

## US EPA 3546

The US EPA method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The procedure uses microwave energy to produce elevated temperature and pressure conditions (100-115°C and 4-12 bar) in a closed vessel containing the sample and organic solvents to achieve analyte recoveries equivalent to those obtained from Soxhlet extraction, using less solvent and taking significant less time than the Soxhlet procedure.

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.

The US EPA method 3546 is applicable to solid samples with small particle sizes. 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 hexane and acetone (1:1) for matrices such as soil, glass-fibers and sand. Hexane is a water-immiscible 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.

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



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

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

## THE NEW MILESTONE



- Ease of use Disposable vials
- Elevated sample throughput 24 samples simultaneously
- Minimum solvent usage
- Low running cost
- Suitable for US EPA method 3546
- Suitable for ASTM method D5765-05
  (Standard Practice for Solvent Extraction of Total Petroleum Hydrocarbons from Soils and Sediments)

## ETHOS X

The overall concept of the ETHOS X for environmental applications has been developed looking at the working routine of several thousands contract laboratories around the world performing solvent extraction, with the aim of helping them, by offering an integrated solution able to render their activity easier, faster and safer. The ETHOS X processes more samples in less time and uses a fraction of the solvent required by other technologies. It is very simple to use and, following an already established US EPA and ASTM methods, it assures consistent results with a minimal running cost.

The new Milestone ETHOS X microwave cavity has a volume in excess of 70 liters, by far the largest currently available. With an installed power of 1900 Watt, the ETHOS X is the most powerful microwave platform system available for extraction. A high definition digital camera allows the chemist to monitor the progress of the extraction whilst fully protected by the all-stainless steel door of the instrument. Should there be a sudden over-pressurization of the cavity, the door slightly opens for rapid and safe pressure release and the microwave power is instantaneously cut off. A built-in magnetic stirrer is designed to ensure vigorous agitation of the solutions in all vessels.

The new Milestone ETHOS X is equipped with the most advanced yet easy to use reaction temperature and pressure sensors for complete quality control of the extraction conditions. It is controlled via a compact terminal with an easy-to-read, bright, full-color, touchscreen display. The terminal runs a completely new icon-driven, multi-language software to provide easy control of the microwave run.

A completely new rotor has been specifically developed by Milestone to fully accomplish the US EPA method 3546 requirements. This new 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.





## TECHNICAL SPECIFICATIONS

Milestone ETHOS X Advanced Microwave Extraction System for Environmental Applications

The complete system includes:

- Microwave cavity: 18/8 stainless steel housing; largest microwave cavity: 43 x 40 x 41 (H) cm (70,5 liters); inlet/outlet ports: upper flange 36 mm ID, plus additional ports on the side walls; chassis protected against corrosive media with polymer coating; door completely made of 18/8 stainless steel with multiple independent safety interlocks to prevent microwave emission in case of improper closure or misalignment.
- Built-in exhaust system located above the cavity and separated from electronics to prevent corrosion.
- Microwave emission with dual magnetron system with rotating diffuser for homogeneous microwave distribution in the cavity; two 950 Watt rated magnetrons, for a total of 1900 Watt; exclusive magnetron protection from reflected microwave power; continuous and PID-controlled microwave emission at all power levels.
- Built-in contact-less infrared temperature control, contact-less pressure control, magnetic stirrer (optional), software-controlled digital camera (with terminal 660).
- Safety standards: EN61010-1:2001; EN61010-2-010:2003; UL61010-1:2004; CAN/CSA-C22.2 No 61010-1:2004; CAN/CSA-C22.2 No 61010-2-010:2004; IEC 61010-2-010:2003; EN61326-1:2006.
- Terminal 660, touch screen; 6,5" TFT display; 640x480 VGA resolution with 64k colors; 5 USB ports, 1 RS-232 port, 1
  LAN port, 2 video ports; terminal 480, touch screen; 4,3" TFT display; 480x272 VGA resolution with 16M colors.
- Icon-driven multi-language operating software (Chinese, English, French, German, Italian, Japanese, Polish, Portuguese, Russian, Spanish and Turkish) software allowing the user to edit, save and run a virtually unlimited number of methods.
- Extraction rotor complete with 24 vessels, self-regulating pressure valves and disposable glass vials.
- Weight: ca. 84 kg.
- Dimensions: 54 x 64 x 69 (H) cm.
- Power supply: 230-240V/50 or 60Hz, 3,5 kWatt.

HELPING CHEMISTS – Milestone has been active since 1988 in the field of microwave sample preparation. With over 20000 instruments installed worldwide, we are the acknowledged industry leader in microwave technology. Milestone vision is to help chemists by providing the most technologically advanced instrumentation for research and quality control. Our products offer a wide range of applications, such as microwave acid digestion, solvent extraction, synthesis and ashing. Furthermore we manufacture products for acid purification and direct mercury determination in solid, liquid and gas samples. We offer our customers the highest level of application support, building up over the years a relationship based on trust and responsibility.



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