

# ETHOS X APPLICATION REPORTS

Microwave Extraction for Environmental Applications



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# Introduction

## EPA METHOD 3546 & ASTM D-6010

EPA 3546 is intended to be guidance method which contains 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 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.

## SCOPE AND APPLICATION

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.

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).

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.

This method is only applicable to solid samples with small particle sizes. If practical, soil/sediment sample may be airdried 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 sulphate 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. This method has been validated using a solvent mixture of Acetone/Hexane (1:1 v/v) from matrices such as soil, glassfibers and sand. This solvent system or other solvent systems may be employed, provided that adequate performance is demonstrated for the analytes of interest.

# Application Report

## INSTRUMENTATION

Microwave solvent extraction apparatus - FastEX-24 rotor consists of a 24-position carousel, which holds large pressure vessels made of an innovative and unique inert polymer material. At the core of the vessel there is a disposable and inexpensive 100 mL glass vial. A self-regulating pressure cover assures safe operations of the system. Temperature and pressure are monitored and controlled in all vessels by non-contact sensors. A fast easy-to-use automatic capping tool makes sure all vessels are properly tightened prior the microwave run. The external WEFロン vessel ensure an homogeneous heating inside the vials during extraction.

## SAMPLE WEIGHT AND REAGENTS

Reagent grade chemicals must be used in all tests. 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. Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix, at the concentration 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.

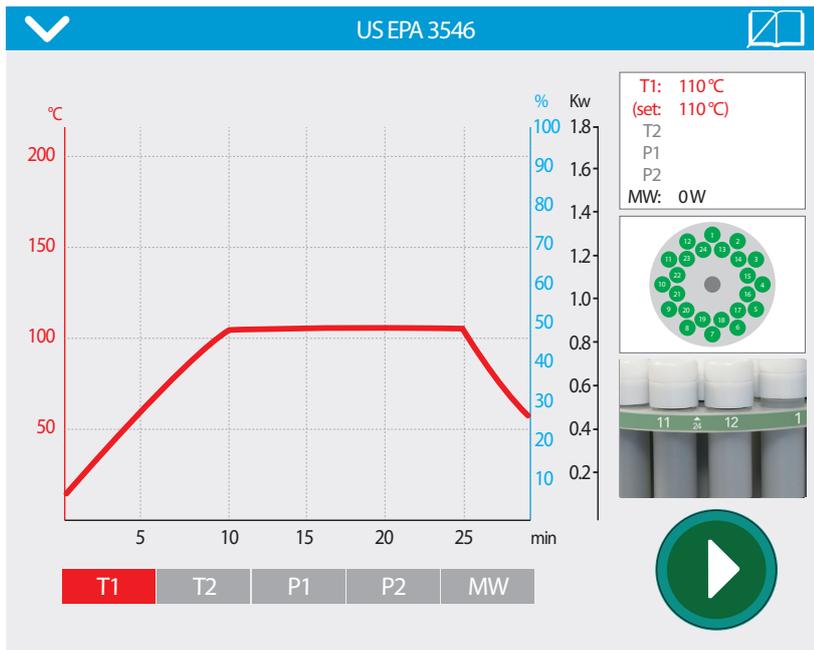
Sample amount	Reagents	Solvents volume	Temperature	Pressure	Time at Temperature
2-20g	Hexane and Acetone (1:1)	25mL	100-115°C	4-12 bar	10-20 minutes

## PROCEDURE

The sample preparation steps vary with the type of sample to be extracted. 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 analytes or potential contamination of the laboratory from high concentration samples, samples may be mixed with a drying agent such as sodium sulphate or pelletized diatomaceous earth prior to extraction.

1. Samples are prepared for extraction by grinding them to a powder and loading them into the extraction vessel. If the sample is wet, mix the sample with an equal volume of anhydrous sodium sulphate or pelletized diatomaceous earth until a free-flowing powder is obtained. Note: the air-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. Add the solvent mixture into the extraction vessel.
3. Introduce the Weflon button and the stirring bar inside the vessel and close it.
4. Heat the vessel to the extraction temperature in accordance with the microwave program and extracted for at least 10 min.
5. Cool the extraction mixture to room temperature.
6. Open the vessel and filter the content rinsing the solid material with the mixture of solvents used for the extraction.
7. If required, clean up the extract before performing analyte determination.

## TEMPERATURE PROFILE



## COMMENTS

US EPA method 3546 temperature profile, suitable for the microwave solvent extraction of:

- Chlorinated pesticides
- Semivolatile organics
- PAHs
- PCBs
- Chlorinated herbicides
- Phenols
- Organophosphorus pesticides
- Dioxins
- Furans