinative methods should be added to all samples prior to extraction. See Methods 3500, and 8000, and the appropriate determinative methods for more information.

9.8 As noted earlier, use of any extraction technique, including microwave extraction, should be supported by data that demonstrate the performance of the specific solvent system and operating conditions for the analytes of interest, at the levels of interest, in the sample matrix.

10.0 CALIBRATION AND STANDARDIZATION

There are no calibration or standardization steps directly associated with this sample extraction procedure, other than establishing the extraction conditions in Sec. 11.9.

11.0 PROCEDURE

11.1 Sample preparation

The sample preparation steps vary with the type of sample to be extracted, as described below. Where practical, samples should be air-dried and ground to a fine powder before extraction. However, where such steps are not practical because of concerns about loss of the more volatile analytes or potential contamination of the laboratory from high concentration samples, samples may be mixed with a drying agent such as sodium sulfate or pelletized diatomaceous earth prior to extraction.

WARNING: *The drying or grinding of samples for PCDDs/PCDFs generally is not recommended, due to safety concerns regarding worker exposure to these analytes.*

11.1.1 Sediment/soil samples

Decant and discard any water layer on a sediment sample. Discard any foreign objects such as sticks, leaves, and rocks. Mix the sample thoroughly, especially composited samples. When practical, air dry the sample at room temperature for 48 hrs in a glass tray or on hexane-rinsed aluminum foil. Alternatively, mix the sample with an equal volume of anhydrous sodium sulfate or pelletized diatomaceous earth until a free-flowing powder is obtained.

CAUTION: Dry, finely-ground soil/sediment allows the best extraction efficiency for nonvolatile, nonpolar organics, e.g., 4,4'-DDT, PCBs, etc. Air-drying may not be appropriate for the analysis of the more volatile organochlorine pesticides (e.g., the BHCs) or the more volatile of the semivolatile organics because of losses during the drying process. Worker safety may be an issue with the drying of soils containing PCDDs/PCDFs as well. Oven-drying during this step is not recommended for any analytes.

CAUTION: Drying should always be performed in a hood, to avoid contamination of the laboratory.

11.1.2 Waste samples

Prepare multiphase waste samples by the phase separation method in Chapter Two before extraction. *This extraction procedure is for solids only.*

11.1.3 Dry sediment/soil and dry waste samples amenable to grinding

Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Disassemble grinder between samples, according to manufacturer's instructions, and decontaminate with soap and water, followed by acetone and hexane rinses. The caution notes regarding drying in Sec. 11.1.1 also apply to the grinding process.

11.1.4 Gummy, fibrous, or oily materials not amenable to grinding

Cut, shred, or otherwise reduce in size these samples to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add anhydrous sodium sulfate, pelletized diatomaceous earth, sand, or other clean, dry reagents to the sample to make it more amenable to grinding.

11.2 Grind a sufficient weight of the dried sample from Sec. 11.1 to yield the sample weight needed for the determinative method (usually 10 - 30 g). Grind the sample until it passes through a 10-mesh sieve.

WARNING: *Grinding of samples for PCDDs/PCDFs generally is not recommended, due to safety concerns with samples containing these analytes.*

11.3 Determination of percent dry weight

When sample results are to be calculated on a dry weight basis, a separate portion of sample should be weighed at the same time as the portion used for analytical determination.

CAUTION: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.

11.3.1 Immediately after weighing the sample for extraction, weigh an additional 5-to 10-g aliquot of the sample into a tared crucible. Dry this aliquot overnight at 105 °C. Allow to cool in a desiccator before weighing.

11.3.2 Calculate the % dry weight as follows:

$$\%\ \text{dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

This oven-dried aliquot is not used for the extraction and should be appropriately disposed of once the dry weight is determined.

11.4 Transfer the ground sample to an extraction vessel. The weight of a specific sample that a vessel will contain depends on the bulk density of the sample and the amount of drying agent (if any) that was added to the sample in order to make it suitable for extraction. Analysts should ensure that the sample aliquot extracted is large enough to provide the necessary analytical sensitivity.

11.5 Prepare a method blank using an aliquot of a clean solid matrix such as quartz sand of the approximate weight of the samples. If a drying agent is added to the field samples being extracted, it must also be added to the method blank, in order to assess the possible contribution of the drying agent to the blank results.

11.6 Add the surrogates listed in the determinative method to each sample and method blank. Add the surrogates and the matrix spike compounds appropriate for the project to the two additional aliquots of the sample selected for spiking. If a drying agent is added to the field samples being extracted, it must also be added to the matrix spike aliquots, in order to assess the effect of the drying agent.

11.7 Add approximately 25 mL of the appropriate solvent system to the vessel and seal the vessel as instructed by the manufacturer.

11.8 Place the extraction vessel into the instrument and proceed with apparatus setup as instructed by the instrument manufacturer. If recommended by the manufacturer, include additional vessels containing water or other materials to the apparatus in order to ensure that all samples are exposed to a consistent amount of microwave energy across extraction batches.

11.9 Recommended extraction conditions

Temperature: 100 - 115 EC
Pressure: 50 - 150 psi
Time at Temperature: 10 - 20 min
Cooling: To room temperature
Filtering/Rinsing: With same solvent system

11.9.1 Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, since it is a result of the solvent system vapor pressure at the elevated temperature.

11.9.2 Once established, the same procedure should be used for all samples extracted for the same type of analysis.

11.10 Begin the extraction according to the instructions provided by the manufacturer.

11.11 Allow the extracts to cool to room temperature once the extractions are complete. After cooling, open the vessels and proceed with filtering and rinsing, combining all the filtrates.

11.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferants and the determinative method to be employed. Refer to Method 3600 for guidance on selecting appropriate cleanup methods. Excess water present in extracts may be removed by filtering the extract through a bed of anhydrous sodium sulfate. When using certain cleanup and/or determinative methods, a solvent exchange may be necessary prior to cleanup and/or analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

There are no calculations explicitly associated with this extraction procedure. See the appropriate determinative method for the calculation of final sample results.

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 Reference 4 presents a large body of information and specific data on a number of analytes. It provides the basis for a major portion of the performance work associated with this procedure. References 3 and 5 are reports of similar, more specific, studies. References 6 to 8 deal specifically with phenols. All of the method validation studies described in this method were performed using microwave apparatus operating at 2450 MHz.

13.3 Chlorinated pesticides

Single-laboratory accuracy data were obtained for chlorinated pesticides extracted from soil, glass-fiber, and sand matrices. Concentrations of each target analyte ranged between 500 and 1,000 µg/kg. Four real-world split samples contaminated with pesticides and creosotes were also used (obtained from US EPA ERT, Edison, NJ). The latter were extracted by an independent laboratory using standard Soxhlet procedures and results compared to those obtained with this procedure. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by the appropriate determinative method. Method blanks and five spiked replicates were included. Work was also carried out to assess the level of degradation of thermally labile pesticides and it was found that no significant degradation takes place under the procedure described in this method. The data are reported in detail in Reference 4. Data summary tables are included in Method 8081, for guidance purposes only.

13.4 Semivolatile organics

Single-laboratory accuracy data were obtained for semivolatile organics extracted from soil, glass-fiber, and sand samples. Concentrations of each target analyte were about 500 µg/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by the appropriate determinative method. Method blanks and five spike replicates were included. The data are reported in detail in Reference 4. Data summary tables are included in Method 8270, for guidance purposes only.

13.5 PAHs

Single-laboratory accuracy data were obtained for PAHs extracted from five reference materials comprising marine sediments (HS-3, HS-4, and HS-5, all from the National Research Council of Canada), lake sediments (SRM-1491, from the National Institute of Science and Technology), and a soil contaminated with creosote (SRS103-100, from Fisher Scientific, Fairlawn, NJ). Work was also conducted with soil, glass-fiber, and sand samples spiked between 100 and 2,000 µg/kg. All samples were extracted using 1:1 hexane:acetone. One real-world split sample contaminated with creosote and pesticides was also used (obtained from US EPA ERT, Edison, NJ). The latter was extracted by one laboratory using standard Soxhlet procedures and results compared to those obtained with this procedure. Extracts were analyzed by the appropriate determinative method. Method blanks, spikes and five spiked replicates were included. The data are reported in detail in Reference 4. Data summary tables are included in Method 8270, for guidance purposes only.

13.6 PCBs

Single-laboratory accuracy data were obtained for PCBs extracted from three reference materials (EC-1, EC-2, EC-3 -- from Environment Canada). Work was also conducted with soil, glass-fiber, and sand samples spiked between 200 and 10,000 µg/kg (total PCBs). All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by the appropriate determinative method. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are reported in detail in Reference 4. Data summary tables are included in Method 8082, for guidance purposes only.

13.7 Chlorinated herbicides (phenoxyacid herbicides)

Multi-laboratory accuracy data were obtained for chlorinated herbicides extracted from a certified spiked material (obtained from ERA, Arvada, CO). This soil was spiked by ERA at 100 µg/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by Method 8151. Method blanks and three replicates from five laboratories were included. Data summary tables are included in Method 8151, for guidance purposes only.

13.8 Phenols

Single-laboratory accuracy data were obtained for phenols extracted from a number of spiked soils and real-world split soils. Concentrations ranged between 200 and 10,000 µg/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by the appropriate determinative method. The data are reported in detail in References 4 to 8. Data summary tables are included in Method 8041, for guidance purposes only.

Multi-laboratory accuracy data were obtained for phenols extracted from a certified spiked material (obtained from ERA, Arvada, CO). This soil was spiked by ERA at 250 µg/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by Method 8041. Method blanks and three replicates from five laboratories were included. Data summary tables are included in Method 8041, for guidance purposes only.

13.9 Organophosphorus pesticides and chlorinated herbicides

Multi-laboratory performance data were obtained for organophosphorus pesticides extracted from a certified spiked material (obtained from ERA, Arvada, CO). This soil was spiked by ERA at 250 µg/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by Method 8141. Method blanks and three replicates from five laboratories were included. Data summary tables are included in Method 8141, for guidance purposes only.

13.10 Dioxins and furans

Single-laboratory accuracy data were obtained for dioxins and furans extracted from two soil reference materials (DX-1 from Environment Canada and SRM-1944 from NIST) containing the analytes of interest at concentrations between 10 and 6,000 ng/kg. All samples were extracted using 1:1 hexane:acetone. Extracts were analyzed by the appropriate determinative method. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are reported in detail in References 9 and 10.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. 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 St., NW Washington, D.C. 20036, http://www.acs.org.

14.3 Extraction of organic compounds using microwave extraction conforms with EPA's pollution prevention goals. The volumes of solvent employed are generally smaller than with other extraction procedures.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted 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 at the address listed in Sec. 14.2.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

There are no tables or figures associated with this method.