

## Application Note

Multiwave 3000 Microwave Sample Preparation System

# EPA methods 3015A, 3051A and 3052

### EPA-methods

The Office of Solid Waste of the US-EPA has released the publication *SW-846 "Test Methods for Evaluating Solid Waste"*, an official compendium of approved analytical and sampling methods complying with RCRA regulations. SW-846 is a multi-volume document that changes over time as new information and data are developed. First issued in 1980, it is continually reviewed and currently in its third edition. Advances in analytical instrumentation and techniques are continually reviewed and incorporated into periodic updates to support changes in the regulatory program and to improve method performance and cost effectiveness.

An electronic copy of the complete SW-846 manual is available at:

<http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

### Environmental Microwave Sample Preparation Standards

Currently the US EPA specifies three closed-vessel microwave assisted sample preparation procedures for the analysis of up to 26 elements. Two are leaching methods (3015A and 3051A), one is flexibly adaptable for total dissolution (3052).

For some originally hot-plate based open vessel methods (3031, 3050 and 3060) microwave heating is now applicable, too, in order to gain better precision.

Further there are ASTM, NPDES and AOAC sample preparation standards based on microwave technology. In some other countries (e.g. France, China) national microwave standard methods have been established, too, but internationally the EPA methods are most important. As the use of microwave for sample preparation gains importance more standards can be expected.

Generally the aim is to improve analytical performance and reliability.

### Abbreviations

EPA	US Environmental Protection Agency
OSW	Office of solid waste
RCRA	Resource Conservation Recovery Act
ASTM	American Society for Testing and Materials
NPDES	National Pollutant Discharge Elimination Systems

### Requirements

The accuracy of all the methods depends on precise replications of the reaction conditions. For this reason the EPA methods clearly prescribe both chemistry and temperature profiles. Other topics are microwave apparatus requirements, sample collection, preservation and storage, calibration and standardization, description of the procedure and data analysis and calculations.

### Transfer of methods

EPA methods do not depend on the used instrument, they can be done on different kinds of analytical equipment.

Earlier methods (3015 and 3051) prescribed the used power settings. More advanced methods like 3015A, 3051A and 3052 prescribe as performance criteria a defined chemistry and reaction temperature versus time.

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## How Multiwave 3000 meets EPA

The Multiwave 3000 is delivered with a stored methods library which includes the three closed-vessel EPA methods.

We recommend to use the rotor MF100 loaded with 8 vessels for the EPA method 3015A. The use of the optional p/T sensor accessory is essential. Besides the simultaneous control of pressure and temperature on one position, all 8 positions are controlled by an IR temperature sensor for full documentation.

Methods 3051A and 3052 can be done with a fully loaded MF100 rotor with 16 vessels in parallels. Again the IR-sensor shows any vessel-to-vessel deviation in temperature.

The rotor 16HF100, which has higher temperature and pressure specifications, can be used for performing EPA methods as well.

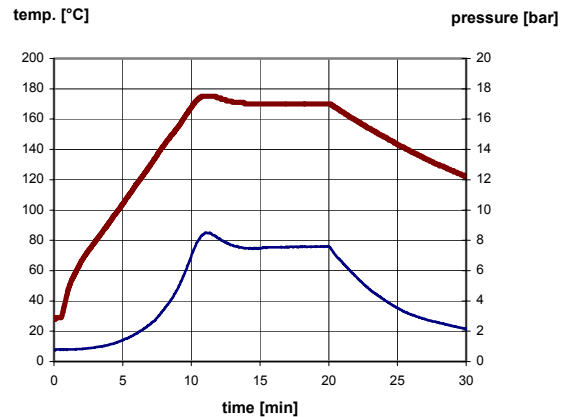
## Benefits of the Multiwave 3000 technology

- EPA methods are installed in the methods library
- Vessel specifications meet EPA temperature-, volume- and pressure requirements
- Simultaneous temperature/pressure control in one vessel with an uncertainty of  $\pm 2\text{ }^{\circ}\text{C}$  and  $\pm 0.2\text{ bar}$
- Automated, software-controlled calibration accessory for routine re-calibration of the temperature sensors by the user
- IR-temperature control on all positions
- Temperature – pressure – power – diagrams and full documentation of each single sample for GLP compliance
- Built-in-cooling system to minimize cool-down times for high sample throughput
- Venting valves for safe and controlled release of reaction gases after digestion
- Unpulsed microwave power over the full range of 1400 W for precise reaction control
- 10-fold overpressure safety system for safe operation in case of unforeseen reactions

## Multiwave 3000 examples

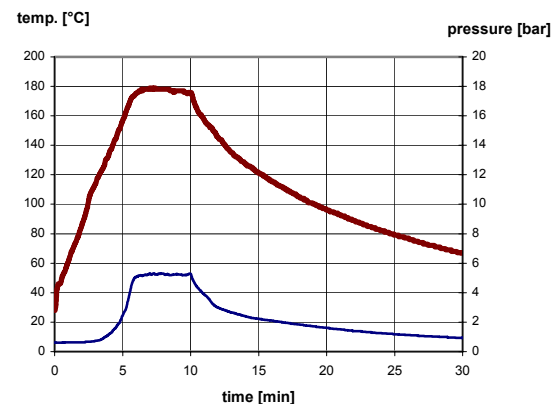
### EPA 3015A – Rotor MF100

45 mL drinking water + 5 mL  $\text{HNO}_3$



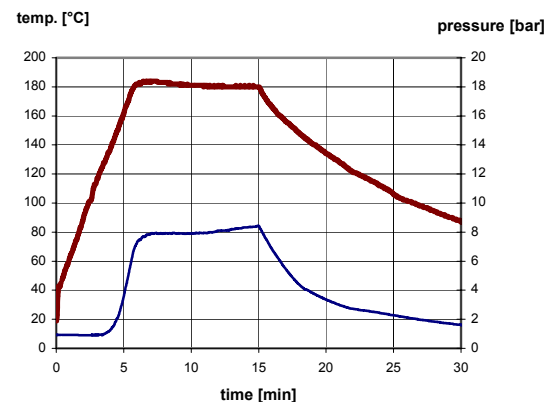
### EPA 3051A – Rotor MF100

0.5 g soil + 10 mL  $\text{HNO}_3$



### EPA 3052 – Rotor MF100

0.25 g soil + 9 mL  $\text{HNO}_3$  + 3 mL HF



# Application Note

## Scope of methods

Method	3015 A	3051 A	3052
<b>Title</b>	Microwave assisted acid leaching of aqueous samples and extracts	Microwave assisted acid digestion of sediments, sludges, soils and oils	Microwave assisted acid digestion of siliceous and organically based matrices
<b>Goal</b>	Perform extraction using microwave heating with nitric acid, or alternatively, nitric acid and hydrochloric acid. This method is not intended to accomplish total decomposition of the sample.	Perform extraction using microwave heating with nitric acid, or alternatively, nitric acid and hydrochloric acid. This method is not intended to accomplish total decomposition of the sample.	Total sample decomposition for general use with judicious choice of acid combinations.
<b>Typical Samples</b>	Aqueous samples, drinking water, mobility-procedure extracts, wastes that contain suspended solids	Sediments, sludges, soils and oils	Ashes, biological tissues, oils, oil contaminated soils, sediments, sludges and soils
<b>Elements</b>	Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Mo, Ni, K, Se, Ag, Na, Sr, Tl, V, Zn	Like 3015 A	Like 3015 A
<b>Sample Wt.</b>	45 mL	0.5 g for soil, sediment, sludge 0.25 g for oil and contaminated soil	0.5 g typ. 0.25 g for oil and contaminated soil
<b>Reagents</b>	5 mL HNO <sub>3</sub> or optionally 4 mL HNO <sub>3</sub> + 1 mL HCl	10 mL HNO <sub>3</sub> or alternatively 9 mL HNO <sub>3</sub> + 3 mL HCl	9 mL HNO <sub>3</sub> + 3 mL HF HF quantities may be varied between 0 and 5 mL. Addition of HCl, H <sub>2</sub> O <sub>2</sub> or water is permitted
<b>Temperature Profile</b>	Ramp: 10 min. Temp: 170 °C ± 5 °C Hold : 10 min. Total: 20 min	Ramp: 5.5 min. Temp: 175 °C ± 5 °C Hold : 4.5 min. Total: 10 min	Ramp: 5.5 min. (up to 10 min. for critical samples) Temp: 180 °C ± 5 °C Hold : 9.5 min. Total: 15 min to 19.5 min.